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Performance of electrodes synthesized with polyacrylonitrile-based carbon nanofibers for application in electrochemical sensors and biosensors

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ABSTRACT

The purpose of this work was to investigate the performance of electrodes synthesized with Polyacrylonitrile-based carbon nanofibers (PAN-based CNFs). The homogenous PAN solutions with different concentrations were prepared and electrospun to acquire PAN nanofibers and then CNFs were fabricated by heat treatment. The effective parameters for the production of electrospun CNF electrode were investigated. Scanning electron microscopy (SEM) was used to characterize electrospun nanofibers. Cyclic voltammetry was applied to investigate the changes of behavior of electrospun CNF electrodes with different diameters. The structure of CNFs was also evaluated via X-ray diffraction (XRD) and Raman spectroscopy. The results exhibited that diameter of nanofibers reduced with decreasing polymer concentration and applied voltage and increasing tip-to-collector distance, while feeding rate did not have significant effect on nanofiber diameter. The investigations of electrochemical behavior also demonstrated that cyclic voltammetric response improved as diameter of CNFs electrode decreased.

1. Introduction

Carbon-based nanomaterials have attracted increasing attentions for the application in electrochemical sensors and biosensors owing to conductivity, biocompatibility, functionality, large surface area, wide potential window, free corrosion process in positive potential and low background current [1–3]. Among these, electrospun CNFs, as one-dimensional nanostructures have been recently received considerable interest due to the fact that they can be simultaneously used both as transducers which relay the electrochemical signal and as matrixes for the immobilization of biomolecules [1,4–7].

Electrospinning is a simple and inexpensive method to fabricate polymer-based nanofibers with the diameter ranging from tens of nanometers to several micrometers [8–15]. In electrospinning process, a high voltage power source, a nozzle and a collector covered with aluminum foil are applied. The potential difference between nozzle and collector leads to stretch of solution and creates a thin jet from polymeric solution toward the collector. During stretch of solution to collector, the solvent evaporates and ultrafine nanofibers form on the collector [16–20].

CNFs prepared by electrospinning method have the high surface area, porosity, uniformity and mechanical strength [21,22]. Among the different precursors for the fabrication of CNFs, PAN is used as a current polymer owing to easy carbonization process and high carbon yield. Unlike other polymers, PAN nanofibers can be directly applied as transducer (electrode) after stabilizing and carbonizing [9,23,24]. Electrospun CNFs in comparison with other carbon materials such as glassy carbon and carbon black present edge sites which make them suitable for functionalizing [25]. Likewise, compared to other carbon materials such as graphite powder, carbon rods and same materials which need to binder, CNFs can be used without any binder, resulting in increase in electrical conductivity of electrodes [23]. Besides, Electrospun CNFs have advantages over carbon nanotubes: (1) they can be synthesized free catalyst during stabilization and carbonization, causing high purity and (2) they may enhance the facilitation of electron transfer due to more edge sites over the carbon nanotubes [26]. V. Vamvakaki et al. reported that CNFs are suitable matrix for the development of biosensors and superior to carbon nanotubes or graphite powder [27]. Electrospun CNF electrodes also have high surface area due to their small diameter to improve electrochemical performance in comparison with other carbon electrodes [23].

Totally, the selection of an efficient electrode with high surface area and excellent electrical conductivity play an important role for electrochemical sensors and biosensors and miniaturizing electrodes to nanoscale increases their efficacy due to enhancement of the signal-to-noise

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Table 1
Electrospinning data for PAN nanofibers.

NO	PAN concentration (wt.%)	Distance between tip to collector (cm)	Flow rate (mL/h)	Voltage (KV)	Mean diameter of nanofibers (nm) ± SD	Morphology
1	7	10	1	20	72.11 ± 23.20	Beaded nanofibers
2	8	10	1	20	90.70 ± 21.68	Nanofibers
3	9	10	1	20	106.76 ± 28.26	Nanofibers
4	10	10	1	20	143.00 ± 44.49	Nanofibers
5	8	15	1	20	84.70 ± 42.82	Nanofibers
6	8	20	1	20	77.05 ± 13.29	Nanofibers
7	8	10	0.5	20	102.05 ± 33.38	Nanofibers
8	8	10	1.5	20	98.41 ± 27.20	Beaded nanofibers
9	8	10	1	15	79.94 ± 22.64	Nanofibers
10	8	10	1	10	67.05 ± 8.96	Nanofibers

ratio and reduction of the sample volume [25,28]. Therefore, the design and fabrication of a nanoelectrode for the application in electrochemical sensors and biosensors is necessary. In this work, the homogenous PAN solutions with different concentrations were prepared and electrospun to acquire PAN nanofibers. The influence of electrospinning process parameters, including tip-to-collector distance, feeding rate and applied voltage on morphology and diameter of PAN nanofibers was evaluated. Electrospun CNFs were then prepared by stabilization and carbonization of PAN nanofibers, and CNFs were directly used as electrode. Then, the performances of electrospun CNFs which can be directly used as nanoelectrode were investigated.

2. Experimental

2.1. Reagents

Polyacrylonitrile (PAN) received from Polyacryl Company (Iran) with a moleculare weight of 150,000 $g \cdot mol^{-1}$. Dimethylformamide (DMF) and Potassium ferricyanide were purchased from Merck Company. Sodium phosphate dibasic and potassium phosphate monobasic were bought from Sigma-Aldrich Company. Phosphate buffer solutions (PBS) were prepared by 0.1 M Na_2HPO_4 and 0.1 M KH_2PO_4. All solutions were prepared using ultra-pure water.

2.2. Electrospinning

The PAN polymer was dissolved in DMF as solvent under continuous and vigorous magnetic stirring at 40 °C for 12 h to obtain a homogenous solution. The PAN solution was electrospun using Electroris (Fanavaran Nano Meghyas Ltd., Co., Tehran, Iran). For every run, the PAN solution was put into a 10 mL plastic syringe fitted with an 18-gauge, stainless steel blunted needle as nozzle and a high-voltage power supply was used to charge the polymer solution between the syringe needle and a rotating collector. A syringe pump injected PAN solution to the tip and accelerated toward the collector and the electrospun nanofibers were collected on an aluminum foil. The effect of electrospinning variations, including polymer concentration, distance between tip-to-collector, feeding rate and applied voltage on the morphology and diameter of PAN nanofibers was investigated according to the data listed in Table 1.

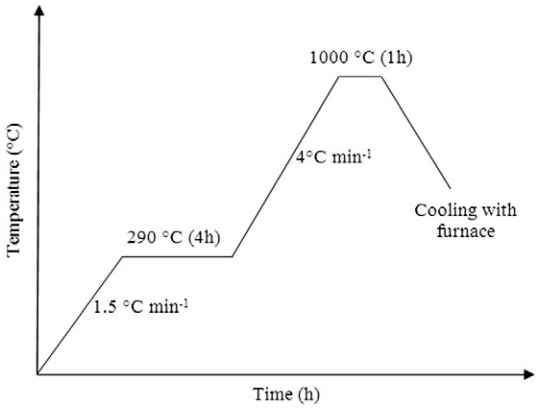

Fig. 1. Scheme of heat treatment process.

2.3. Preparation of CNFs and electrode

Electrospun PAN nanofibers were stabilized in an air atmosphere at 290 °C for 4 h with heating rate 1.5 °C min^{-1} by using a tube furnace, resulting in cyclization of nitrile groups and cross-linking between the chain molecules and hindrance from melting during carbonization step. Then, the stabilized nanofibers were carbonized at 1000 °C for 1 h in an inert atmosphere (high purity nitrogen) with heating rate of 4 °C min^{-1} (as seen in Fig. 1).

The resulted CNFs were spherically cut in size of 5 mm and inserted with copper wire for electrical contact and directly used as working electrode (Fig. 2).

2.4. Characterization of nanofibers

2.4.1. Scanning electron microscopy (SEM)

The morphology and diameter of nanofibers were carried out using SEM at an accelerating voltage of 20.0 kV (Philips XL-30) after sputtering with gold. The mean diameter of nanofibers was estimated about 50 nanofibers by SemAfore (4.01 demo, JEOL, Finland) software.

2.4.2. X-ray diffraction (XRD)

The XRD of CuKα radiation (λ = 1.54056 Å) was performed by a Philips Xpert instrument operating at 40 kV and 30 mA with at a step size of 0.08°/s over the 2θ range of 10–90°.

2.4.3. Raman spectroscopy

Raman spectra were performed with 25 mW power laser and 758 nm laser wavenumber and resolution about 3.0 cm^{-1} (Model: Senterra (2009) Bruker (Germany)). The spectra were recorded over a spectral range of 90–3500 cm^{-1} at room temperature. The measurement was taken at three various spots on the sample to realize the homogeneous nature of the CNFs.

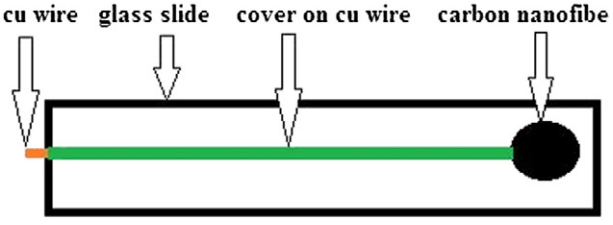

Fig. 2. Scheme of carbon nanofiber electrode.

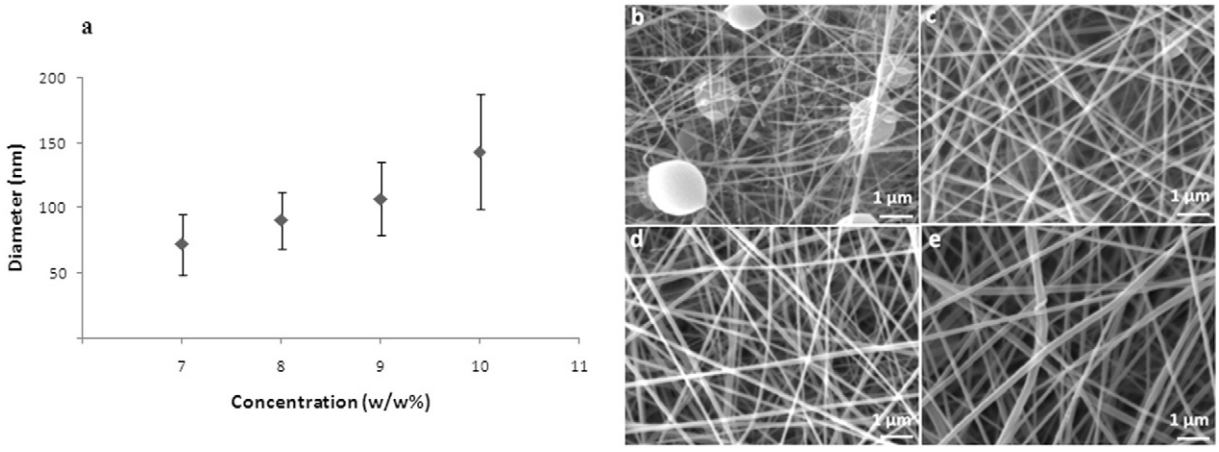

Fig. 3. (a) Effect of PAN solution concentration (wt.%) on nanofiber diameter; morphology of the nanofibers synthesized with (b) C = 7.00 wt.%, (c) C = 8.00 wt.%, (d) C = 9.00 wt.%, (e) C = 10.00 wt.% PAN solution.

2.4.4. Electrochemical behavior

All electrochemical experiments were performed on a computer-controlled μAutolab type III Potentiostat (Eco Chemie, Utrecht, The Netherlands). A platinum wire and an Ag/AgCl (saturated KCl) electrode were the auxiliary and reference electrodes, respectively.

2.4.5. Statistical analysis

The effects of different parameters on mean PAN nanofiber diameter were evaluated by using linear regression model through SPSS 16.0 software. Linear regression was applied to investigate the linear association between nanofibers diameter and electrospinning factors.

3. Results and discussion

3.1. Morphology and nanostructure of PAN nanofibers

Table 1 summarizes experiments conducted in this study to investigate different parameters affecting electrospun PAN nanofibers diameter and morphology as precursor of CNFs. The function of concentration as an effective factor on nanofibers diameter is shown in Fig. 3a. In these experiments (Table 1, No. 1–4), the applied concentrations were 7.00, 8.00, 9.00 and 10.00 wt.% whereas other three parameters were constant. The nanofibers diameter decreased from about 143 to 72 nm as the polymer concentration reduced from 10.00 to 7.00 wt.%, which was statistically significant (p-value < 0.0005). The literature also show that nanofibers diameter reduce by decreasing concentration [29–32]. The reason for this finding could be associated with the viscosity. Less viscous solution increase polymer chain mobility and decrease polymer chain entanglement, resulting in more flexible during electrospinning process and the formation of thinner nanofibers [33,34]. Besides, PAN-beaded nanofibers appeared on the collector for concentration of 7.00 wt.% (Fig. 3b) whereas uniform nanofiber formed by increasing concentration from 8 to 10 wt.% (Fig. 3c, d, e).

Likewise, tip-to-collector distance affects nanofibers diameter. In this experiment, 8.00 wt.% solution concentration was applied and the distance as a variable parameter enhanced from 10 to 20 cm and the other factors were constant. As seen in Fig. 4, the nanofibers diameter decreased from about 90 to 70 nm as tip-to-collector distance increased while other factors were fix (Table 1, Nos. 2, 5–6) and our results was statistically significant (p-value < 0.04). This decrease in nanofibers diameter may be due to the formation of two or several jet originated from a jet, resulting in thinner nanofibers. The literature also confirms that increase in tip to collector distance usually lead to smaller nanofibers [34,35]. Although some reports presented a direct relationship between the nanofibers diameter and tip to collector distance [36] which may be because of the electrostatic field strength, causing less stretching and bigger nanofibers [37].

The effect of flow rate on mean nanofiber diameter is evaluated according to Table 1 (Nos. 2, 7–8). In these experiments, 8.00 wt.% solution concentration was used and the flow rate as a variable parameter enhanced from 0.5 to 1.5 mL/h and the other factors were constant. As seen in Fig. 5, decrease in the mean diameter of nanofibers was observed by increasing feeding rate from 0.5 to 1 mL/h whereas the mean diameter of nanofibers enhanced as feeding rate increased from 1 to 1.5 mL/h. The effect of flow rate on the nanofibers diameter is contradictory. The literature shows that increasing flow rate of polymer solution typically leads to enhancement of the nanofibers diameter [38] while in some reports, it has a reverse relationship with nanofibers diameter [39]. The reason for this reverse relationship can be that the

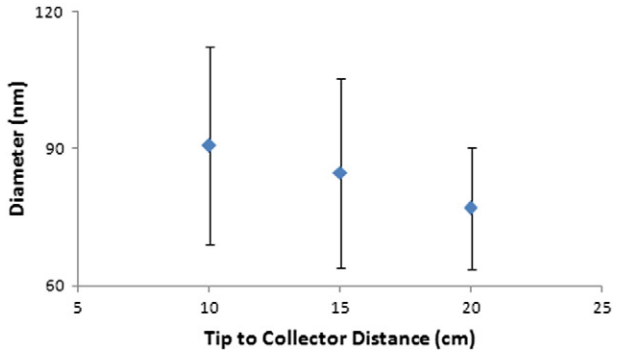

Fig. 4. Effect of tip-to-collector distance (cm) on nanofiber diameter.

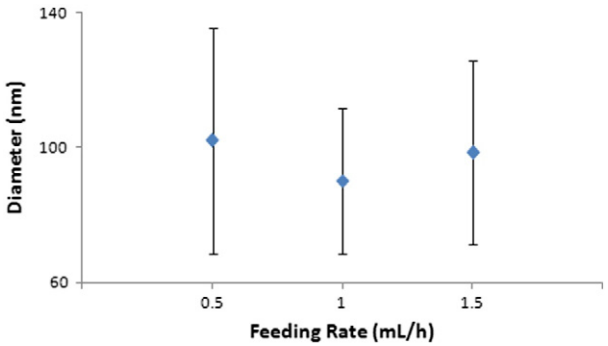

Fig. 5. Effect of feeding rate (mL/h) on nanofiber diameter.

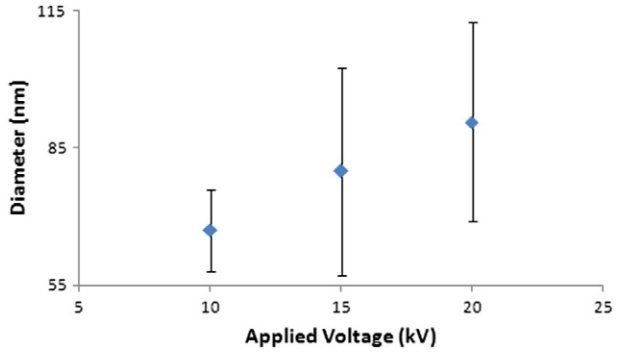

Fig. 6. Effect of applied voltage (kV) on nanofiber diameter.

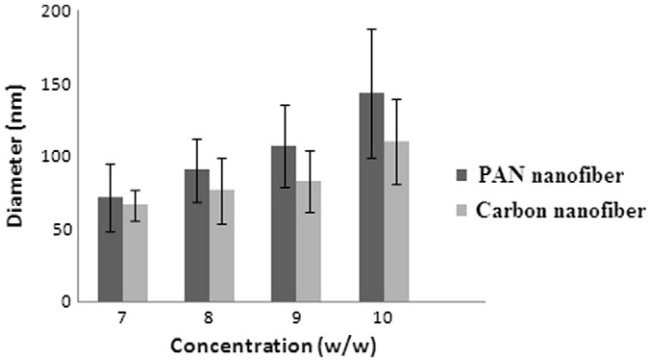

Fig. 8. Comparison of diameter of PAN nanofibers with CNFs.

Table 2
Results of linear regression analysis demonstrating associations between nanofibers diameter and electrospinning factors.

	B	p-Value
Concentration	21.861	0.000
Distance	-1.817	0.037
Feeding rate	-3.640	0.698
Voltage	2.807	0.007

solvent on the tip of the needle is evaporated at low injection rate, in comparison with high one, resulting in increase in concentration of solution and finally nanofibers diameter.

The effect of applied voltage on mean nanofiber diameter is depicted in Fig. 6 In this experiments, the solution concentration was 8.00 wt.%, while the applied voltage enhanced from 10 to 20 kV and the other factors were constant (as shown in Table 1, Nos. 2, 9–10). The mean nanofiber diameter is increased from about 67 nm to 90 nm which is statistically significant (p-value < 0.05) as the applied voltage enhanced from 10 to 20 kV. This increase in nanofibers diameter can be described that by increasing applied voltage, the solution move faster from tip of nozzle to collector and polymer jet is stretched in less time, resulting in bigger nanofibers diameter [40,41].

Totally, Table 2 shows the linear association between nanofibers diameter and electrospinning factors. All electrospinning factors were associated with nanofibers diameter except feeding rate and the strongest association was for concentration (B = 21.861).

3.2. Morphology and structure of CNFs

PAN nanofibers prepared with concentration of 7.00, 8.00, 9.00 and 10.00 wt.% were used to produce CNFs while other parameters were constant (according to Table 1). It is observed in Fig. 7a that the PAN-based CNFs diameter decreased from about 110 to 66 nm as PAN concentration reduced from 10.00 to 7.00 wt.%. Besides, CNFs which were prepared with 7 wt.% PAN solution formed the beaded CNFs (Fig. 7b) whereas CNFs prepared with 8.00, 9.00 and 10.00 wt.% PAN solution formed nanofibers without beads (Fig. 7c, d and e).

As shown in Fig. 8, the CNFs diameter decreased in comparison with PAN nanofibers diameter. This decrease in diameter can be attributed to heat treatment. By placing PAN nanofibers in the stabilization temperature, the formation of dense ladder-polymer structure start at above 200 °C to hinder from melting during carbonization. In carbonizing step, non-carbon elements were removed in form of various gases such as NH_3, HCN and H_2O which lead to formation of a network structure and shrinkage of nanofibers diameter [9].

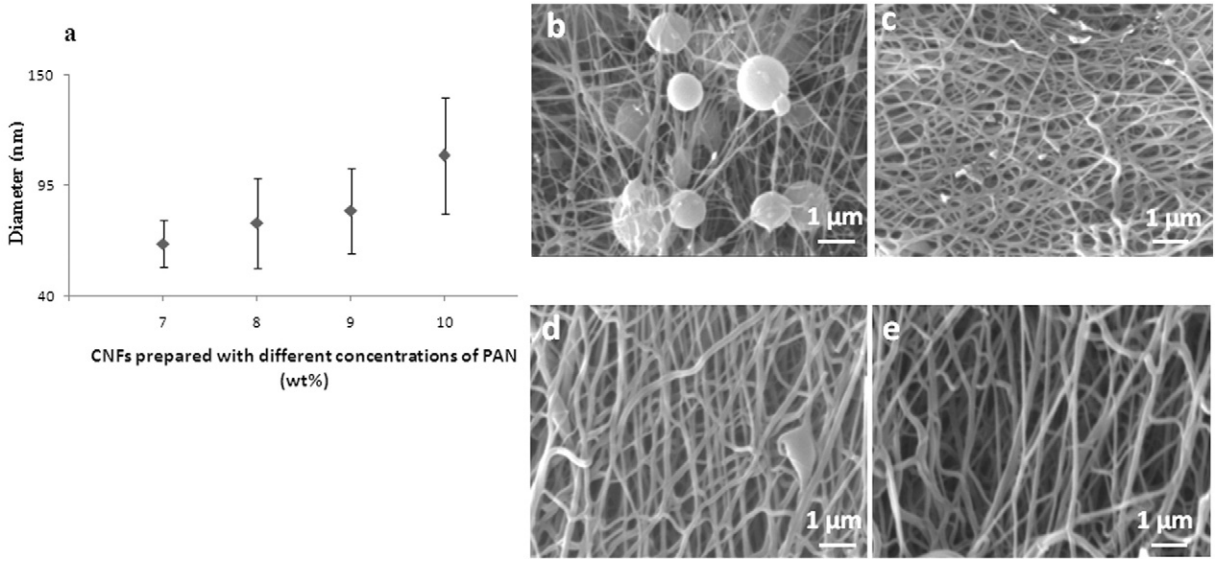

Fig. 7. (a) Effect of PAN solution concentration (wt.%) on PAN-based CNFs diameter; morphology of CNFs prepared with (b) C = 7.00 wt.%, (c) C = 8.00 wt.%, (d) C = 9.00 wt.%, (e) C = 10.00 wt.% PAN solution.

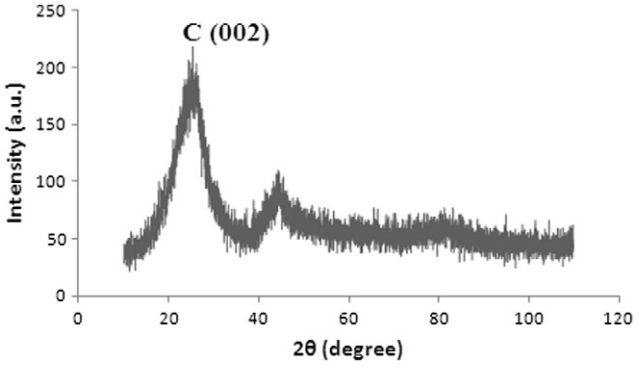

Fig. 9. XRD curve of PAN-based CNFs.

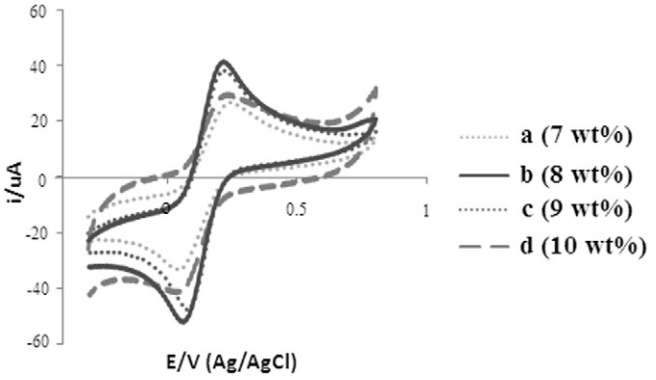

Fig. 11. Cyclic voltammetric behavior of CNF electrode prepared with different concentrations (a) 7 wt.%, (b) 8 wt.%,(c) 9 wt.%,(d) 10 wt.% for 5 mM $K_3Fe(CN)_6$ in 0.1 M PBS at pH 7 and scan rate 100 $mV \cdot s^{-1}$.

3.3. XRD and Raman spectroscopy analysis

XRD pattern acquired from electrospun CNFs is presented in Fig. 9. The diffraction peak around the 2θ ~ 25.5 can be assigned to the (002) crystallographic plane of graphic layer [42].

Raman spectroscopy was also performed for CNFs to acquired information about carbon nanostructures, which have a close relationship with the electrochemical behavior. As shown in Fig. 10, two peaks can be observed at around the wavenumber of 1298 cm^{-1} (D-band) that is associated with disordered turbostratic carbon structures and the wavenumber of 1598 cm^{-1} (G-band) that indicate ordered graphitic structures [43,44].

3.4. Cyclic voltammetric behavior of PAN-based CNF electrode

Cyclic voltammetry (CV) can be used as a method to study the electrode behavior. Fig. 11 shows CV behavior of CNF electrodes with different diameters for 5 mM $K_3Fe(CN)_6$ in 0.1 M PBS at pH 7 and scan rate 100 $mV \cdot s^{-1}$. It can be observed that the cyclic voltammetric response of ferricyanide redox at CNF electrodes increased as CNFs diameter decreased except curve (a) in Fig. 11. The increase in cyclic voltammetric response of ferricyanide redox can be attributed to conductivity of CNFs, that is, the conductivity of CNFs electrode increased as the CNFs diameter decreased. However, CNF electrode with the smallest diameters (about 66 nm) had the least cyclic voltammetric response (curve (a) in Fig. 11). The reason may be due to the formation of beads on CNFs to reduce conductivity of CNF electrode. In our experiments, the CNF electrode with the mean diameter about 76 nm (curve (b) in Fig. 11) had the best cyclic voltammetric response. The results of our experiments demonstrated that the desired diameter of CNFs for the application in electrochemical sensors and biosensors as an electrode is from approximately 75 to 80 nm, because the cyclic voltammetric response in CNFs electrode reduced due to increase in CNF diameter. On the other hand, CNFs with diameter less than approximately 75 nm also have a lot of beads, resulting in decrease in cyclic voltammetric response.

In our experiments, the variable parameters for CNFs with the mean diameter about 76 nm are No. 2 in Table 1 (concentration: 8 wt.%, distance between tip to collector: 10 cm, feeding rate: 1 mL/h, applied voltage: 20 kV).

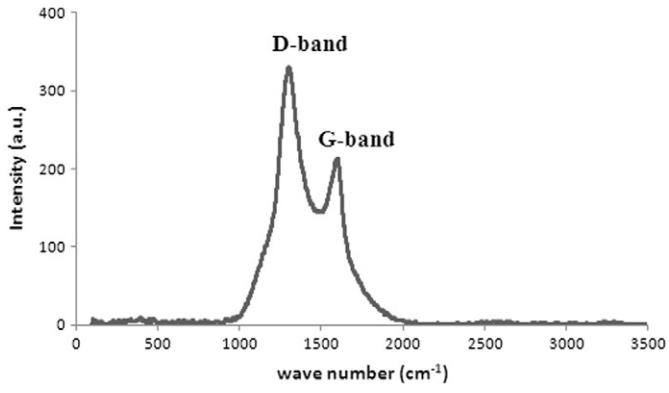

Fig. 10. Raman spectra of PAN-based CNFs.

4. Conclusion

This is first report from effective parameters on the fabrication of electrospun CNF electrode. The results demonstrate that the 8.00 wt.% provides enough chain entanglements for nanofiber formation. PAN nanofiber diameter increased as the solution concentration and applied voltage enhanced. It was also observed that increasing the tip to collector distance led to increase in nanofiber diameter while the feeding rate did not have considerable effect on PAN nanofiber diameter. The results of our experiments demonstrated that the desired diameter of electrospun CNFs which can be directly used as nanoelectrode is from about 75 to 80 nm. Electrospun CNFs with diameter less than approximately 75 nm due to beaded nanofibers reduce cyclic voltammetric response and electrospun CNFs with diameter more than approximately 80 nm decrease the cyclic voltammetric response owing to increase in CNFs diameter, resulting in decrease in conductivity. Therefore, electrospun CNFs with diameter about 75 to 80 nm which are directly used as working electrode have the desired performance in electrochemical sensors and biosensors.

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