Scientia Iranica F (2015) 22(6), 2757–2765

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Sharif University of Technology
Scientia Iranica
Transactions F: Nanotechnology
www.scientiairanica.com

Evaluation of the influence of cadmium salt addition on iodinated poly (vinyl alcohol)-derived carbon nanofibers

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Received 21 February 2015; received in revised form 28 April 2015; accepted 25 July 2015

KEYWORDS
Carbon nanofiber;
Electrospinning;
Iodine treatment;
Carbonization;
Cadmium salt.

Abstract. Electrospinning of polyvinyl alcohol (PVA) solution followed by a 24 h iodination treatment for stabilization of PVA for carbonization yields carbon nanofibers. When cadmium acetate dehydrate [$(CH_3COO)_2Cd.2H_2O$] salt is added to the PVA spinning solution, the carbon nanofibers derived from a iodinated PVA and cadmium/PVA nanofibers precursor could increase the carbon yield to 30% and 34% after carbonization which is much higher than the yield (< 1%) for untreated spinning solution. Several techniques, such as TGA, FE-SEM, XRD, Raman spectroscopy, and HR-TEM, are utilized for characterization of carbon nanofibers. The size of crystalline nanofibers is in the range of 100-350 nm.

1. Introduction

Recently, one-Dimensional (1D) nanostructured carbon materials with different forms and compositions were researched intensively due to their unique structures, functionalities, and many intriguing applications. Continuous carbon nanofibers (CNFs) are one example of such a structure [1]. Although the most common methods for synthesizing CNF are thermal treatment of polymeric fibers and Chemical Vapor Deposition (CVD), one of the promising methods of fiber preparation is polymer electrospinning and subsequent thermal treatments, because this combination offers easier processing and lower cost and is environmentally benign [1,2]. These fabricated CNFs present unique characteristics, such as extremely long fiber length, high surface area, and complex porous structure [3,4]. Various typical polymers have been utilized as a precursor in CNFs preparation, some of which are poly(ethylene oxide), poly(vinyl alcohol), poly(aniline), pitch, poly(amic acid), poly(p-xylene tetrahydrothiophenium chloride), polyimide, polybenzimidazole, poly(furfuryl alcohol), poly(vinylidine chloride), polyimide, poly(acrylonitrile), phenol Resin, and poly(vinylidene fluoride) [2,5].

In order to produce CNFs from polymeric nanofibers, they should be stabilized and subsequently carbonized. Hence, carbon yield of precursor nanofibers is a crucial factor when synthesizing CNFs. The stabilization process converts the thermoplastic precursor to highly condensed thermosetting fibers through intricate chemical and physical reactions such as dehydrogenation, cyclization, and polymerization. During the carbonization process, non-carbon elements

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can be eliminated by pyrolysis and dehydration, leading to formation of micro porosity [6,7]. Poly (vinyl alcohol), a member of vinyl polymers, has been used to produce carbon materials due to its hyperfine, which splits the hydroxyl groups in the polymer chain. Also it has high carbon content (ca. 54.5%), excellent chemical and physical properties, non-toxicity, process ability, good chemical resistance, wide range of crystallinity, and complete biodegradability. Among all merits, there exists one problem, which is the decomposition of PVA at temperatures slightly higher than its melting point. As a result, a large mass loss will occur under direct carbonization in air condition. Presence of PVA precursor with high thermal stability, therefore, is vital so as to obtain a high carbon yield. Sashio and Bin [8,9] reported that the treatment with chemical agents has a capability to improve its stabilization. In addition, Fatema et al. [2] shows that iodine acts as an appropriate stabilizer of PVA and boosts dehydrogenate polymerization during carbonization. The single bond hydrocarbon structure converts to a double bond polyene-like structure in PVA through iodine treatment, making PVA molecules difficult to melt, even at the relatively high temperature of carbonization. The analogous impacts of the iodine treatment have been observed in several other carbon precursors, some of which are polyvinyl alcohol, polyacetylene, mesophase pitch, silk fibroin, and wood [10].

Another method to prevent PVA nanofibers from rapid melting is application of transition metal salt [11]. Use of metal salts can not only lower the carbonization temperature, but also increases the carbon yields [12]. Cases in point, chromium nitrate and Fe $(AC)_2$ produce finer nanofibers from PVA. Also, iron metal salts are used to enhance PVA films graphitization at relatively lower temperatures [11,13]. It is reported that addition of small amounts of Ni metal catalyst improves PVA dehydrogenation during carbonization and appears as nanoparticles on the surface of nanofibers, producing unique properties in CNFs [2].

The present work investigates the effects of iodine treatment on electrospun PVA nanofibers and the effects of cadmium acetate dehydrate addition to the PVA spinning solution, aiming to improve the carbon yield and morphology of iodinated PVA-derived carbon nanofibers. Several characterization tools, such as XRD, FTIR, TGA, FESEM, HR-TEM, and Raman spectroscopy, were employed to characterize the chemical composition, phase structure, and morphology of resultant CNF.

2. Experimental

Polyvinyl alcohol (87-89% hydrolyzed, Mw = 13,000-23,000, Sigma-Aldrich), $Cd(CH_3COO)_2.2H_2O$ (Mw = 266.52, Merck), pure iodine (Mw = 253.81, Acros), and distilled water were used as the precursors without further purifications. For preparing 8 wt% PVA solution, PVA was dissolved in distilled water at 80° C for 15 h. In the case of metal salts incorporation, cadmium acetate dehydrate was added to PVA solution and mixed thoroughly to obtain homogeneous viscose gel. Then, the solution was drawn into a syringe for the electrospinning procedure.

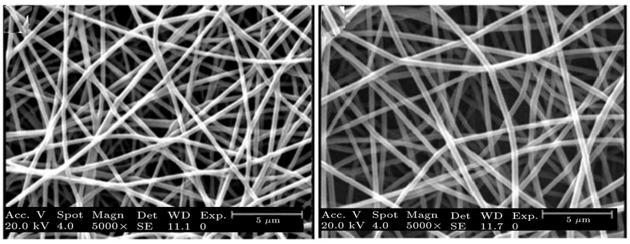
In the electrospinning process, a high electric potential in the range of 18-24 KV was applied to a droplet of PVA precursor solution at the tip of a syringe needle with 0.4 mm inner diameter. A syringe pump was used to provide a constant flow of PVA precursor solution. The electrospun nanofibers were collected on a target plate (aluminum foil), which was placed at a distance of 9-12 cm from the syringe tip. The output of the injection pump was between 6-12 μlit/min. A charged jet was formed and ejected towards the applied field. The evaporation of solvent from the PVA precursor solution (8 wt%) jet took place as the jet moved towards the collector and the fine fibers were collected on the grounded target. According to our previous studies, the Response Surface Methodology (RSM) was used for planning and optimizing the electrospinning process. These optimum conditions are as follow: concentration = 8 wt%, distance = 11 cm, voltage = 21 kV and flow rate = 9 μlit/min [14]. The PVA nanofiber mat was enclosed with an adequate amount of solid iodine in a glass vessel. The sealed vessel was placed in oven at 80°C for 24 h, in which iodine was vaporized and the nanofibers were dehydrated. Iodinated PVA and PVA-Cd nanofibers called PVA-I and PVA-Cd-I nanofibers. A ceramic tubular furnace (AMF-9P-III THV, Asahi Rika Seisakujo, Japan) was used for the carbonization process. The electrospun PVA-based nanofibers were carbonized in the furnace under argon atmosphere at a temperature in the range of 700-1100°C for 2 h. The heating rate was 5°C/min. Thermogravimetric analysis (TGA) was carried out with a gakuThermoplus II TG 8120 under argon atmosphere from room temperature to 1000°C at a heating rate of 10°C/min. The morphologies of the nanofibers were observed with a SEM (Philips XL-30 (Germany)) and FESEM (TESCAN, Czech). The diameter of the nanofibers was analyzed from SEM images using measurement software. The experimental results of nanofiber diameter represent the average of 40 individual measurements at different places. Raman spectra were recorded in the range of 100-4200 cm^{-1} by using an Almega Thermo Nicolet Dispersive Raman Spectrometer, UK, Nd: YAG laser (λ = 532 nm) with 32 scans, generated at 100 mW. XRD patterns were recorded at room temperature with an X PERT-PRO powder diffractometer system, PAN Alytical-Netherlands. TEM analysis was performed by Philips, EM 208 model.

3. Results and discussion

3.1. Characterization of PVA and PVA-I nanofibers

Figure 1(a) and (b) show the SEM images of as-spun PVA fibers. As can be seen, uniform and bead free fibers were obtained at concentrations above 6%wt. Some conditions, which used the lowest PVA concentration and the greatest spinning distance, produced too many droplets by electrospraying instead of electrospinning. With increasing PVA solution concentration to 12%wt, electrospinning was found to be difficult due to sticking of the solution at the nozzle tip.

The TGA curves of PVA and PVA-I nanofibers in Figure 2 show that PVA nanofiber begins to degrade and a slight weight loss occurs from about 95°C to 120°C due to the evaporation of water and volatile components. A drastic weight loss between 300°C and 470°C is associated with the decomposition of the main polymer chains and most of the PVA nanofibers noticeably degrade into gases, such as CO and CO_2, by thermal decomposition. At the last step of destruction and decomposition, the weight loss rate slowed down at above 470°C and residual weight percentage reached less than 1% at 500°C. On the other hand, the thermal decomposition behavior of PVA nanofibers is considerably influenced by iodine treatment. For PVA-I nanofiber, the weight loss was nearly constant to 130°C and the sharp thermal decomposition started at around 130°C. The initial weight loss was mainly attributed to vaporization of the iodine component in the nanofiber structure. The slope of TG curve became steeper at above 270°C – where the cross linking occurred – due to thermal degradation of PVA nanofibers accompanied by oxidation reaction by iodine component. The weight loss in the range of 320-450°C was due to the hydration under carbonization. The yield obtained at 500°C for iodinated nanofibers was ca. 30 wt% under dynamic heating condition. The TGA results showed that the incorporation of iodine into PVA nanofibers played a significant role as a catalyst in the dehydration of alcohols and caused the intermolecular cross linking of PVA molecules by iodination, which formed poly-n type molecules in nanofiber structures that improved thermal stability and gave higher yield of the iodinated nanofibers than the PVA nanofibers [15].



(a) (b)

Figure 1. PVA nanofibers produced at (a) concentration = 6 wt%, voltage = 15 kV, distance = 11, and rate = 9 μlit/min, and (b) concentration = 8 wt%, voltage = 21 kV, distance = 11 and rate = 9 μlit/min.

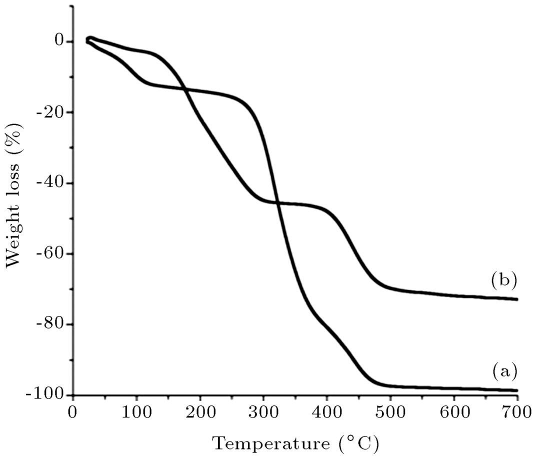
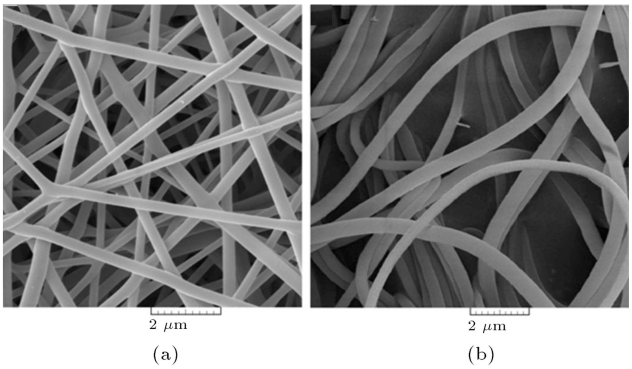


Figure 2. TGA thermographs of (a) PVA nanofiber, and (b) PVA-I nanofiber.

Figure 3 shows the FESEM images of PVA and PVA-I nanofibers before carbonization process. As can be seen in Figure 3(b), there is a noticeable deformation in iodinated nanofiber due to the rising temperature in iodine treatment process. Also, the absorption of iodine molecules into the structure of nanofibers leads to microscopic enlargement of the PVA nanofibers. The diameter of iodinated nanofibers (350 ± 50 nm), in contrast to PVA, shows a modest increase after iodine treatment. Raman spectra of PVA and I-PVA nanofibers are shown in Figure 4. The Raman spectrum of the as-spun PVA nanofibers, shown in Figure 4(a), gives noticeable scattering peaks assigned as the stretching vibration of CH and CH_2 bonds at ca. 2900-3000 cm^{-1}. There is a low intensity peak at 3100 cm^{-1}, which is associated with OH bonds [16]. In contrast, the Raman spectrum of PVA-I nanofibers (Figure 4(b)) shows one strong peak at around 1500 cm^{-1} that confirms intermolecular cross linking of the PVA molecules and the formation



(a) (b)

Figure 3. FE-SEM images of (a) PVA, and (b) PVA-I nanofibers (20000X).

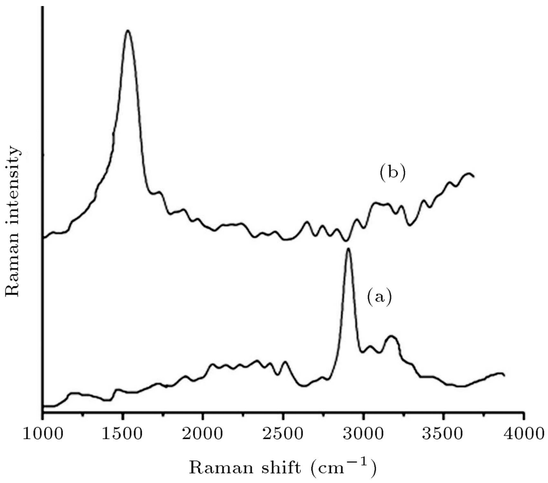


Figure 4. Raman spectra of (a) PVA, and (b) PVA-I nanofibers.

of polycyclic structures due to the dehydration of PVA [17]. The XRD patterns of PVA and PVA-I nanofibers are shown in Figure 5. As shown in Figure 5(a), the PVA nanofiber has a peak in the range of $2\theta = 17-21°$ that indicates the crystalline structure of nanofiber. With an iodine treatment, as shown in Figure 5(b), the crystalline structure of the nanofibers is lost and this is mainly attributed to the large X-ray absorption coefficient of iodine and therefore decrease in the crystalline orientation of the nanofibers [18,19]. Figure 6 shows FESEM images of carbon nanofibers obtained from pure and iodinated PVA nanofibers that were carbonized at 1100°C. During the carbonization process, the fibrous structure of PVA nanofibers was lost, while in iodinated nanofibers, with increasing the thermal stability, the fibrous structure was maintained and did not melt during the carbonization process. After carbonization of PVA-I nanofibers, the average diameter of carbon nanofibers decreases to 180-400 nm. The FE-SEM images of carbonized nanofibers at different temperatures of 700°C, 900°C, and 1100°C were shown in Figure 7. The diameter of carbon nanofibers showed a slight reduction with the increase of carbonization temperature from 700°C to 900°C that is associated with the decomposition of the residual main polymer chains, intermolecular changes, and bond formation, while there was no change in nanofiber diameter with increasing temperature from 900°C to 1100°C.

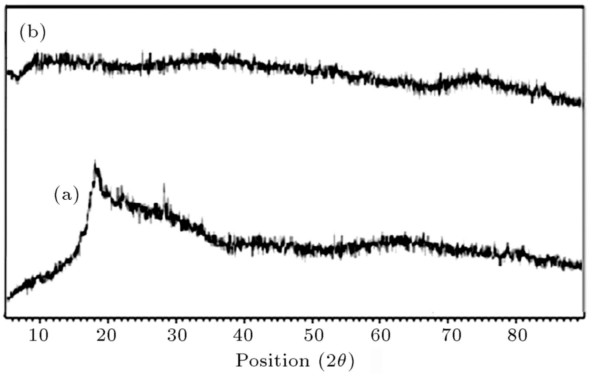
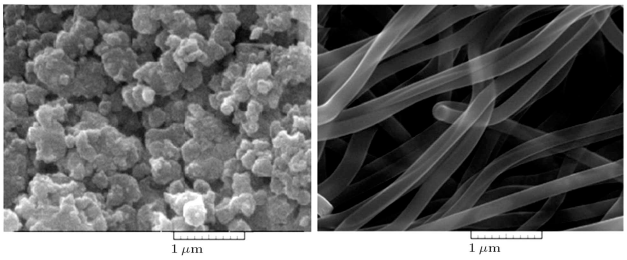


Figure 5. XRD patterns of (a) PVA, and (b) PVA-I nanofibers.

Raman spectra of carbonized PVA-I nanofibers at different temperatures are shown in Figure 8. The sharp absorption bands at ca. 1340 cm^{-1} and 1590 cm^{-1} are assigned to the D (characteristic of a disordered sp^3 phase) and G bands (characteristic



(a) (b)

Figure 6. FESEM images of carbonized PVA nanofibers synthesized from (a) PVA, and (b) PVA-I (30000X).

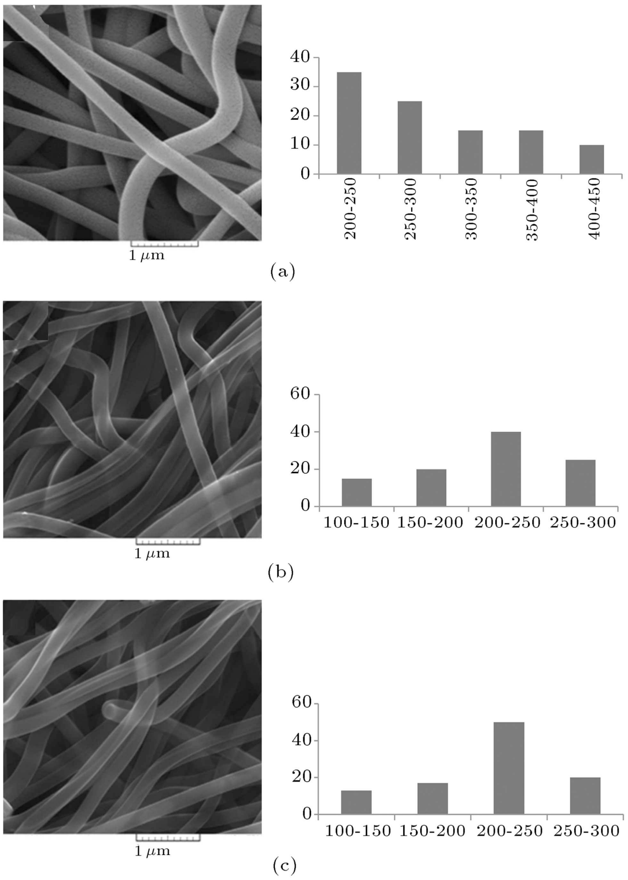


Figure 7. FESEM images and distribution plots of carbonized PVA-I nanofibers at (a) 700°C, (b) 900°C, and (c) 1100°C (40000X).

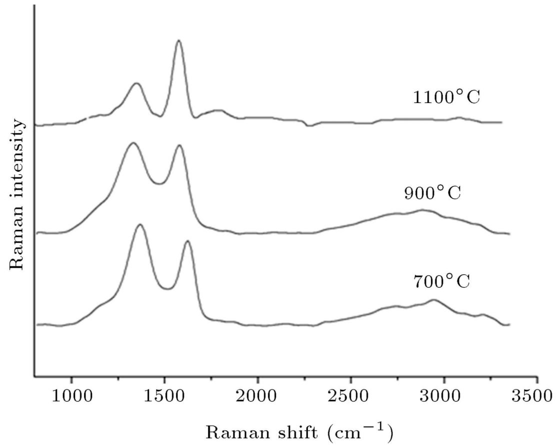


Figure 8. Raman spectra of carbonized PVA nanofibers at different temperatures.

of the E_{2g} vibration of the infinite crystal, associated with graphitic carbon atoms with sp^2 electronic configuration), respectively. Polycrystalline graphite has a distinct peak at 1340 cm^{-1} arising from the A_{1g} mode, which is attributed to the diamond-like carbon atoms with sp^3 configuration. With an increase in the carbonization temperature, the peak position did not change, but the intensity of G band increased mostly for the sample which was carbonized at 1100°C. This shows that the graphite structure has been developed well at 1100°C, compared to the carbon nanofibers which carbonized at 700°C and 900°C [20,21]. Figure 9 shows XRD patterns of the carbonized PVA-I nanofibers in different temperatures. After carbonization, one broad peak at $2\theta = 24°$ for carbon nanofibers was obtained by heating at both 700°C and 900°C, which indicates the low crystallinity [15]. The existence of carbon structures in the sample, which carbonized at 1100°C, is confirmed by the peaks at around $2\theta = 18-28°$ and $43-45°$, which correspond to the 002 and 10 reflections of disordered micro graphite stacking, respectively. The main peak (002) is very weak, because the temperature of carbonization was not high enough [22,23]. According to both of the morphological and structural analyses, and given rise to the formation of graphite structures in the sample carbonized at 1100°C, this temperature was chosen as the optimum carbonization temperature for PVA nanofibers. There was no change in carbon efficiency with varying carbonization temperatures and it was observed for each temperature equal to 30%.

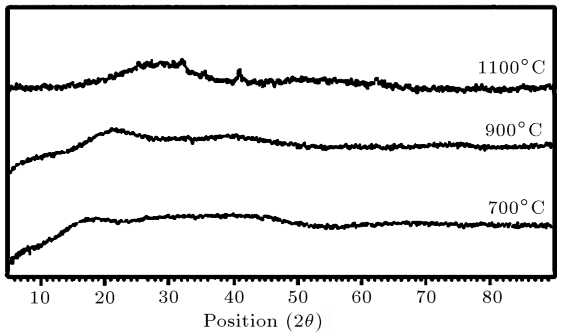
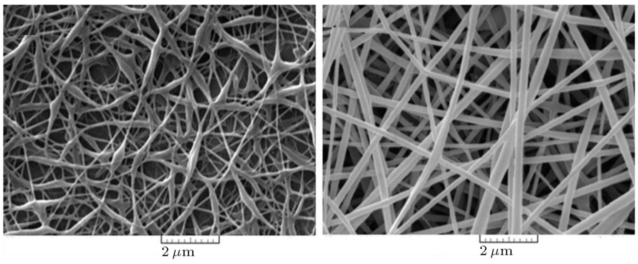


Figure 9. XRD patterns of carbonized PVA nanofibers at different carbonization temperatures.

3.2. Characterization of PVA-Cd and PVA-Cd-I nanofibers

The effect of inorganic cadmium salt addition on the electrospinning of PVA solution is investigated. As a result of the addition of an ionic salt, the solution conductivity increases, as reported by other researchers [24]. Highly conductive solutions exhibit less spinnability and also lead to more bead formation during the electrospinning process (Figure 10(a)) [25]. In order to obtain bead less nanofiber, different voltages and distances were applied. The optimal experimental conditions for electrospinning PVA-Cd acetate solution (8 wt%) can be found in Figure 10(b). The TGA curves of the PVA, PVA-Cd nanofibers are shown in Figure 11(a) and (b). PVA-Cd begins to degrade with the evaporation of water, taking place at around 100°C, which is shown in the first step of weight



(a) (b)

Figure 10. PVA-Cd nanofibers produced at (a) concentration = 8 wt%, voltage = 21 kV, distance = 11 cm, and rate = 9 μlit/min, and (b) concentration = 8 wt%, voltage = 17 kV, distance = 8 cm, and rate = 9 μlit/min (20000X).

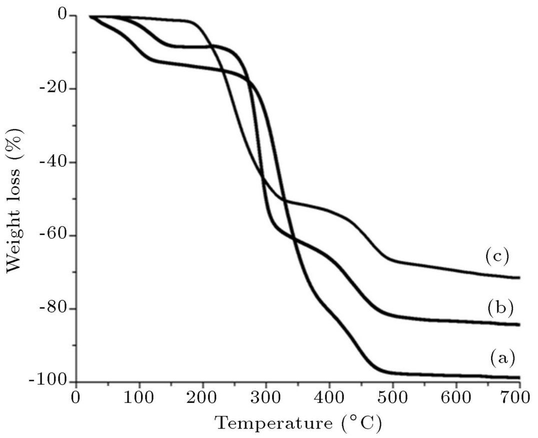


Figure 11. TGA curves of (a) PVA, and (b) PVA-Cd, and c) PVA-Cd-I nanofibers.

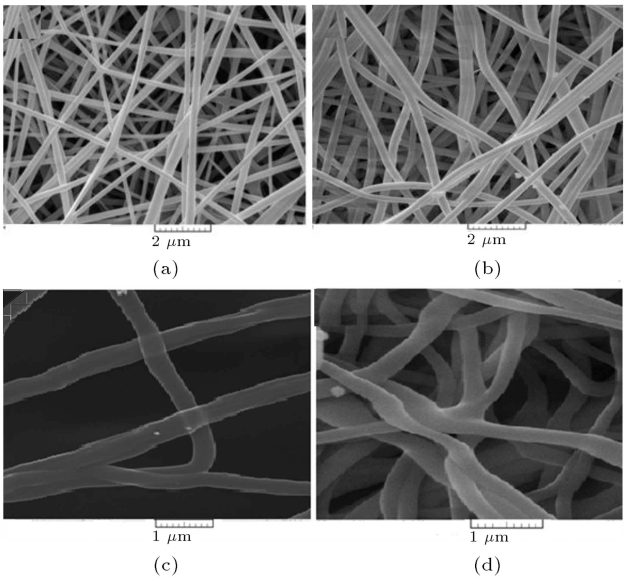


Figure 12. FESEM images of (a) PVA-Cd, (b) PVA-Cd-I nanofibers (20000X) and carbonized, c) PVA-Cd, and (d) PVA-Cd-I nanofibers at 1100°C (40000X).

loss in TGA curves. The dramatic weight loss in the range of 290-500°C is attributed to the pyrolysis of the nanofibers which indicates the decomposition of PVA-Cd. The weight loss slowed down at above 550°C to give a yield of 16%. As can be seen, the metal ions affect the thermal decomposition and the carbonization behavior of vinyl polymers [2]. Harada et al. [13] reported that the addition of a small amount of Ni metal catalyst promotes the dehydration reaction of PVA during carbonization. There is a drastic weight loss which starts at 200°C in the TGA of PVA-Cd -I nanofiber (see Figure 11(c)). This is associated with the dehydration process which forms C=C bonds and aromatic structures. The obtained higher yield from iodinated PVA would be related to the intermolecular cross-linking of PVA-Cd by iodination, which causes inhibition of volatile component containing carbon from PVA-Cd during heat treatment [23].

Figure 12 shows the FE-SEM images of the raw and carbonized PVA-Cd, PVA-Cd-I nanofibers at 1100°C. As shown in Figure 12(a), the electrospun PVA-Cd nanofibers display a smooth and uniform structure. The high-magnification FESEM image clearly shows that the electrospun nanofibers are straight with an average diameter of approximately 300±50 nm. Furthermore, it can be seen that iodinated PVA-Cd nanofibers become swollen and obvious, and the average diameter of nanofiber increases to 350 ± 50 nm with iodine treatment. Figure 12(c) displays the FESEM image of the PVA-Cd nanofibers carbonized at 1100°C. The FESEM image indicates that due to the low thermal stability of PVA-Cd nanofibers, melting process and surface roughness happenes in the nanofibers structure. But after carbonization, iodinated PVA-Cd nanofibers are well maintained in fibrous structures (Figure 12(d)). It can be assumed that the average diameter of nanofibers is reduced from 350 ± 20 nm to 300 ± 20 nm by adding iodine during the carbonization process.

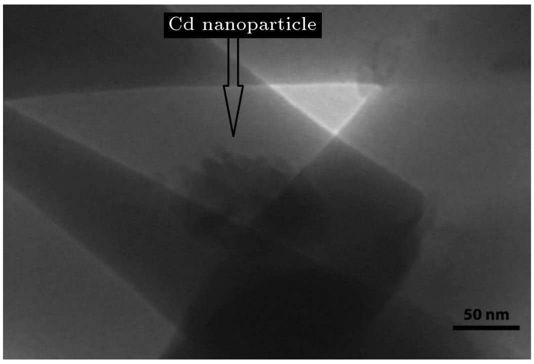


Figure 13. TEM images indicating cadmium metal inside the structure of PVA-Cd nanofiber.

The TEM analysis is carried out on the carbonized PVA-Cd at 1100°C nanofibers and the results are depicted in Figure 13. Cadmium metal is readily distinguishable in this image. The Raman spectra of as-spun PVA-Cd, PVA-Cd-I nanofibers and carbonized PVA-Cd, PVA-Cd-I nanofibers are shown in Figures 14 and 15. According to the Raman spectra (Figure 14(a)), addition of Cd-acetate to the PVA solution had no significant effects on the molecular structure of nanofiber. But in the Raman spectra of PVA-Cd-I (Figure 14(b)), the peak positioned at 1590 cm^{-1}, assigned to C=C bands, which indicates the formation of polycyclic aromatic structures by intermolecular cross linking of polyenes in the PVA nanofibers after iodine treatment. The Raman spectra

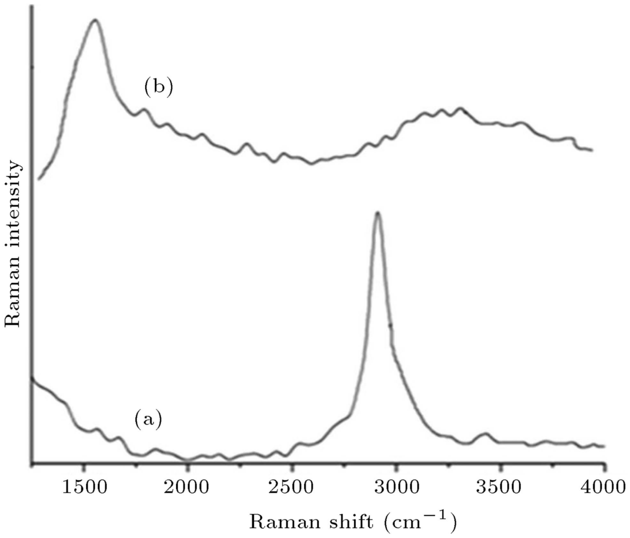


Figure 14. Raman spectra of (a) PVA-Cd nanofibers, and (b) PVA-Cd-I nanofibers.

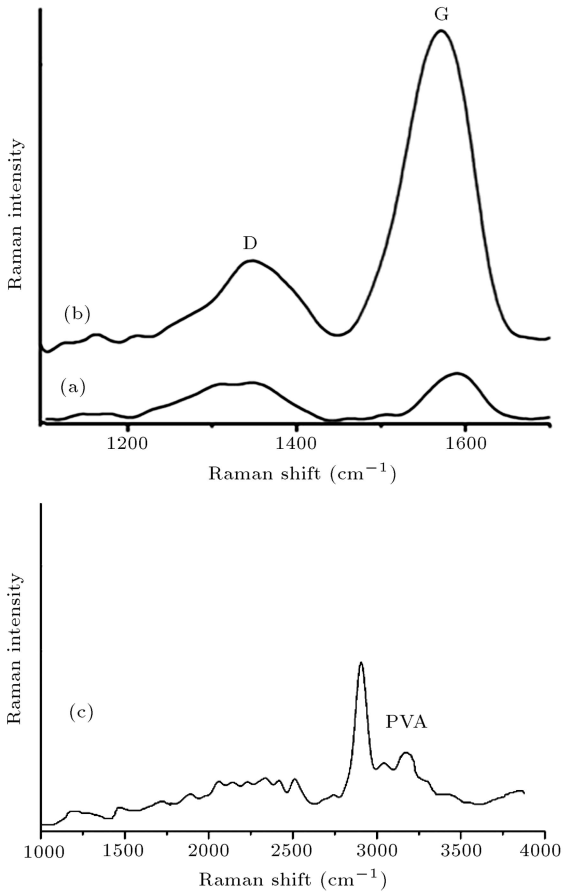


Figure 15. RAMAN spectra of carbonized (a) PVA-Cd, (b) PVA-Cd-I, and (c) PVA nanofibers.

of carbonized PVA-Cd and PVA-Cd-I nanofibers at 1100°C (Figure 15(a) and (b)) show two Raman bands at ca. 1360 cm^{-1} (D-band) and ca. 1580 cm^{-1}, corresponding to graphite peak (G band). The intensity ratio (known as the R value) of the "D-band" to the "G-band" (≈ 1) in the spectrum of carbonized PVA-Cd nanofibers indicates that nanofibers have a large amount of disordered carbon structures and a little ordered graphitic structure in comparison with the carbonized PVA-Cd-I nanofibers [26]. The XRD patterns of carbonized PVA-Cd and PVA-Cd-I nanofibers are shown in Figure 16. The pattern has two broad peaks in the range of $2\theta = 28 - 18°$ and 45-43° specifying the scattering of the (002) and (10) graphite planes and the formation of carbon structures in both samples.

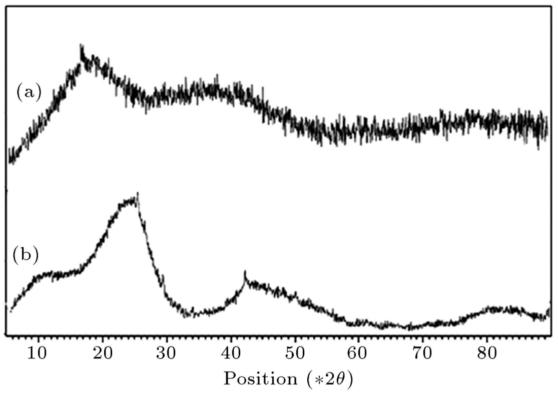


Figure 16. XRD patterns of (a) carbonized PVA-Cd nanofibers, and (b) carbonized PVA-Cd-I nanofibers.

4. Conclusions

Carbon nanofibers from electrospun PVA nanofibers with developing two simple and effective methods, iodine treatment and adding cadmium salt, were obtained. It is found that carbonization temperature at 1100°C for 2 h is suitable to obtain good carbon nanofibers in terms of carbon yield and morphological and structural properties of the carbon nanofiber. The iodine process causes structural changes in the PVA nanofibers and increases thermal stability to improve the carbonization process, which results in high carbon yield and intact nanofiber structure. Moreover, Cd metal nanoparticles were successfully incorporated into the PVA nanofibers using a spinning solution doped with Cd-acetate, which changes the electrospinning conditions for constant electrospinning. Furthermore, the carbon nanofiber derived from a PVA-Cd-I nanofiber precursor resulted in higher carbon yield (%34). The results also reveal that Cd, together with iodine treatment, significantly accelerate the preparation of carbon nanofiber, while remaining nanofiber structural after carbonization at 1100°C.

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