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# Development of a Novel Preparation Method for Conductive PES Ultrafine Fibers with Self-formed Thin PES/CNTs Composite Layer by Vapor Treatment

## Treatment

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Abstract: Conductive electrospun fibers have attracted widespread interest in electromagnetic field. However, how to improve the electrical conduction of electrospun fibers effectively has still not been adequately addressed. In this work, a simple and effective new method was introduced to improve significantly the conductive properties of fibers. PES/PVA fibers with previous addition of 20 wt% PVA were chosen as a matrix due to the big parallel porous structure. The carbon nanotubes (CNTs) were firstly absorbed by the PES/PVA fibers, and then a thin polymer/CNTs composite layer was self-form on the surface of porous fibers by the vapor treatment. Most importantly, a CNTs networks structure was also formed in this vapor process, which easily made the conductive of porous fibers get a significant enhancement with a small amount of CNTs. The electrical conduction tests showed that the conduction of fiber increased with increasing CNTs content, and got the maximum value when the amounts of CNTs was about 7 wt%. The absorption time and the DMSO vapor treatment time were optimized to obtain the most excellent thin polymer/CNTs composite layer. The surface microstructure of composite layer was observed using a scanning electron microscopy (SEM) and TGA. The results showed that the novel powerful method could be potentially used to prepare novel kinds of conductive polymer fibers.

Key words: Electrospun, CNTs, conductive, vapor treatment, networks

## Introduction

Electrospinning is a straightforward and versatile way to fabricate ultrafine fibers down to micro-/nanometer scale. So far as we know, this is the only universal method that could be applied to produce nanofibers with broad materials from polymers to hybrids to inorganics, and thus it has attracted much more attention in the recently years<sup>1,2</sup>. By virtue of a high surface area to mass ratio<sup>3,4,5</sup>, electrospun ultrafine fibers are of potential importance to technological applications such as catalytic carrier, energy-storage system, tissue engineering, wound dressing, sensor, drug deliver, and so on. Especially, conductive electrospun ultrafine<sup>6</sup> fibers exhibit extra excellent electrical conductivity, thermal conductivity, electromagnetic shielding and absorption properties, and can be widely applied in electronics, conductive clothing, electromagnetic shield, etc.

At present, the traditional way to make electrical conductive ultrafine fibers was directly electrospinning electrical conductive polymer e.g. polyacetylene<sup>7</sup>, polypyrrole<sup>8</sup>, polyaniline<sup>9</sup>, and so on. Fibers produced by this method had good conductivities and the preparation step was relatively simple. However, most the known polymers were not electrical conductive polymer, so it was more urgent to improve the electrical conductivity. However, this method was not appropriate to such kinds of polymer ultrafine fibers. Much research has been done on this topic. Up to now, two approaches were used to improve the electrical conductivity of electrospun non-electrical conductive polymer fibers. One was high temperature carbonization, which resulted in the formation of carbon atoms in the macromolecules main chains and then improved their electrical conductivity properties significantly. The best example was the carbon nanofibers prepared by the carbonization of electrospun polyacrylonitrile (PAN) nanofibers<sup>13,14,15</sup>. Fibers produced by this method had good conductivities but the cost of preparation was high and the preparation step was relatively complex. The other way was adding conductive additives in the electrospun solution to prepare composite ultrafine

fibers<sup>16,17</sup>, such as carbon nanopowders<sup>18,19</sup>, carbon nanotubes<sup>20,21,22</sup>, metal and so on. This method was the universal method to improve the conductivity of electrospun fibers. However, for application in different kinds of polymer, the amount of conductive additives was high. According to previous reports<sup>23, 24</sup>, the electrical conductivity of fiber would be improved only with a mass of CNTs (40%) in this method. Large amounts of conductive additives would not only cause the cost of preparation increase but also make the weight of fibers increase which losing the advantage of lightweight. Above all, most CNTs dispersed in the matrix, only little CNTs could connected with each other to make contribution to the conductive of fibers. All risk factors would slow down the development of this method, only 20 publications reported on this method by the end of 2014. Therefore, it was necessary and interesting to develop a novel method to improve the electrical conductivity of electrospun ultrafine fibers, which was easier and also needs less amounts of additives to obtain high electric conductivity than the reported methods.

In this work, polyethersulfone (PES) and PES/PVA porous ultrafine fibers were prepared via electrospinning firstly. Then they would be used as the matrix materials to adsorb the electrical conductive additives material of CNTs. CNTs will fall off because of the weak non-covalent interaction between the electrical conductivity additives and the fiber matrix. To avoid this, vapor treatment was used to make CNTs anchored. In this way, little amounts of CNTs connected with each other and formed the net-work on the surface of fibers only to improve the electric conductivity high-efficiency. At the same time, vapor treatment was simple and universal to most polymer. The suitable parameters such as porous structure for adsorption, solvent vapor treatment parameters were investigated. Finally, after the preparation of the electrical conductive fibers, the electrical conductivity of the fibers with different amount of CNTs was addressed.

## Experimental

### Materials

Polyethersulfone (PES, Ultrason E 6020P, CAS No.25608-63-3) was purchased from BASF Chemical Company (Germany). Polyvinyl alcoholvinylalcoholpolymer (PVA, Mw=30000 g/mol, alcoholysis=88%) was obtained from SC Weinilun Co., China. Dimethyl sulfoxide (DMSO, analytical, 99+%) and ethanol were obtained from Chengdu Chemical Reagent Co. Ltd. (China). Carbon nano-tubes (CNTS, TNIM4, the diameter of inner was 5-10 nm, the diameter of outer was 10-30 nm, and the length was 10-30 microns) were purchased from the Chengdu Organic Research Institute of Chinese Academy of Science. All the chemicals used were of analytical grade and without further purification unless they were described specially. Distilled water passed through ion-exchange columns was used throughout all the studies.

### Preparation of PES-based porous ultrafine fibers

PES with and without PVA were dissolved in DMSO at 60°C until the solution became homogeneous. The solutions were still pellucid after 24 h. The concentration of PES in PES/DMSO solution was 20.3% w/v and the content of PVA was 0, 10, and 20 wt%. For electrospinning the solutions, a direct current high voltage generator (ZGF ChuanGao Electro-tech Inc, China) was used to provide a voltage of 15 kV. The solution was placed in a 50-mL syringe, to which a needle tip of 0.5 mm inner diameter was attached. The positive electrode of high-voltage power supply was connected to the needle. Grounded rotating drum was used as a collector and the speed of drum was 0.6 m/s. The distance between the tip and the collector was maintained at 16 cm. The relative humidity was ranging from 71 % to 76 % without special instruction. The extraction of the PVA molecules from as-spun PES/PVA blend

ultrafine fibers matrix was carried out by washing them with distilled water for several days at 90 °C.

### **Preparation of conductive PES-based porous fibers**

Dispersion of different contents CNTs in the ethanol with the ultrasonic technology (KQ-500, KunShanUltrasonicInstrumentsCo.,Ltd) firstly. Then PES-based porous ultrafine fibers were immersed in the previous suspension to absorb large number of CNTs by the ultrasonic technology for 2 h. After drying the samples, the PES-based porous ultrafine fibers with large amount of CNTs were post-treated by the vapor of DMSO with the concentration of  $10^{-2}$  L/L for certain time for making the CNTs being attached on the surface of the fibers. The removal of un-immobilized CNTs from porous ultrafine fibers matrix was also carried out by washing them with ethanol by ultrasonic technology for 2 h.

### **Characterization**

The morphologies of PES-based porous ultrafine fibers were observed with scanning electron microscopy (SEM and EDS Inspector F, FEI Company, all operation at an accelerating voltage of 20 kV). The samples were coated by an E-1045 ion sputter coater with Au/Pd to reduce charging.

### **Thermal gravimetric analysis**

Thermogravimetric analysis of the PES-based porous ultrafine fibers was carried out by using thermal gravimetric analysis (TGA/TA Q600, TA Instruments Co., USA) under a nitrogen atmosphere from 50 to 800°C at a heating rate of 10 °C/min.

The real content of CNTs in the PES-based porous ultrafine fibers samples were estimated by using the following formula:

$$\omega_c = C_c - C_B$$

Where  $C_c$  is the weight loss percentage of PES-based surface composite ultrafine fibers;  $C_B$  is the weight loss percentage of PES-based ultrafine fibers;  $\omega_c$  is the content of CNTs in the PES-based porous ultrafine fibers.

The thermal properties of the PES-based porous ultrafine fibers with and without CNTs were explored by differential scanning calorimetry (DSC). DSC experiments were performed on a Netzsch DSC 200 PC thermal analysis instrument (Netzsch, Selb, Germany). All experiments were conducted in the temperature ranging from 30 to 300 °C with a constant heating and cooling rate of 10 K·min<sup>-1</sup> and a waiting period of 2 minutes at the minimum temperature. Glass transition temperatures ( $T_g$ ) were determined from the first heating run.

### **The conductivity analysis**

Conductivity analysis of the PES-based surface composite ultrafine fibers with different CNTs content was carried out by using low DC desistance (Changzhou Tonghuicompany, Jiangsu, China) under an air atmosphere. Each sample was tested for 10 times to get the mean value under the condition of electric current 1 μA.

### **Results and Discussion**

In this work, PES-based porous ultrafine fibers were chosen for the adsorption of CNTs because of the high surface area and the porous structure. For comparison, PES pure and PES/PVA porous ultrafine fibers were prepared via electrospinning technique for the further use of adsorption. Figure 1 shows the SEM images of pure PES and PES/PVA porous ultrafine fibers. It indicated that the surface and the internal structure of both ultrafine fibers were porous. Pure PES porous ultrafine fibers displayed a smaller, denser holes for the whole

fiber matrix (Fig. 1a and b), which caused by the rapid solvent evaporation during the electrospinning processing. Differently, the larger and parallel porous structures were clearly observed for the surface and inner structure of PES/PVA porous ultrafine fibers. The porous structure formation mechanism might be the poor solubility of PVA in DMSO. During the electrospinning, the phase separation of PVA preferentially occurred with the instantly increasing of solution concentration caused by the rapid solvent evaporation. The PVA micro-phase region formed by the phase separation will be stretched and oriented during rapid spinning jet extension. Therefore, after the removing of the PVA, the big and parallel porous structure was formed in the whole fiber matrix.

**Fig. 1**

Thereafter, the CNTs absorption capacity of pure PES and PES/PVA porous fibers were investigated. Three kinds of porous fibers, PES porous fibers, PES/PVA porous fibers prepared with previous addition amounts of 10wt% and 20wt% PVA, were prepared as the CNTs adsorbing medium in this work. As seen in Fig. 2a, quite small amounts of CNTs could be observed on the surface of pure PES porous fibers. Compared with the pure PES porous fibers, there were more CNTs absorbed on the surface of the PES/PVA porous fibers prepared with previous addition amounts of 10wt% PVA. It indicated the bigger pores would own a better compared with the smaller porous structure. Further, increasing the amount of PVA to 20 wt%, much more CNTs were absorbed on the surface as a more and bigger porous structure was formed. This suggests a better possibility to form a continuous conductive surface layer for the big and parallel porous structure. In this case, the PES/PVA porous fibers prepared with



previous addition amounts of 20wt% PVA were considered to be the most suitable fibers to absorb the CNTs in this work and were used as the matrix porous fibers in all the following work.

### Fig. 2

To immobilize the CNTs on the surface of fibers, the DMSO vapor treatment was applied. The DMSO vapor could slightly dissolve the polymer on the surface of the fibers. Because of the surface tension of the dissolved fibers surface, the porous structure on the fibers surface would be closed and the CNTs were anchored on the surface porous structure. The vapor treatment time would have a big influence on the formation of the conductive surface layer. The PES-based porous fibers would be destroyed if the treatment time was too long. While if the treatment time was not long enough, the CNTs would not be effectively embedded and immobilized. Figures 3 shows the SEM images of DMSO vapor treated PES/PVA/CNTs porous ultrafine fibers for 0, 1.5, and 2 hours, respectively. The results showed that with the increasing time of vapor treatment, less amounts of the CNTs on the surface of fibers could be observed and meantime the surface porous structure was disappeared. Because there was only few CNTs on the surface of fibers after the 2h's vapor treatment, the maxim vapor treatment time was considered as 2h in this work. There would be two possible reasons for the phenomenon. One is that the CNTs were rinsed off from the surface of the fibers after the porous structure was closed. While, the other one is that the CNTs was embedded into the surface layer and formed a thin conductive layer around the fibers.

### Fig. 3

As shown in Fig. 4, there were almost no CNTs on the internal of fibers and only some ones could be observed on the surface, it indicated that the un-immobilized CNTs on the fibers mats was removed by ethanol washing process. The cross section SEM images of the PES/PVA porous fibers displayed that there was no CNTs in the inner porous structure, while in the surface layer some CNTs could be observed. Therefore, this result indicated that when the surface porous structure was closed, some CNTs were also enclosed into the porous structure.

**Fig. 4**

The TGA results could further proved this result. As seen in Fig. 5, CNTs is not degraded at the N<sub>2</sub> atmosphere until 800°C. For both PES/PVA porous ultrafine fibers and PES/PVA porous ultrafine fibers with the surface absorption of CNTs, there were two regions in their weigh lost trace. The first weight loss is attributed to the decomposition of PVA while the second weight loss is that of PES, which showed that there was still some PVA left in the fiber matrix. The decomposition temperatures of PES component and PVA component were 495.8 °C and 220.5 °C, respectively. With the addition of CNTs, the thermal stability of electrospun porous PES/PVA porous ultrafine fibers increased. The start decomposition temperatures of the PES/PVA porous fibers was about 150 °C while it increased to 260 °C for the fibers after the surface adsorption of CNTs. Based on equation 1, the content of CNTs in the PEA/PVA porous fibers was about 11 wt%. Based on the SEM characterization results, there were quite few CNTs on the surface and no ones in the inner of the fibers. Therefore, in this case, the only place

for the presence of CNTs was the surface layer of the fibers, which made the conductive layer on the surface of fibers and could improve the conductivity properties of fibers with the very less amounts of CNTs.

### Fig. 5

For easily understand of the whole procedure, the scheme was provided as Fig. 6. The CNTs were absorbed on the surface and surface porous structure. After the vapor treatment, the porous structure on the surface of fibers would disappear and thus the CNTs were embedded, forming a thin polymer/CNTs composite layer around the ultrafine fibers. During electrospinning process the fibers already formed interweaved structures, the thin conductive composite layer could improve the conductivity of the fiber mats significantly.

### Fig. 6

As can be seen in Table 1 and Fig. 7, with the content of the CNTs increasing, the surface resistance of fibers mats reduced significantly at the beginning, which meant the obviously improvement for the conductive properties of PES/PVA porous ultrafine fibers. When the concentration of suspension of CNTs reached  $3.0 \times 10^{-3}$  g/L, the surface resistance decreased to the  $10.98 \text{R}/\square$  and further increase the concentration the surface resistance was not changed. It suggests that the formation of CNTs network in the thin composite layer was fulfilled at this concentration. At this time, the CNTs were forming the networks to make electric charge move freely. With further increasing the concentration, the content of CNTs in conductive thin

composite layer would be increased. While the additional content offers no contribution to the improvement of mats' conductivities. As Table 2 shown, the TGA results of PES/PVA porous ultrafine fibers with different contents of CNTs are different. The TGA results showed that the real content of the CNTs in the PES/PVA porous ultrafine fibers mat was about 7 wt% when the concentration of suspension of CNTs was  $3.0 \times 10^{-3}$  g/L. It meant that only a content of CNTs of 7 wt% was required for the formation the fulfilled continuous conductive layer. Therefore, a low content of CNTs could result in a good conductivity for the fibers mats.

**Table. 1**

**Fig. 7**

**Table. 2**

The influence of surface polymer/CNTs composite layer on thermal properties of PES-based ultrafine fibers was assessed by DSC investigations. As displayed in Figure 7, no melting peak but only the glass transition region could be observed in DSC heating curves for all of PES-based porous fibers, which indicated that the PES was an amorphous polymer. The glass transition temperatures ( $T_g$ ) determined for PES-based porous ultrafine fibers with different surface CNTs content were ranged from 228-231 °C. All of them were larger than that of the pure PES ultrafine fibers (220 °C). It might be due to the restrictive movement of macromolecular chains by incorporating of CNTs in the fibers surface layer.

**Fig. 8**

**Table 3****Conclusion**

In this work, PES and PES/PVA porous fibers were prepared via electrospinning technique. The SEM characterization results showed that both the internal and surface structure were porous. Then they were applied to adsorb the CNTs in the ethanol. The results indicated that because of the big and parallel porous structure PES/PVA fibers with PVA addition amounts of 20wt%, it was more suitable to absorb CNTs. The DMSO vapor was applied to close the surface porous structure and immobilized the CNTs and the vapor treatment time of 2h was chosen as the optimal time. After the vapor treatment and clean of ethanol, the thin polymer/CNTs composite layer was self-formed on the surface of PES-based ultrafine fibers. It was proved by further SEM characterization and TGA results. The conductive properties test showed that only 7 wt% CNTs could form the continuous conductive layer and significantly increase the conductivity. It will be no further enhancement of conductive properties for further increase of the amount of CNTs. The results showed that this method was available and could be used to prepare other kinds of conductive polymer fibers.

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### Table Captions

**Table 1** The different concentration of CNTs in ethanol used for the adsorption

**Table 2** The TGA results of PES/PVA porous ultrafine fibers with different CNTs concentration 0a),  $0.03 \times 10^{-2} \text{g/L}$ (b),  $0.2 \times 10^{-2} \text{g/L}$ (c),  $0.3 \times 10^{-2} \text{g/L}$ (d),  $0.6 \times 10^{-2} \text{g/L}$ (e),  $2.4 \times 10^{-2} \text{g/L}$ (f) in ethanol

**Table 3** The DSC data determined from the DSC curves



## Figure Captions

**Figure 1** SEM image of the surface (a) and cross section structure (b) of pure PES porous fibers and the surface (c) and cross section (d) of PES/PVA porous fibers. The Magnification of those images is 50,000.

**Figure 2** SEM image of pure PES porous fibers (a), PES/PVA(10/1, wt/wt) porous fibers (b), and PES/PVA(10/2, wt/wt) porous fibers (c) after the CNTs adsorption. Magnification of all of the images is 50,000.

**Figure 3** SEM images of DMSO vapor treatment of PES/PVA porous fibers after the absorption of CNTs with time of 0, 1.5, and 2h. The magnification of all the images is 50 000.

**Figure 4** SEM images of the top-view (a) and cross section (b) of PES/PVA porous fibers containing CNTs with the vapor treatment and then after rinsed by ethanol. (Please move a and b to the left side)

**Figure 5** TGA curves of PES/PVA porous fibers (a) and PES/PVA porous fibers together with CNTs (b).

**Figure 6** The scheme diagram of the preparation of conductive fibers for the whole procedure.

**Figure 7** The electrical conductivity of PES/PVA porous ultrafine fibers with different contents of CNTs.

**Figure 8** The DSC curves of PES based with different contents of CNTs 0%(a), 4.08%(b), 5.56%(c), 6.99%(d), 12.69%(e), 15.00%(f).

Table 1

NO.	0#	1#	2#	3#	4#	5#
$C(\text{ g/L}) * 10^{-2}$	0	0.03	0.2	0.3	0.6	2.4

Table 2

<b>Samples</b>	<b>a</b>	<b>b</b>	<b>c</b>	<b>d</b>	<b>e</b>	<b>f</b>
<b>Content(%)</b>	0	4.08	5.56	6.99	12.69	15.00

Table 3

<b>No.1</b>	<b>a</b>	<b>b</b>	<b>c</b>	<b>d</b>	<b>e</b>	<b>f</b>
<b><math>T_g</math>(°C)</b>	220	229	230	231	231	231

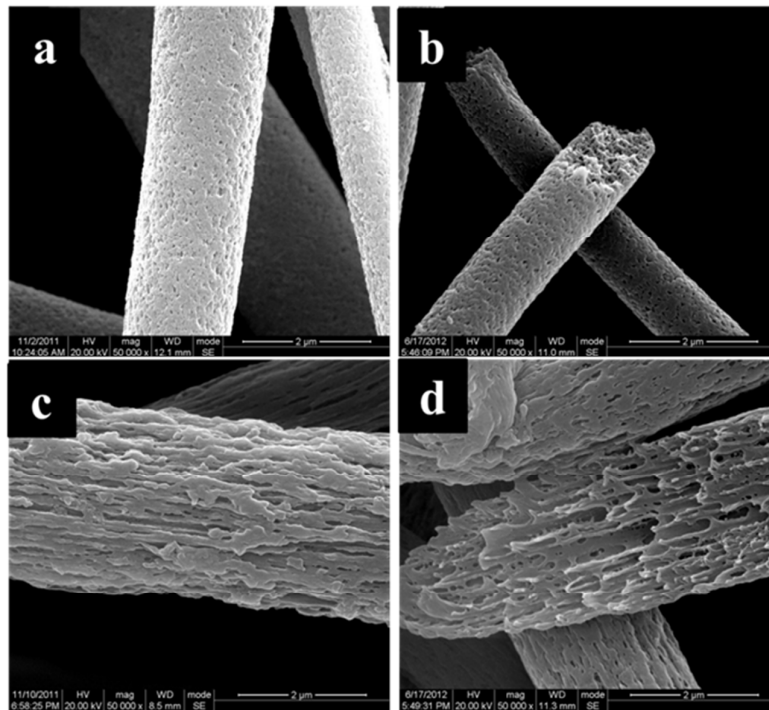


Fig. 1

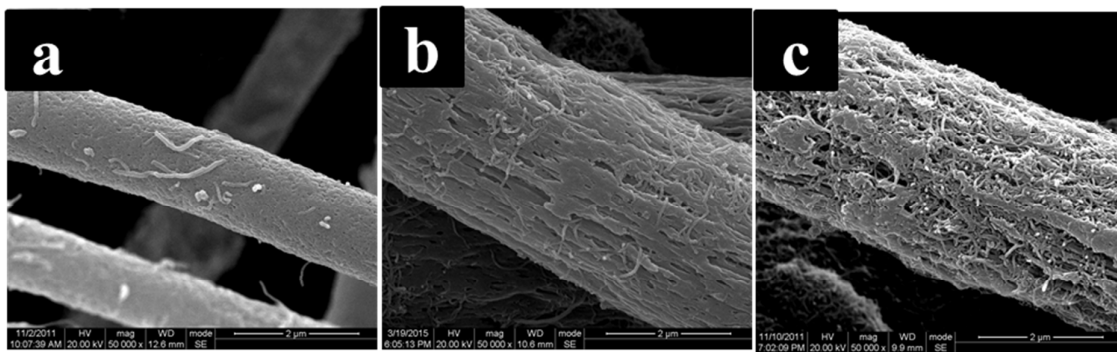


Fig. 2

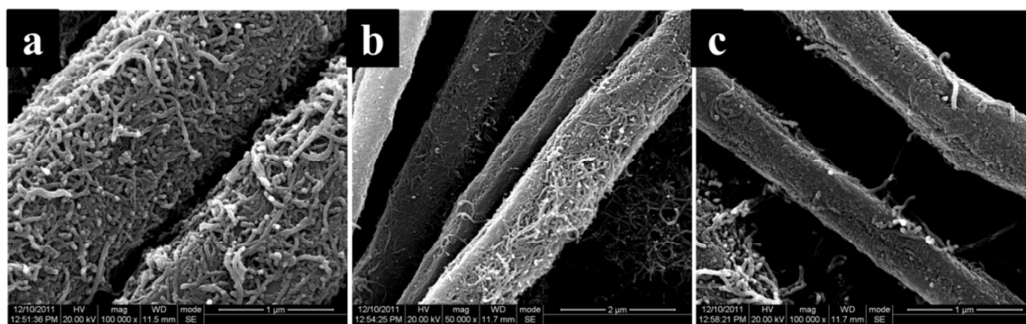


Fig. 3

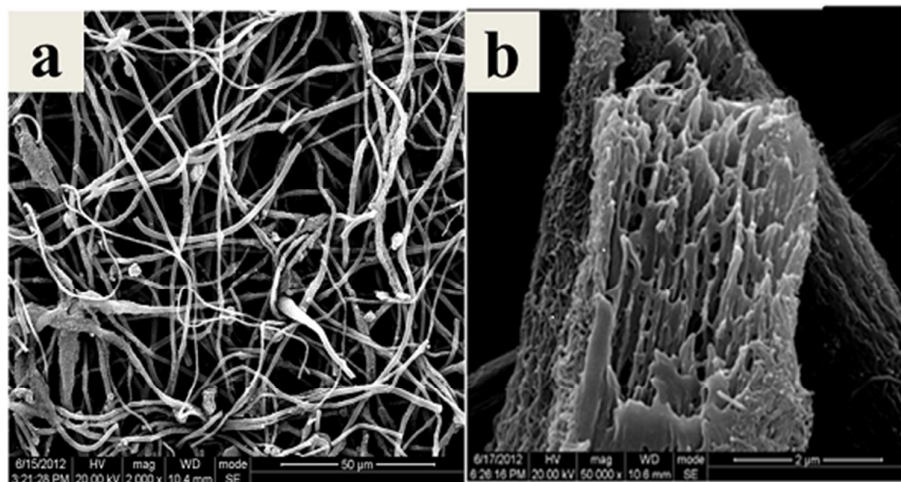


Fig. 4



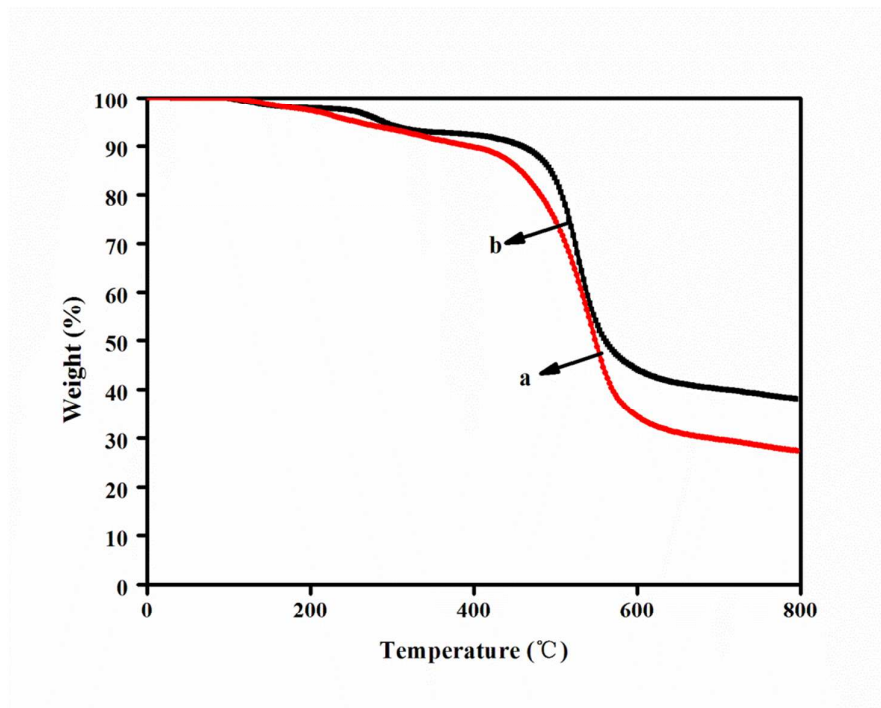


Fig. 5

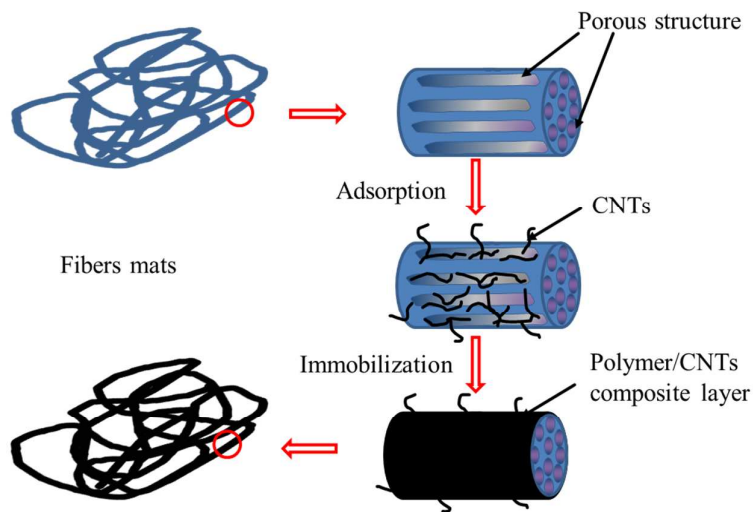


Fig. 6

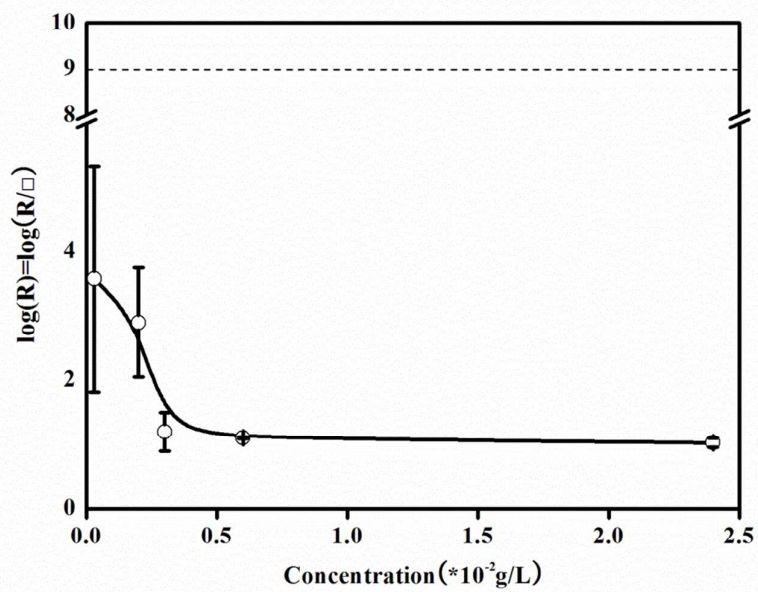


Fig. 7

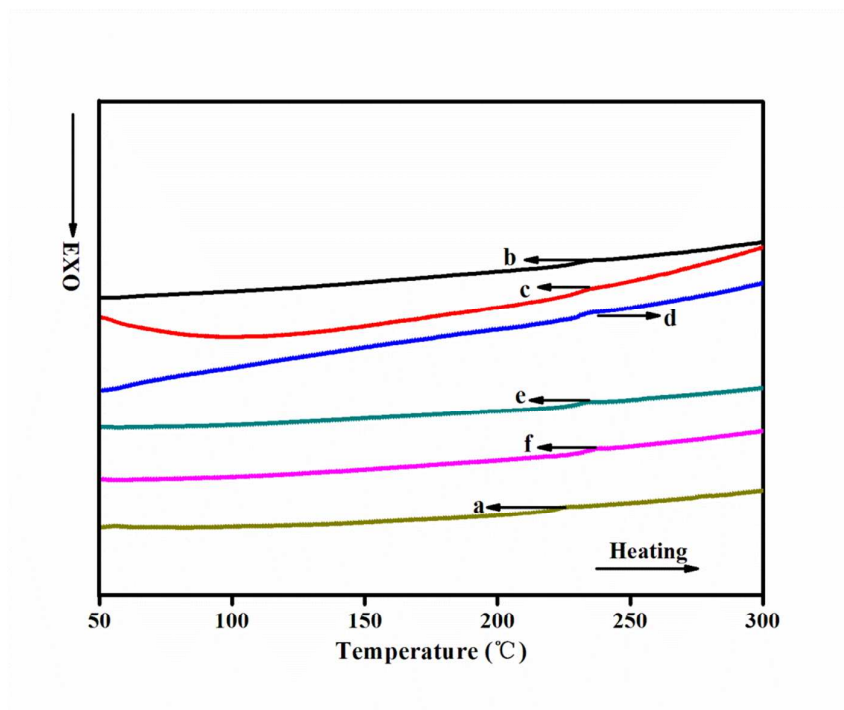


Fig. 8