

Electrically and Electrochemically Active Composite Based on Polyester/Reduced Graphene Oxide/Polypyrrole with Remarkable Washing Durability

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Abstract- The present work explores a facile route to prepare a durable conductive fabric by using reduced graphene oxide and polypyrrole. Prior to coating of active materials, polyester surface experienced a modification which resulted in high uptake of materials. Then, dip-coating approach was used to deposit graphene oxide on the modified polyester. After reduction of graphene oxide, polypyrrole particles grew on the fabric surface through an *in situ* polymerization method. SEM, XRD, FTIR, and TGA were employed to investigate the morphology and chemical structure of the samples. A high electrical conductivity of 0.98 S.cm^{-1} was obtained which arises from establishing the numerous conduction routes in the structure. A mere decrease in conductivity after 20 laundry cycles confirms the excellent washing durability of the conductive fabric. Moreover, a high specific capacitance of 8.3 F.g^{-1} was recorded for this fabric by cyclic voltammetry in a three-electrode measurement system.

Keywords: polyester, reduced graphene oxide, polypyrrole, conductive fabric, surface modification, alkaline hydrolysis

I. INTRODUCTION

Conductive fabrics are vitally important components of smart textiles such as wearable electronics [1-4]. They not only take advantage of textiles such as flexibility, low cost, light weight, safety, and breathability but also provide an electrical conductive substrate [5]. A common method to prepare conductive fabric is coating conventional fabrics with electrically conductive materials [6,7]. In fact, the porous nature of fabrics allows the significant uptake of active material. In addition, owing to presence of reactive

functional groups in fabrics structures, they are often able to interact chemically with loaded materials which results in durability of coating. Polyester (polyethylene terephthalate, PET) is an exception because the poor wettability as well as the lack of functional groups makes it difficult to interact chemically. Hence, surface modification of PET is considered as an important pre-treatment prior to coating [8]. Through the modification, some functionalities are inserted on the surface of PET. The type of introduced functional group depends on the chemical activation method. The most common method is alkaline hydrolysis of PET. Generally, alkaline hydrolysis is defined as the cleavage of ester bonds on the PET surface caused by reaction with strong base such as sodium hydroxide (NaOH). This leads to formation of hydroxyl and carboxyl groups on the PET surface. Furthermore, as a consequence of modification, a great number of small-sized voids are created on the fiber surface and porosity increases [8].

Metals, conducting polymers (CPs) and carbon materials have been frequently employed to decrease electrical resistivity of textiles [7,9-12]. Among them the composite of carbon materials and CPs have drawn a considerable attention in the recent years. The free standing paper-like film of reduced graphene oxide/polypyrrole (rGO/PPy) which synthesized by vacuum filtration and further HI reduction achieved a conductivity of 142 S.cm^{-1} . The prepared solid-state supercapacitor showed a capacitance of 345 F.g^{-1} and 9.4% drop in capacitance after 1000 cycles [13]. Sahoo *et al.* prepared a composite of graphene/PPy nanofiber by use of sodium alginate, and the conductivity of 1.45 S.cm^{-1} was obtained. Sodium alginate has been used in order to form an expanded network structure as well as making nanofiber morphology of PPy. The highest specific capacitance value was 466 F.g^{-1} at 10 mV.s^{-1} [14]. Liu *et al.* reported the specific capacitance of 400 F.g^{-1} at 0.3 A.g^{-1} current density for chemically reduced graphene oxide and PPy nanotube [15].

Rather than free standing films, the active materials are also coated on textiles. Molina *et al.* reported

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PPy-coated PET fabric in which graphene oxide (GO) acted as agent to neutralize the positive charge of PPy. They stated that conductivity of PET/PPy/GO could be tuned by varying the GO content in the synthesis solutions and by increasing GO content, the surface conductivity decreased [12]. Berendjchi *et al.* prepared PET/rGO/PPy composite with surface resistivity of $5 \Omega \cdot \text{sq}^{-1}$ which was notably lower than that obtained for PET/rGO and PET/PPy. The voltammogram of PET/rGO/PPy revealed a capacitive response that was markedly weak [7]. Xu *et al.* deposited rGO and PPy on cotton fabric by thermal reduction of GO and chemical polymerization of pyrrole, and the conductivity of $1.2 \text{ S} \cdot \text{cm}^{-1}$ corresponding to the uptake of $1.5 \text{ mg} \cdot \text{cm}^{-2}$ was obtained. Such electrode offered the capacitance of $336 \text{ F} \cdot \text{g}^{-1}$ at a current density of $0.6 \text{ mA} \cdot \text{cm}^{-2}$ [16]. Our group developed a PET/rGO/PPy composite by use of facile dip-coating of GO followed by chemical reduction and *in situ* polymerization of pyrrole. It was demonstrated that higher GO concentration leads to better conductivity and supercapacitive performance. The conductivity was measured to be $0.81 \text{ S} \cdot \text{cm}^{-1}$ associated to the uptake of $6.86 \text{ mg} \cdot \text{cm}^{-2}$ [17]. Liang *et al.* reported the maximum capacitance of $570 \text{ mF} \cdot \text{cm}^{-2}$ at $1 \text{ mA} \cdot \text{cm}^{-2}$ by an asymmetric supercapacitor of carbon nanotube (CNT)/rGO and PPy-coated cotton electrodes with mass loading of 7.7 and $5.7 \text{ mg} \cdot \text{cm}^{-2}$, respectively [18].

In the present work, firstly the surface of PET fabric is modified by alkaline hydrolysis treatment. Then, GO is coated on the modified PET and reduced to rGO by using ascorbic acid. Semi-spherical particles of PPy are further deposited on the surface through chemical oxidative polymerization method. Ultimately, structure and morphology of the composite are discussed and electrical and electrochemical responses are investigated.

II. EXPERIMENTAL

A. Materials and Reagents

The polyester fabric was provided from Yazdbaf Company with weight of $107 \text{ g} \cdot \text{m}^{-2}$, average diameter of $15 \mu\text{m}$ and plain weave. It was washed before tests by a non-ionic detergent at $60 \text{ }^\circ\text{C}$ for 1 h. Graphite flakes ($<20 \mu\text{m}$), sodium nitrate (NaNO_3), phosphorus pentoxide (P_2O_5), potassium persulfate ($\text{K}_2\text{S}_2\text{O}_8$), sulphuric acid (H_2SO_4), hydrochloric acid (HCl), hydrogen peroxide (H_2O_2), pyrrole ($\text{C}_4\text{H}_4\text{NH}$), polyvinyl alcohol ($\text{Mw}=89000\text{-}98000$, 99% hydrolyzed), ferric chloride (FeCl_3), and ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$) were supplied from Sigma-Aldrich Company.

B. Methods

B.1. Alkaline Hydrolysis of PET

In order to provide a PET surface with $-\text{COO}-$

functionalities, raw fabric was immersed in boiling 5 w/v% sodium hydroxide solution for 30 min. The sample was washed and dried at room temperature and named MPET.

B.2. GO Deposition on the MPET and the Reduction to rGO

GO powder was prepared through the modified Hummers method. The details can be found in [17]. The $5 \text{ mg} \cdot \text{mL}^{-1}$ of GO solution was prepared and the GO sheets were exfoliated by the ultrasonic radiation for 1 h. Then, MPET sample was immersed in GO solution for 1 h at $70 \text{ }^\circ\text{C}$ and it was followed by a thermal treatment at $80 \text{ }^\circ\text{C}$ for 1 h. It was repeated 5 times to increase the GO uptake by MPET. The prepared sample was named MPET/GO. Subsequently, MPET/GO was immersed in the 100 mM of ascorbic acid solution at $95 \text{ }^\circ\text{C}$ for 15 min. Afterward, the sample was washed several times and dried at $80 \text{ }^\circ\text{C}$ and named MPET/rGO.

B.3. Polypyrrole Deposition on the MPET/rGO

The 1 M of pyrrole solution was prepared and transferred to an ice-bath. The MPET/rGO was soaked in the solution and after 30 min the 0.5 M of FeCl_3 was added to the solution. The sample was taken out after 2.5 h, then washed and dried. Thus, MPET/rGO/PPy was obtained.

B.4. Washing Durability Evaluation

To investigate the washing durability of the conductive fabric, the composite was exposed to a non-ionic detergent aqueous solution for 30 min at $60 \text{ }^\circ\text{C}$ and then dried at ambient conditions. This was repeated 20 times and at the end the conductivity value of composite was measured.

C. Characterization

The percentage of weight loss ($\Delta\text{W}\%$) after the alkaline modification was defined according to Eq. (1):

$$\Delta\text{W}\% = \left[\frac{W_2 - W_1}{W_1} \right] \times 100 \quad (1)$$

Where, W_1 and W_2 are the weight of sample before and after treatment, respectively. The morphologies of the modified PET and the various composites were observed by scanning electron microscopy, SEM (FEI Quanta 200F FEG-ESEM). Fourier transform infrared spectroscopy (FTIR, Thermo Nicolet Nexus 670) was applied to characterize the functional groups of MPET as well as MPET/rGO/PPy. X-ray diffractometry (MiniFlex 600, Rigaku) was used to collect powder X-ray diffraction (XRD) patterns. The air permeability of the samples was investigated by an SDL Atlas air permeability tester. Thermogravimetric analysis (TGA) was performed in a Netzsch STA 449F1 thermobalance equipped with a SiC furnace under

N_2 gas and up to 900 °C by employing a ramp rate of 10 °C.min⁻¹. The electrical conductivity of composites was measured by using a four-point probe method in a BT-112 membrane conductivity cell (Scribner Associates, Inc.) and determined according to Eq. (2):

$$\sigma = \frac{s \times I}{E \times w \times t} \quad (2)$$

Where, σ is the electrical conductivity (S/cm), I is the applied current (mA), E is the measured voltage (mV), w is the width of the sample (1.5 cm), t is the thickness (cm), and s is the distance between the voltage probes (0.425 cm). In order to characterize the electrochemical performance of the electrodes, cyclic voltammetry (CV) measurement was conducted in three-electrode arrangement with a Gamry Reference 3000 Potentiostat/Galvanostat/ZRA in which a platinum sheet and a saturated calomel electrode served as counter and reference electrodes, respectively. The areal and specific capacitances were calculated according to Eqs. (3) and (4), respectively:

$$C_{\text{areal}} = \frac{\int IdE}{A\nu\Delta E} \quad (3)$$

$$C_g = \frac{\int IdE}{m\nu\Delta E} \quad (4)$$

Where, C_{areal} is the areal capacitance (F.cm⁻²), $\int IdE$ is the total charge integrated from CV curves (C.V.s⁻¹), ν is the scan rate (V.s⁻¹), ΔE is the potential window (V), A is the area of the device (cm²), C_g is the specific capacitance (F.g⁻¹), and m is the loading mass of electrically active materials (g).

III. RESULTS AND DISCUSSION

Fig. 1 demonstrates the synthesis process to prepare MPET/rGO/PPy. After exposing upon NaOH solution at boil, PET experiences 12% of weight loss according to Eq. (1). The surface modification of polyester with sodium hydroxide via the nucleophilic attack of the hydroxyl ions to the carbonyl groups of PET leads to the chains cleaving

and the removal of low molecular weight fragments. The more prolonged exposure time results in the more cleavage and the higher weight loss consequently the lower mechanical strength [8]. However, in this case, it was tried to reach a compromise between the fabric mechanical strength and the ability to uptake of the active materials. Moreover, the decline in the strength is further modified by the consecutive cycles of the active materials coating.

The amount of active material uptakes as well as composites thicknesses are provided in Table I. According to the table, the surface modification of PET improves notably the ability for the uptake of active materials. In the case of the untreated PET, we reported previously the mass loading of 2.88 and 3.98 mg.cm⁻² for GO and PPy, respectively [17]. These values are by far lower than those obtained in the case of MPET.

A. X-ray Diffraction

Fig. 2 shows the X-ray diffraction patterns of GO, PET, MPET/GO, and MPET/rGO/PPy. A crystalline phase is detected at $2\theta=11.5^\circ$ for the GO powder. PET shows three peaks at 17.5, 22.5, and 26° corresponding to the crystal planes of (010), (110), and (100), respectively. After coating with GO, the GO peak is detectable in the MPET/GO pattern and after the chemical reduction with vitamin C, this peak disappears and shifts towards the pristine graphite peak at higher angle and merges with the broad characteristic peaks of PET. It is owing to the decrease in the inter-planar spacing between the graphitic sheets

TABLE I
THE ACTIVE MATERIAL UPTAKE BY SAMPLES AS WELL AS
THE SAMPLE THICKNESSES

Composite	Uptake of active materials (mg.cm ⁻²)	Thickness (mm)
Untreated PET	-	0.11
MPET	-	0.11
MPET/rGO	5.89	0.13
MPET/rGO/PPy	30.01	0.17

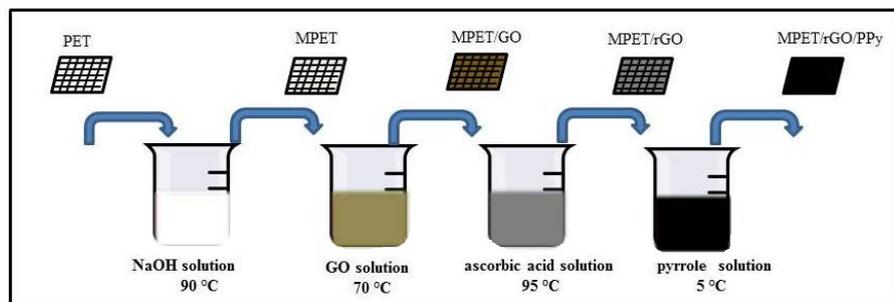


Fig. 1. Schematic representation for synthesis procedure of MPET/rGO/PPy.

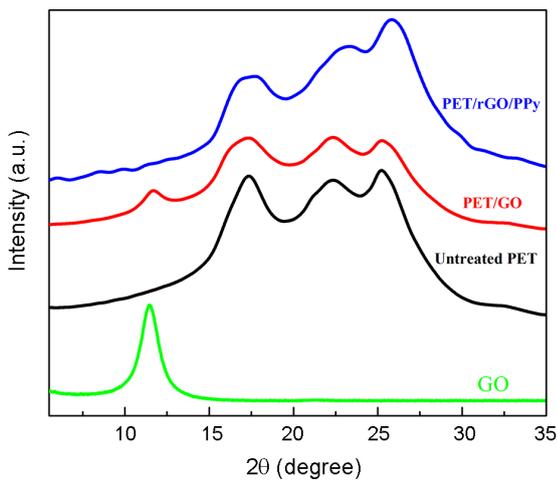


Fig. 2. XRD patterns of GO, PET, PET/GO, and PET/rGO/PPy.

caused by removal of oxygenated functional groups upon reduction of GO.

B. FTIR Spectroscopy

Fig. 3a compares the FTIR spectra of untreated PET with MPET. Initially, the IR spectra were normalized to the IR absorption value of CH_3 bond at 2920 cm^{-1} . After exposure upon NaOH solution, the intensification of hydrophilic groups such as -C=O and -OH is evident at 3432 and 1720 cm^{-1} , respectively. The stretching in the region of $3600\text{--}3200\text{ cm}^{-1}$ specifies the presence of the hydroxyl terminal groups on the surface of polyester fabric, which is caused by the interaction of the hydroxide ions with the electron-deficient carbonyl groups [19].

The FTIR study was also conducted to investigate the formation of PPy on the surface of the ternary composite of MPET/rGO/PPy. According to Fig. 3b, the characteristic bands of the bending vibration of pyrrole can be found at 790 , 1039 , and 1176 cm^{-1} . The band at 846 cm^{-1} can be related to C-H rocking. The peak at 962 cm^{-1} is assigned to the C-C in-plane ring deformation mode. The band at 1092 cm^{-1} is attributed to the $\text{N}^+\text{-H}$ in-plane deformation. The vibrations at 1238 cm^{-1} are ascribed to the C-H ring stretching. The characteristic bands of PPy for C-C and C-H ring stretching are found at 1400 and 1258 cm^{-1} , respectively. The stretching vibrations of pyrrole ring are also observed at 1541 cm^{-1} [12,16,20].

C. Air Permeability

Air permeability is determined as the air flow rate passing perpendicular through a circular area of 5 cm^2 , under a specified air pressure differential between the two surfaces of a material. These measurements were carried out to compare the space between fibers in the structure of the untreated PET and MPET. This space can be considered

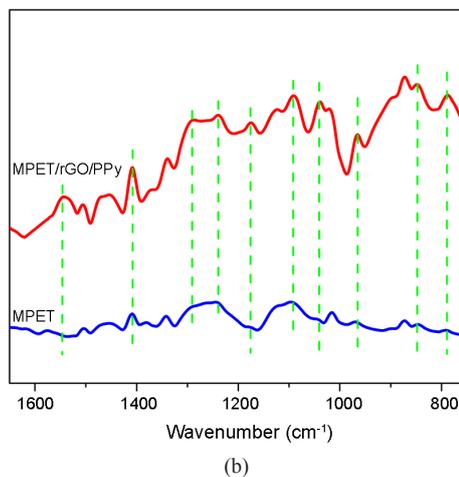
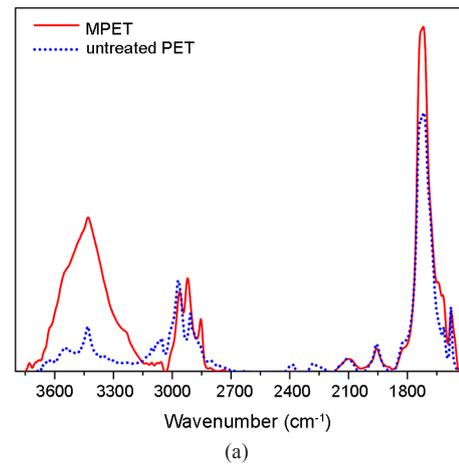


Fig. 3. FTIR spectra of: (a) PET and MPET and (b) MPET and MPET/rGO/PPy.

as a criterion for the load capacity of active materials. The air permeability of samples was measured in three random areas and the mean value was recorded according to Fig. 4. The untreated PET reaches the air permeability of

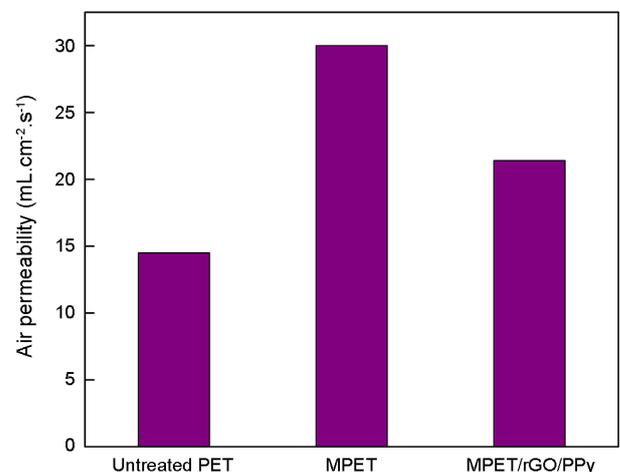


Fig. 4. Air permeability of PET and MPET.

14.5 mL.cm⁻².s⁻¹ while this value rises to 30 mL.cm⁻².s⁻¹ in the case of MPET. This suggested providing an ample space for loading of electrically active material caused by hydrolysis. Air permeability of the raw PET fabric has been boosted twofold after alkaline hydrolysis. According to the SEM images the tight structure of PET fabric has become more open as a consequence of weight reduction induced by NaOH treatment. In addition, thermal treatment during reduction process also leads to loose construction and subsequently the higher air permeability. That is a possible reason for higher air permeability of the coated fabric. However, the air permeability value decreases to 21.5 mL.cm⁻².s⁻¹ after coating of active materials.

D. Morphological Investigation

The SEM micrographs of PET, MPET, MPET/rGO, and MPET/rGO/PPy are demonstrated in Fig. 5. Polyester fibers have a smooth surface with cylindrical shape (Fig. 5a). After alkaline treatment with sodium hydroxide, the obvious pitting and also the change in the surface roughness can be observed in Fig. 5b. A good coverage on the MPET fibers is achieved after the consecutive GO deposition.

Moreover, the wrinkle and folded morphology of GO sheets is demonstrated in Figs. 5c and 5d. As shown in Figs. 5e and 5f, PPy particles grow in nanospherical shape with the diameter ranging from 54 nm to 180 nm.

E. Thermal Gravimetric Analysis

The TG curves of MPET and MPET/rGO/PPy are demonstrated in Fig. 6. The thermal degradation of MPET consists of two stages: (1) free radical formation, (2) chain fragmentation process [21]. After the deposition of rGO/PPy, the higher amount of weight left at the end of the thermal decomposition that is ascribed to the carbonaceous residue obtained from rGO and PPy [22]. The difference of the mass loss at 900 °C between MPET and MPET/rGO/PPy is nearly 28% indicating a high uptake of rGO/PPy.

F. Electrical Conductivity

Embedding the electrically active materials into the non-conductive PET introduces the electrical conduction property to the fabric. The GO deposition does not have a remarkable effect on the conductivity [17]. The reason for this is the alteration in the electronic features of the pristine

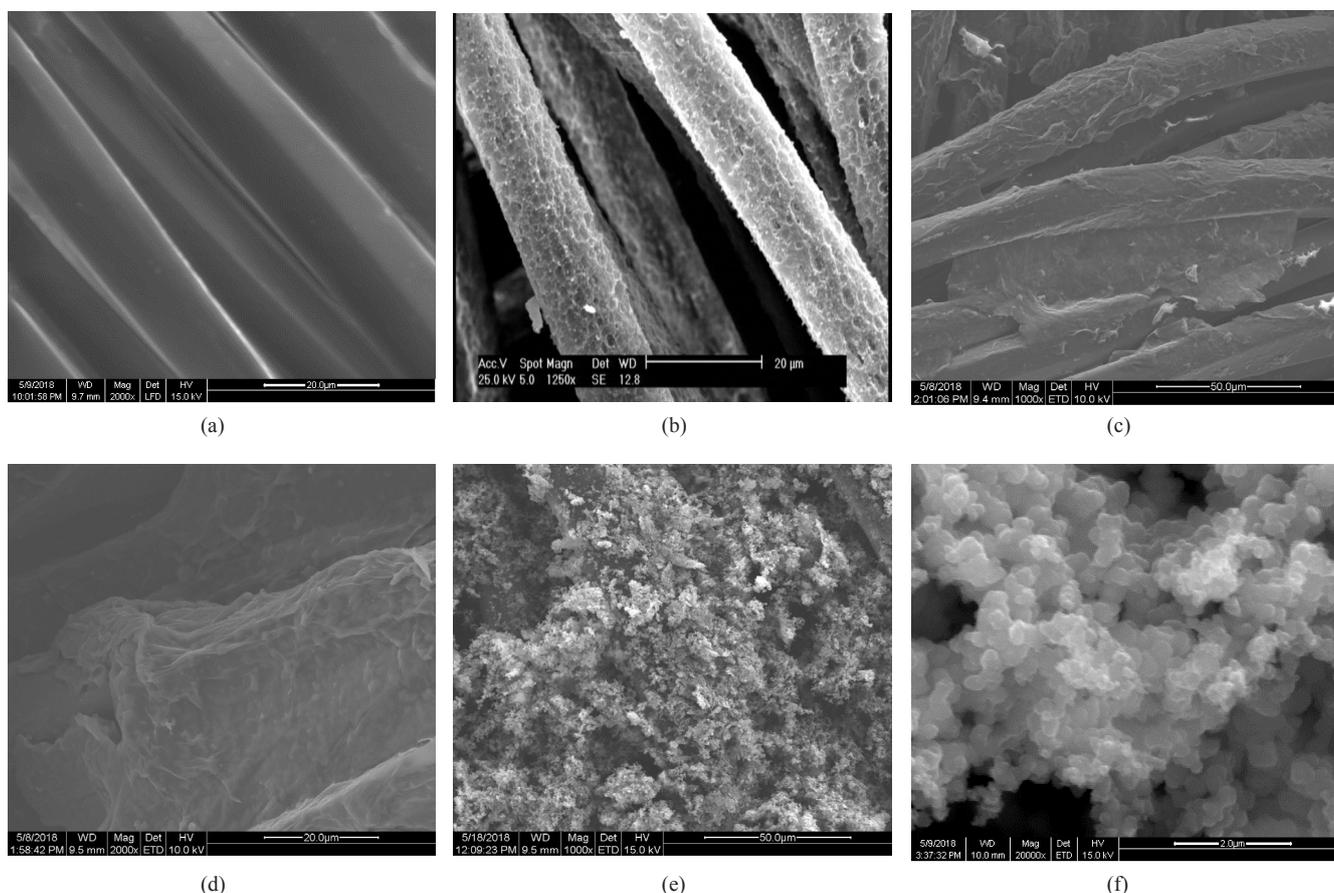


Fig. 5. SEM images of: (a) untreated PET, (b) MPET, (c,d) MPET/GO, and (e,f) MPET/rGO/PPy.

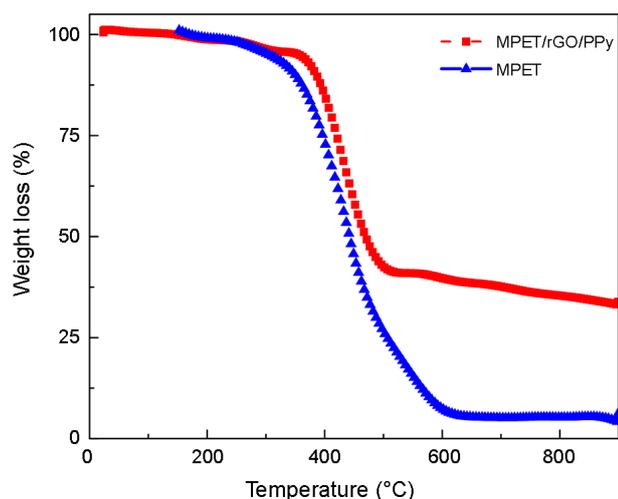


Fig. 6. TGA curves of MPET and MPET/rGO/PPy.

graphite owing to the introduction of functional groups in the sp^2 carbon layer. Hence, unlike graphite, GO has the poor conduction property. On the contrary, the reduction treatment with ascorbic acid leads to the recovery of the sp^2 hybridized carbon atoms and the improvement of the electron mobility along the graphite layers. In fact, the interaction among the 2D carbon planes is intensified after reduction process.

As indicated in Table II, the value of conductivity is observed to rise to three orders of magnitude after the growth of PPy spherical particles on the MPET/rGO. In fact, meanwhile the pyrrole is oxidized in the presence of Fe^{3+} ions, chloride ions are responsible for doping of polymer and formation of charge carriers. The reason of rising in the electrical conductivity is ascribed to the formed charge carriers as well as the π electrons of pyrrole rings [16,17].

The conductivity values of MPET/rGO and MPET/rGO/PPy are higher than those obtained for PET/rGO and PET/rGO/PPy in the same condition [17]. This is owing to the higher uptake of rGO and PPy by MPET which is an implication of surface modification.

It is worth highlighting that after 20 washing cycles, the decrease in the amount of conductivity value is negligible.

TABLE II
ELECTRICAL CONDUCTIVITY VALUE OF VARIOUS SAMPLES

Composite	Electrical conductivity ($S.cm^{-1}$)
MPET/rGO	9.3×10^{-4}
MPET/rGO/PPy	0.98
MPET/rGO/PPy after 20 washing cycles	0.90

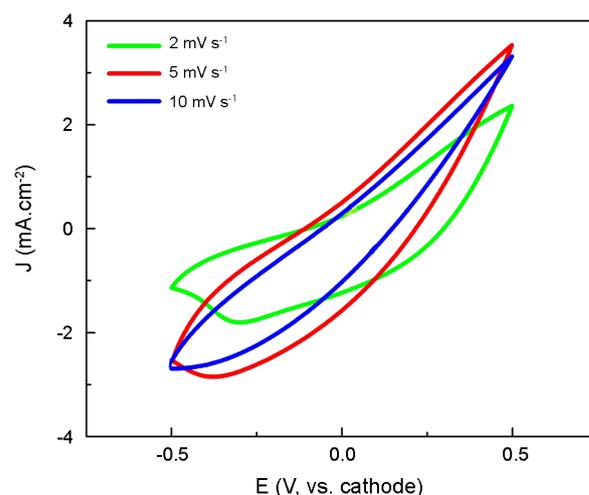


Fig. 7. Cyclic voltammogram of MPET/rGO/PPy in 1 M H_2SO_4 at different scan rates.

That is an indication of excellent washing durability of conductive fabric.

G. Electrochemical Performance

In order to investigate the electrochemical performance of MPET/rGO/PPy, the CV curves were collected in an aqueous 1 M H_2SO_4 solution at different scan rates. From Fig. 7, it can be speculated that charge transfer was conducted through the rGO/PPy network because MPET is an intrinsically insulator material. As illustrated in Fig. 7, the redox peak of PPy is evident at -0.3 V. However, as scan rate increases, the peak gradually disappears. Slow kinetics of such kind of fabric electrodes is responsible for this behavior [16,17,23]. Nevertheless, such fabric electrode delivers the specific and areal capacitances of 8300 mF.g^{-1} and 640 mF.cm^{-2} , respectively which are remarkable among textile-based electrodes [24-26]. The high specific capacitance confirms its potential as a flexible electrode.

IV. CONCLUSION

By the way of conclusion, a highly conductive PET fabric was developed by deposition of rGO/PPy. The synergistic effect of rGO and PPy has a marked influence in the performance of the composites. Meanwhile rGO provides a high surface area and stable conductive framework for PPy deposition, PPy is able to boost the charge conduction property. A good adhesion of active material was obtained due to the abundant π - π stacking between the aromatic rings. As a consequence, a negligible decrease in conductivity was observed after 20 laundry cycles. In addition the electrochemical response of the fabric in three-electrode configuration suggests the promising potential for application as flexible electrode.

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