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Morphology and conductivity of polyaniline nanofibers prepared by 'seeding' polymerization

Shuangxi Xing [a,b], Chun Zhao [a,b,*], Shengyu Jing [a], Zichen Wang [a,**]

[a] College of Chemistry, Jilin University, Changchun 130012, People's Republic of China
[b] Joint State Key Laboratory on Integrated Optoelectronics, Jilin University, Changchun 130012, People's Republic of China

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Abstract

Conventional polymerized polyaniline (PANI) dissolved in dimethyl sulphoxide (DMSO) was used as 'seed' to induce the polymerization of aniline and hence prepare PANI nanofibers. The different preparation conditions including acid concentration, reaction time, aniline concentration, PANI/DMSO amount, PANI/DMSO concentration, acid and organic solvent kinds were investigated to discuss the influence of these conditions on the morphology and conductivity of the resulting samples. FT-IR spectra were measured to give further proof for the different effect on the conductivity. The scanning electro microscope images of PANI product extracted from the reaction solution at different time were collected and a comparison between this 'seeding' polymerization, conventional and rapidly mixing polymerization was also made to make clear the advantage of this approach and the mechanism of the formation of PANI nanofibers.

Keywords: Polyaniline; DMSO; 'Seeding' polymerization

1. Introduction

Polyaniline (PANI) as one of the conducting polymers has received considerable investigation due to its unique electronic and optical properties and hence its potential application in many fields, such as electronic devices and different sensors [1–3]. In recent years, people put much attention to the synthesis of PANI with nanostructure like nanorod and nanofiber because, for example, the nanostructured PANI sensors showed much higher response than that of the conventional polymerized ones [4–6]. As for the synthesis of nanostructured PANI, many approaches including template and nontemplate process have been used and developed [7–20]. Surfactants are usually good candidates for the template-synthesis of PANI with nanostructure because they provide appropriate manner to guide the polymerization of aniline with certain orientation [7,8]. Utilization of some functionalized protonic acid, such as *p*-aminobenzenesulfonic acid, naphthalenesulfonic acid or others provides an effective way to prepare nanostructured PANI without template [9–12]. Recently, interfacial polymerization as a facile approach has been employed for the synthesis of PANI nanofibers, where aniline polymerizes at the interface between two immiscible liquids [14,15]. Apart from that, a 'nanofiber seeding' polymerization as a new typical template approach was used for the synthesis of PANI nanofibers by using a small amount of nanofibers regardless of their chemical nature, producing a precipitate with bulk fibrillar morphology [18]. Especially Huang et al. reported a rapidly mixing polymerization of the aniline to obtain PANI nanofibers in order to make clear the formation mechanism of the PANI nanofibers [21]. They considered the nanofibers formed at the early stage of the reaction and the interfacial and rapidly mixing polymerization suppressed the secondary growth of the particles. Nevertheless, there are still many problems, such as the improvement of the conductivity, the control of the morphology and the application of these products. Here, we provided a simple approach to synthesize PANI nanofibers via using conventional polymerized PANI powder dissolved in dimethyl sulphoxide (DMSO) as 'seed' and the product showed much high conductivity and homogeneous nanofiber morphology. The morphology of the PANI product extracted from the reaction solution at different time was observed and the effects of different conditions on the formation of the PANI nanofibers were discussed in detail in

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* Corresponding authors. Address: Joint State Key Laboratory on Integrated Optoelectronics, Jilin University, 10 Qianwei Road, Changchun, Jilin 130012, People's Republic of China. Tel.: +86 431 516 8241; fax: +86 431 849 9134.
E-mail addresses: zchun@jlu.edu.cn (C. Zhao), wangzc@mail.jlu.edu.cn (Z. Wang).

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order to further investigate the function of the 'seed' and the mechanism of the formation of the nanofibers. On the other hand, a comparison between different approaches including conventional polymerization and rapidly mixing polymerization has been made. It should be noted that our approach was different from the 'nanofiber seeding' polymerization reported by Manohar SK et al., in which the nanofiber should be pre-synthesized with a relatively complex process [18].

2. Experimental

2.1. Materials

Aniline was distilled under vacuum before use and other regents were used as received.

2.1.1. Synthesis of PANI nanofibers

Twenty milligrams of conventional polymerized PANI powder in doped state was dissolved in 20 mL of DMSO, which was used as the 'seed' in the polymerization. The 'seed' was added into the aniline/hydrochloride solution followed by dropping of ammonium sulfate/hydrochloride solution with the speed of one drop every 2 s. The mixture was stirred at room temperature for a period of time and the resulting precipitate was collected by filter. After the product was washed by deionized water and ethanol continuously, the product was dried under vacuum at room temperature for 48 h. The detailed preparation conditions were listed in Table 1.

2.2. Characterization

The morphology of the PANI nanofibers was observed using scanning electron microscopy (SEM, XL-30 ESEM FEG) with gold coating.

The conductivity of powder pellets at room temperature was measured by a typical four-probe method (SDY-5).

FT-IR spectra of the different samples were measured on a FT-IR-8400s (Shimadzu) spectrometer in the transmission mode. Standard KBr technique was applied. Resolution of the measurements was equal to 4 cm^{-1}.

X-ray diffraction pattern was taken with a Shimadzu XRD 6000 instrument at a 5°/min scanning speed from 5 to 60°.

3. Results and discussion

3.1. Morphology

As for the formation of PANI nanofibers, we assumed that when the PANI powder dissolved in DMSO was dispersed in water, it would act as 'seed' to induce the polymerization similar to 'nanofiber seeding' or oligomer-assisted synthesis of PANI nanofibers [18,22]. However, in our experiment, the PANI 'seed' was homogeneously dispersed in the reaction system because DMSO could be partly dissolved in water. Then the monomer aniline would contact with PANI nearly at the same time, which could both accelerate the induction time and lead the aniline to polymerize in a certain orientation. As a result, PANI with fiber morphology was generated at the early stage and ultimately orchestrated the bulk formation of the similar nanostructure.

Table 1
Preparation conditions and conductivity of the samples

No	Approach	Acid	Acid concentration (M)	Solvent	'Seed' amount (mL)	'Seed' concentration (mg/mL)	Aniline concentration (M)	Reaction time (h)	Conductivity (S/cm)
1	Seeding	HCl	0.01	DMSO	1	1	0.1	8	0.84
2	Seeding	HCl	0.1	DMSO	1	1	0.1	8	2.98
3	Seeding	HCl	0.5	DMSO	1	1	0.1	8	25
4	Seeding	HCl	1	DMSO	1	1	0.1	8	34.5
5	Seeding	HCl	1	DMSO	1	1	0.1	4	33.28
6	Seeding	HCl	1	DMSO	1	1	0.1	12	30.32
7	Seeding	HCl	1	DMSO	1	1	0.025	8	7.4
8	Seeding	HCl	1	DMSO	1	1	0.05	8	9.52
9	Seeding	HCl	1	DMSO	1	1	0.2	8	7.7
10	Seeding	HCl	1	DMSO	0.1	1	0.1	8	14.28
11	Seeding	HCl	1	DMSO	0.2	1	0.1	8	14.28
12	Seeding	HCl	1	DMSO	0.5	1	0.1	8	25
13	Seeding	HCl	1	DMSO	1	0.1	0.1	8	8.16
14	Seeding	HCl	1	DMSO	1	0.2	0.1	8	20
15	Seeding	HCl	1	DMSO	1	0.5	0.1	8	22.24
16	Seeding	H_2SO_4	1	DMSO	1	1	0.1	8	7.4
17	Seeding	H_3PO_4	1	DMSO	1	1	0.1	8	13.04
18	Seeding	HCl	1	DMF	1	1	0.1	8	7.15
19	Seeding	HCl	1	DMAc	1	1	0.1	8	7.32
20	Seeding	HCl	1	NMP	1	1	0.1	8	5.16
21	Conventional	HCl	1	–	–	–	0.1	8	4.76
22	Rapidly mixing	HCl	1	–	–	–	0.1	8	5.72

The acceleration effect of the seed to induce the reaction could be directly seen from the color change of the solution. For the ‘seeding’ polymerization, the solution turned blue in 80 s, while the conventional one would take nearly 400 s. On the other hand, this effect could also be observed by monitoring the change of the solution temperature (Fig. 1) [22]. The time for seed polymerization to start was about 300 s faster than that of conventional reaction, which was consistent with the color change. This faster induction time might be helpful to the formation of uniform morphology by suppressing consequent growth and aggregation of the nanofibers [21].

To further make clear the formation of the PANI nanofibers, small amounts of product for SEM studies were periodically extracted from the reaction system as soon as the green color of PANI was observed. The samples were dropped onto a piece of clean glass and quickly dried in air via the aid of infrared lamp in order to avoid the further polymerization of the aniline. From Fig. 2(a), we can find PANI nanofibers formed at the early stage in the polymerization process. As more APS was fed into the reaction solution (30 min after the solution turned green), no apparent aggregation was found (Fig. 2(b)) and it did not show much difference when the reaction was carried out for a longer time, e.g. 1 h as presented in Fig. 2(c). This result was quite different from that of the conventional polymerization of aniline, in which the aggregation turned serious along with the reaction time [21]. Therefore, in our experiment, we consider the ‘seed’ really played an important role to help forming, stabilizing and dispersing the PANI nanofibers.

3.1.1. The effect of acid concentration

The SEM images of the PANI nanofibers obtained from different acid concentration are given in Fig. 3. Although there existed aggregation and much nanoparticles in the products from lower acid concentration, such as 0.01 and 0.1 M as shown in Fig. 3(a) and (b), it could be found that nanofibers had been formed under such conditions. In our experiments, we observed when low acid concentration was used via conventional or rapidly mixing polymerization, there were nearly no nanofibers formed in the resulting products. So we considered the ‘seeding’ polymerization would do make any effect on the formation of nanofibers though the acid concentration played an important role on the morphology of the products. This could be proved by the samples obtained from a higher acid concentration like 0.5 and 1 M (Figs. 3c and d). In these systems, nanofiber PANI could be easily prepared via this ‘seeding’ polymerization when the acid concentration was enough for the successful polymerization of aniline.

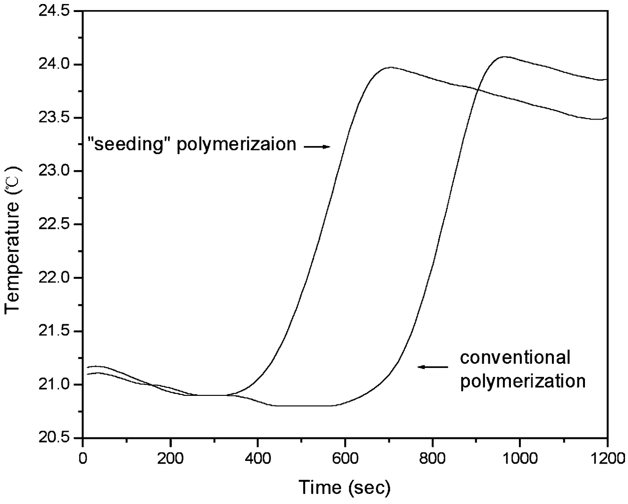

Fig. 1. Temperature change during the induction time.

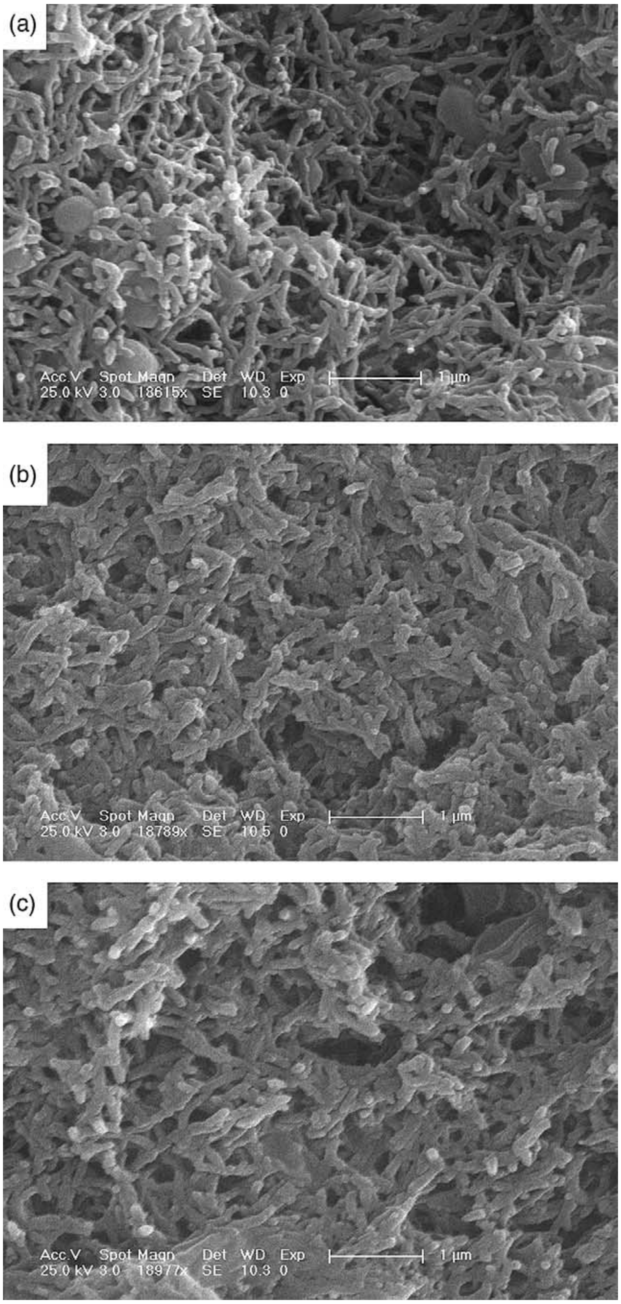

Fig. 2. SEM images of PANI samples extracted from the reaction solution at different time (a) as soon as the green color of PANI became visible; (b) after 30 min; (c) after 1 h.

3.1.2. The effect of reaction time

Fig. 4 shows the SEM images of PANI nanofibers prepared from 4 and 12 h, respectively. Combined with Fig. 3(d), which

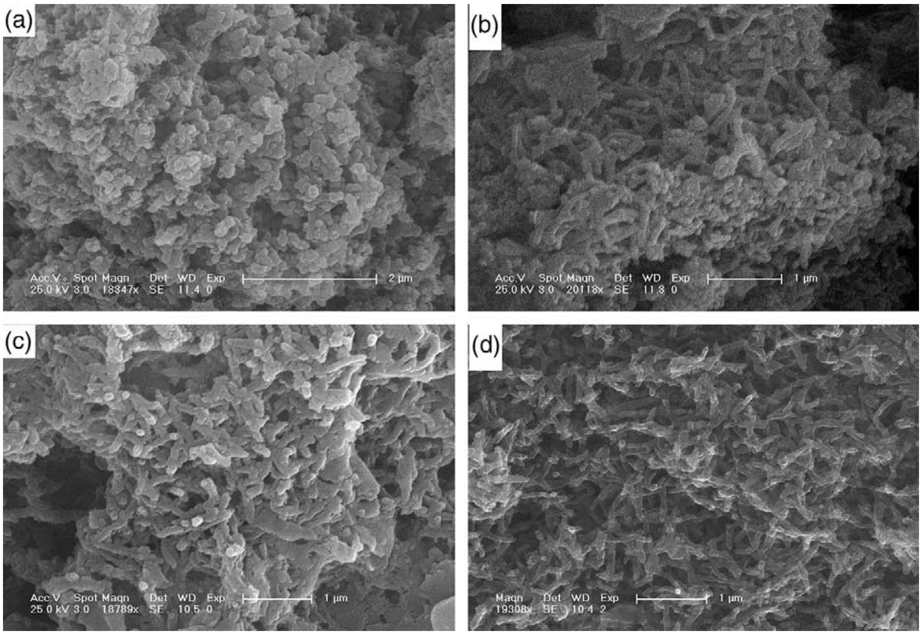

Fig. 3. SEM images of PANI obtained from different acid concentration (a) 0.01 M; (b) 0.1 M; (c) 0.5 M; (d) 1 M.

gives the SEM image of the sample with reaction time of 8 h, one could find there was little difference among these samples by length (ca. 500 nm) and width (ca. 70 nm). However, the obvious aggregation could be found in the sample from 12 h when conventional and rapidly mixing polymerizations were used. Then the 'seed' might act as dispersant with the aid of the organic solvent dissolved in the solution, which would weaken the interaction between the aniline monomers and also the polymerized products.

3.1.3. The effect of aniline concentration

When the aniline concentration was increased from 0.025 to 0.2 M, the length of the nanofibers did not change much with an average size of ca. 500 nm, as can be seen from Fig. 5. However, the width of the nanofibers increased with the aniline concentration from ca. 50 nm at 0.025 M to ca. 160 nm at 0.2 M. This indicated that the aniline monomer had the tendency to reach the 'seed' and began polymerization because of the interaction between the monomer and the PANI already formed with the 'seed' as center. Meanwhile, the polymerization seemed to occur along certain orientation that leaded to the increase of the width of the nanofibers.

3.1.4. The effect of PANI/DMSO amount

Different amounts of PANI/DMSO solution were used as 'seed' and the SEM images of the obtained PANI nanofibers are shown in Fig. 6. It appeared there was not any difference among these samples on their morphology, which might indicate that very few amount of PANI/DMSO solution as 'seed' would be adequate for the formation of PANI nanofibers. Whereas, a more comprehensive observation on the different samples showed a system with lower amount of 'seed' could not result in homogeneous PANI nanofibers perhaps due to the worse dispersion of the 'seed' in the reaction solution.

3.1.5. The effect of PANI/DMSO concentration

To further prove the conclusion that low amount of 'seed' would lead to aggregation of the resulting PANI, different concentrations of PANI/DMSO solution were used. The same volume of organic solvent but different amounts of PANI powder was used and the SEM images of the samples are presented in Fig. 7. It was found that the size of the PANI nanofibers did not show much change not only in length (ca. 500 nm) but also in width (ca. 70 nm), but the aggregation

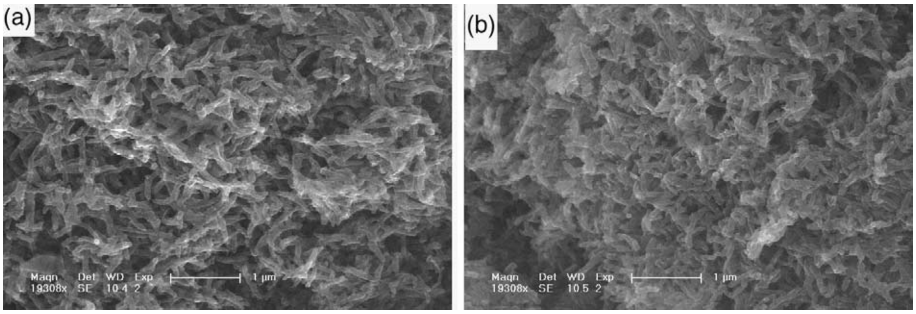

Fig. 4. SEM images of PANI obtained from different reaction time (a) 4 h; (b) 12 h.

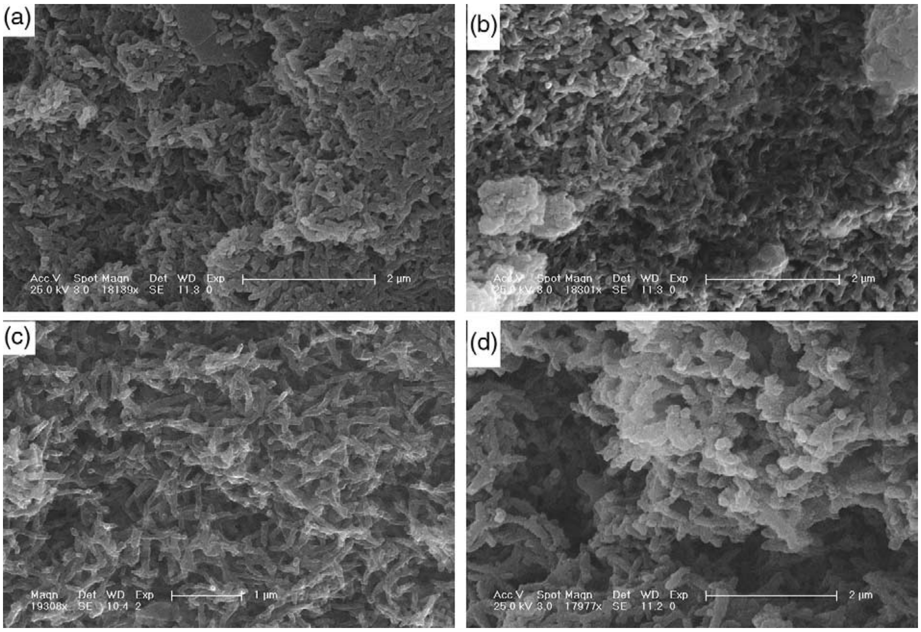

Fig. 5. SEM images of PANI obtained from different aniline concentration (a) 0.025 M; (b) 0.05 M; (c) 0.1 M; (d) 0.2 M.

became obvious when lower PANI/DMSO concentration was used (Fig. 7(a)).

3.1.6. The effect of acid kinds

Fig. 8(a) and (b) gives the SEM images of the samples obtained from H_2SO_4 and H_3PO_4 as mediums, respectively. Although there was some aggregation in these samples, a large amount of nanofibers could be easily observed. On the other hand, when using conventional or rapidly mixing polymerization, we could not get such good results as these samples. So we believed that the 'seed' would operate in spite of the existence of the big counter ions.

3.1.7. The effect of organic solvent kinds

Dimethyl formamide (DMF), *N*,*N*-dimethyl acetamide (DMAc) and *N*-methyl-2-pyrrolidinone (NMP) were also used as solvents to dissolve PANI powder (doped) and made as 'seed' to prepare PANI nanofibers. The SEM images of these samples are shown in Fig. 9. From these images we could find the sample obtained from NMP as solvent showed a relatively bad result with obvious aggregation (Fig. 9(d)). The other two, e.g. DMF and DMAc as solvents (Fig. 9(b) and (c), respectively) presented better morphology of PANI nanofibers, but they were not as good as that obtained from DMSO as solvent (Fig. 9(a)). As we knew, NMP was considered as one of the best solvents for PANI, but why it was not fit for the 'seeding' polymerization? In order to make clear this question, we did another experiment by using undoped PANI powder dissolved in DMSO as 'seed' and the result showed a quite similar morphology as the one shown in Fig. 9(d). NMP, DMF and DMAc were relatively stronger base than DMSO, and the doped PANI would undergo much faster change to intrinsic PANI than in DMSO. So it was not strange to get the above results. Although the exact reason why undoped PANI was not fit for the preparation of PANI nanofibers was not clear, we considered it was related to the interaction between the powder and the solvent and the second dope of PANI in the reaction solution.

3.1.8. The effect of approaches

The SEM images of the samples prepared via conventional and rapidly mixing polymerization are presented in Fig. 10. As for the conventional polymerization, the secondary growth of the PANI nanofibers led to the formation of the amorphous structure (Fig. 10(a)) [21]. However, in rapidly mixing and this 'seeding' polymerization, the secondary growth was weakened or even impeded because of the fast induction period and homogeneous structure of nanofibers could be obtained using these two approaches (Figs. 9(a) and 10(b)).

3.2. Conductivity

As we knew, the conductivity of polyaniline can be mainly affected by the percentage of protonation and the degree of

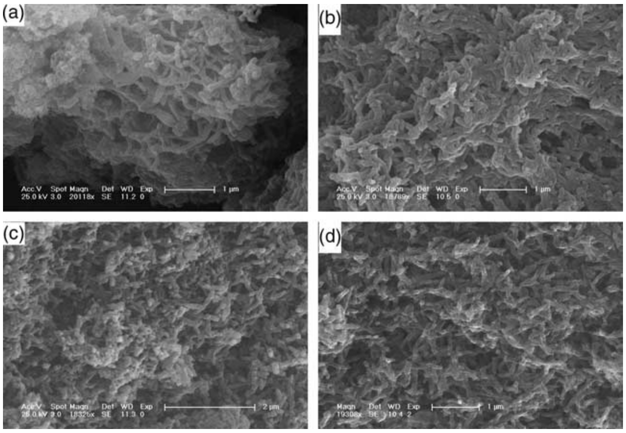

Fig. 6. SEM images of PANI obtained from different 'seed' amount (a) 0.1 mL; (b) 0.2 mL; (c) 0.5 mL; (d) 1 mL.

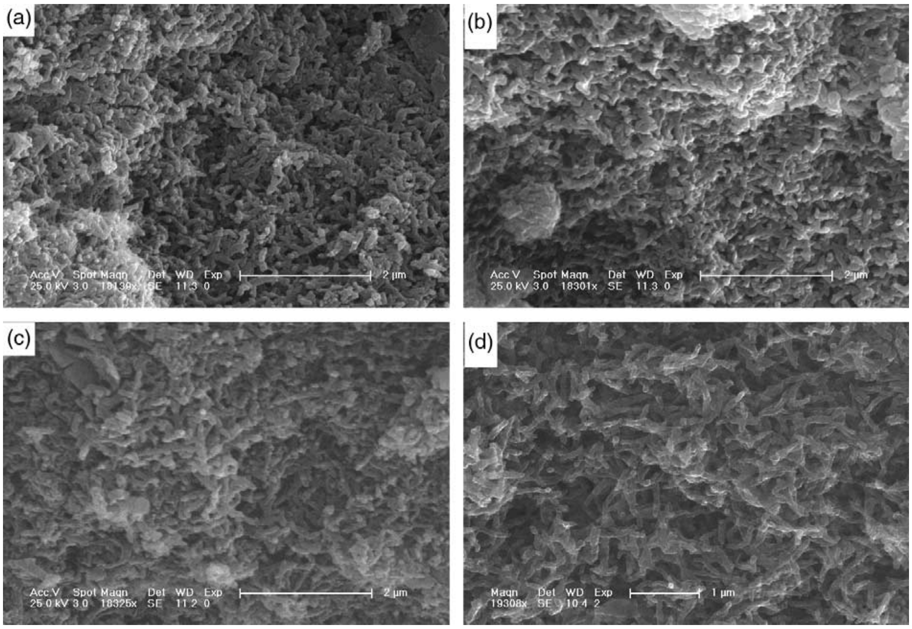

Fig. 7. SEM images of PANI obtained from different 'seed' concentration (a) 0.1 mg/mL; (b) 0.2 mg/mL; (c) 0.5 mg/mL; (d) 1 mg/mL.

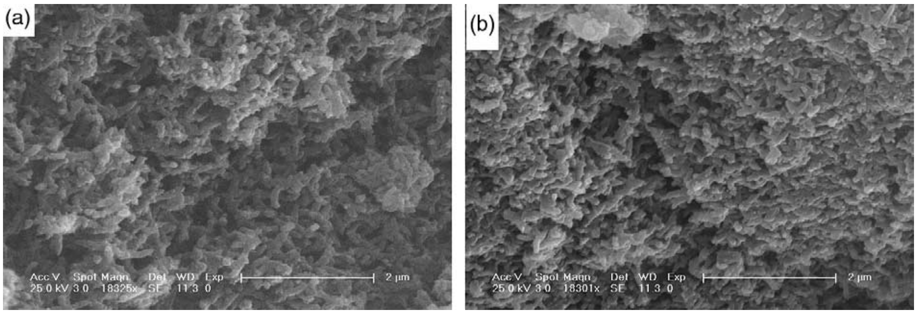

Fig. 8. SEM images of PANI obtained from different acid kinds (a) H_2SO_4; (b) H_3PO_4.

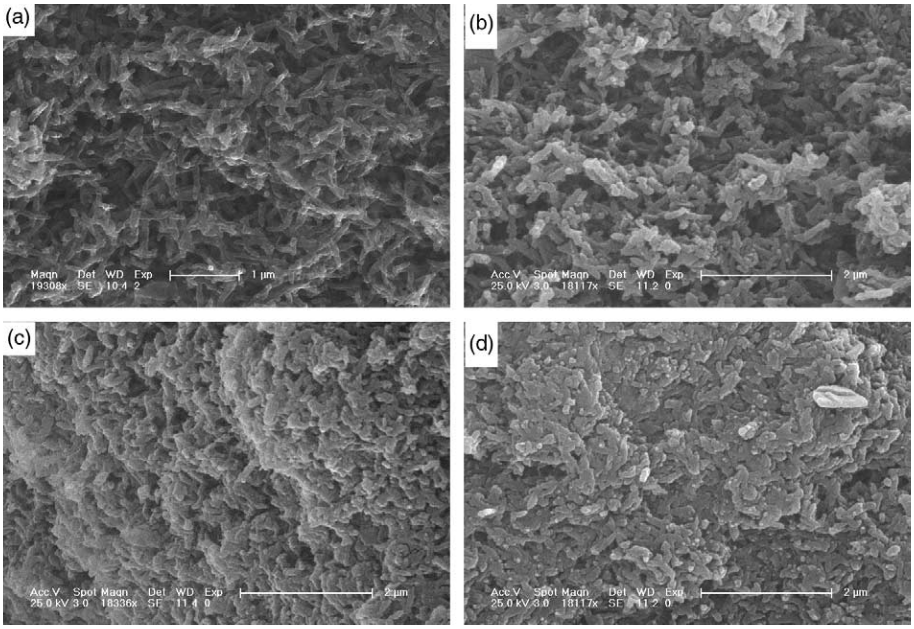

Fig. 9. SEM images of PANI obtained from different organic solvents (a) DMSO; (b) DMF; (c) DMAc; (d) NMP.

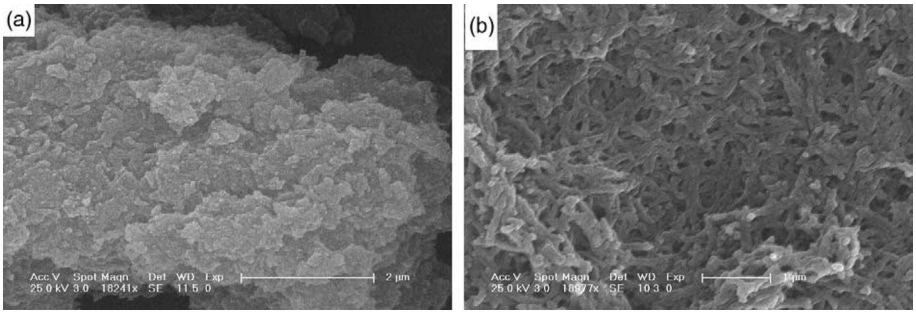

Fig. 10. SEM images of PANI obtained from different approaches (a) conventional polymerization; (b) rapidly mixing polymerization.

crystallization. As shown in Fig. 11, the XRD pattern of the sample 4 with the number listed in Table 1, the main diffraction peaks of the nanofibers are indicated with arrows and indexed, which showed the PANI was partly crystallized [23]. However, it did not show much difference with the conventional polymerized doped PANI. Then it could be learned that this high conductivity was not mainly resulted from a high crystallinity. On the other hand, the FT-IR spectra of PANI from conventional polymerization and our approach (sample 4 in Table 1) are shown in Fig. 12, the peaks at ca. 1600 and 1500 cm^{-1}, which are assigned to C=C stretching of the quinoid and benzenoid rings, respectively and the peak at ca. 1140 cm^{-1} representing C–H aromatic in-plane bending have all shifted to lower frequencies. The peak at about 1140 cm^{-1} is related to the doped structure; therefore, the result signified that PANI using our 'seed' polymerization was more protonated than that from the conventional polymerization and a higher doping level would lead to a higher conductivity. Besides, we believe the nanofibrillar morphology must attribute to the high conductivity due to its orientation.

The conductivity of the samples obtained from different conditions are listed in Table 1 and the FT-IR data of the samples are also shown in Table 2 as reference.

3.2.1. The effect of acid concentration

As can be seen from Table 1, the conductivity of the samples increased with the acid concentration from 0.84 S cm^{-1} in 0.01 M acid solution to 34.5 S cm^{-1} in 1 M acid solution because a higher acid concentration would lead to a higher doping level. As for the FT-IR spectra, the peaks have all showed red shift with different degree as shown in Table 2.

3.2.2. The effect of reaction time

The reaction time seemed having little effect on the conductivity with all values near 30 S cm^{-1} because the introduction of the 'seed' greatly shortened the induction time and hence the polymerization time. The FT-IR spectra also presented similar results with the peaks all around ca. 1110 cm^{-1}, which indicated a good doping level.

3.2.3. The effect of aniline concentration

There was improvement of the conductivity of the samples when the aniline concentration was increased from 0.025 M (7.4 S cm^{-1}) to 0.1 M (34.5 S cm^{-1}), but a decrease occurred as the concentration became 0.2 M with the conductivity of 7.7 S cm^{-1}. Here, we considered the morphology of the samples might play a role because apart from the changing of the doping level as shown in Table 2, the sample obtained from 0.2 M aniline concentration gave apparent aggregation (Fig. 5(d)), which might decrease the conductivity because ordered arrangement of the polymer chains favors higher conductivity. On the other hand, when they had the same amount of 'seed', the system with lower aniline concentration would probably lead to disorder in orientation than that with higher aniline, which might decrease the conductivity.

3.2.4. The effect of 'seed' amount and concentration

The conductivity of the samples obtained from different amount or concentration of seed did not appear much to change with the value around 20 S cm^{-1} except the one from the system with the 'seed' concentration of 0.1 M, which showed a relatively lower value of 8.16 S cm^{-1}. We considered though the seed was different either in amount or concentration, it had already made effect on the formation of the PANI nanofibers. Once the nanofibers had been formed, they would orchestrate the whole polymerization of the aniline. Besides, the high conductivity must be related to the nanofiber morphology because there was no apparent red shift based on the FT-IR data listed in Table 2.

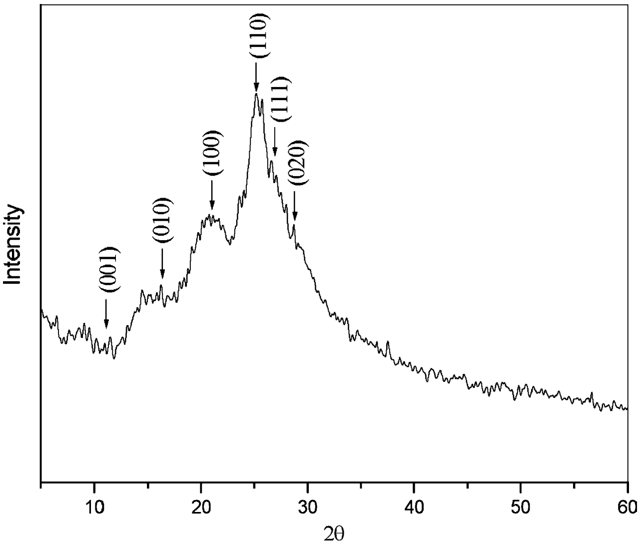

Fig. 11. XRD pattern of PANI (sample 4 listed in Table 1).

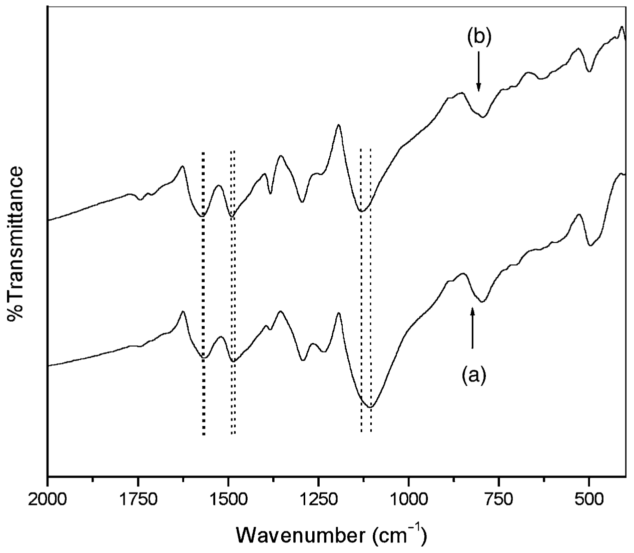

Fig. 12. FT-IR spectra of PANI (a) ‘seeding’ polymerized PANI and (b) conventionally polymerized PANI.

3.2.5. The effect of acid kinds

Typically the PANI synthesized from H_2SO_4 or H_3PO_4 solution had conductivity a bit lower than that from HCl solution with the similar conditions [24,25]. In our experiment, the samples using H_2SO_4 and H_3PO_4 as mediums showed higher conductivity than those obtained from the conventional approaches with the conductivity of 7.4 and 13.04 S cm^{-1}, respectively. As can be seen from Table 2, the main peaks of the two samples did not shift to a much lower frequencies, which indicated that there was little improvement in the doping level of these samples. Then the homogeneous nanostructured morphology (Fig. 8(a) and (b)) would be benefit for the higher conductivity.

Table 2
FT-IR data of different samples (the numbers are consistent with those in Table 1)

No	Main peak position (cm^{-1})			
1	1581	1501	1147	826
2	1572	1489	1138	803
3	1558	1485	1123	797
4	1562	1485	1107	795
5	1558	1485	1110	793
6	1554	1485	1109	795
7	1572	1489	1132	798
8	1573	1490	1134	799
9	1561	1485	1130	798
10	1558	1487	1126	799
11	1559	1486	1124	797
12	1557	1486	1122	795
13	1558	1483	1135	803
14	1562	1483	1130	796
15	1556	1485	1120	794
16	1562	1490	1130	803
17	1562	1491	1131	803
18	1562	1489	1130	803
19	1557	1492	1130	803
20	1557	1489	1138	796
21	1574	1490	1137	804
22	1561	1485	1126	794

3.2.6. The effect of organic solvent kinds

We have already discussed above that DMF, DMAc and NMP were not quite fit for this ‘seeding’ polymerization because they are strong alkali. We found the samples using these solvents showed lower value in conductivity compared with those using DMSO. Meanwhile, the FT-IR spectra showed little red-shift in these samples as shown in Table 2.

3.2.7. The effect of approaches

The conventional polymerization resulted in a sample with conductivity of ca. 5 S cm^{-1} as many papers reported and the sample from rapidly mixing polymerization showed similar value though it had homogeneous nanofiber morphology, which could be concluded that using the latter approach could not improve the doping level of the PANI as our approach did and their morphology perhaps did not have a beneficial orientation for the improvement of the conductivity as we discussed above in Section 3.1.3.

4. Conclusions

In summary, homogeneous PANI nanofibers with high conductivity were prepared via ‘seeding’ polymerization, in which the conventional polymerized PANI powders dissolved in DMSO were used as ‘seed’. The morphology of the samples prepared under different conditions were studied and the results showed lower acid concentration, PANI/DMSO solution amount and PANI/DMSO solution concentration would all resulted in aggregation, though the later two were not as serious as the former one. On the other hand, the size of the obtained nanofibers increased with the aniline concentration, but it did not change much along with the reaction time. Different kinds of acid like H_2SO_4 or H_3PO_4 as reaction medium showed little influence on the morphology of the obtained samples, however, various organic solvent used for dissolving PANI powder as ‘seed’ gave different results because of their different alkalescence. The conductivity of the samples was mainly affected by the doping level and the morphology. PANI with higher doping level as shown in FT-IR spectra and more ordered morphology would give higher conductivity.

Acknowledgements

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