Thermoelectric properties of poly (3,4-ethylenedioxy thiophene)/poly (sulfonic acid diphenyl aniline) composites synthesized in the absence and presence of magnetic field

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Abstract

Poly(3,4-ethylenedioxy thiophene)/polysulfonic diphenyl aniline (PEDOT:PSDA) composites in the ratios of 1:0.5, 1:1, 1:1.5 and 1:2 were synthesized by oxidative chemical polymerization in the absence and presence of external constant magnetic field which the flux density is 3 kGa. The samples were characterized by UV–vis, FTIR-ATR and XRD. Their thermoelectric properties were obtained by means of electrical conductivities and Seebeck coefficients measurements. It was found that both the conductivity and Seebeck coefficients of the PEDOT:PSDA composites were increased in the presence of magnetic field. The conductivities of PEDOT:PSDA composites are lower whereas their Seebeck coefficients are considerably higher than that of commercial PEDOT:PSS. The highest power factor was obtained as 32 μW m⁻¹K⁻² for the composite PEDOT:PSDA = 1:1 synthesized in the presence of magnetic field. The power factor of this sample was enhanced to 185 μW m⁻¹K⁻² by a secondary doping with sodium dodecyl benzene sulfonate.

1. Introduction

Many forthcoming applications such as flexible solar cells, touch screens and wearable electronics require flexible, highly conductive, transparent, printable, and low-cost-production electrodes [1]. The most promising candidate as a hole-injecting electrode instead of hard indium tin oxide is poly (3,4-ethylene dioxide) (PEDOT) which is almost transparent, highly conductive and stable in thin films [2]. 3,4-ethylene dioxythiophene (EDOT) can be easily polymerized in the presence of an oxidizing agent in a poly (styrene sulfonate) (PSS) solution to give a dark blue aqueous dispersion including the polyelectrolyte complex gel particles of PEDOT:PSS [3]. PSS behaves as a template for PEDOT during polymerization and also as a dispersant in use. Sulfonate anionic groups of PSS are the counterions used to provide for electroneutrality of the positive charge carriers of PEDOT, occurring during the polymerization reaction. A large number of commercially available PEDOT:PSS dispersions are available on the market for use in a wide variety of applications. Since the as-prepared PEDOT: PSS films from its aqueous solution have a lower conductivity, around 1 S cm⁻¹, it has been discovered that the conductivity of PEDOT:PSS can be increased by exposing secondary doping. At first, Kim et al succeeded in the conductivity enhancement of PEDOT:PSS by adding dimethyl sulfoxide or dimethylformamide into PEDOT: PSS aqueous solution [4]. Later, polar organic solvents such as alcohols, ethylene glycol, N-methyl pyrrolidone, sorbitol [5], some inorganic acids [6], and some organic salt solutions [7] were used as secondary dopants to achieve a conductivity enhancement by up to 4 orders of magnitude [8]. This remarkable result is thought to be due to the PEDOT chain alignment, depletion of insulating PSS shell and growth of the PEDOT domains.

Besides the various research fields, PEDOT:PSS is one of the most promising p-type semi-conductive polymer in the thermoelectric (TE) energy harvesting field [9]. TE materials can directly convert the heat to electricity, or vice versa. TE devices made of TE materials can be either used as power generators or be utilized as solid-state coolers/heaters. TE materials have been received great attention due to their several advantages such as no moving parts, a noiseless and long operating lifetime. The energy conversion efficiency of a TE device is...
modules currently on the market are made of scarce semiconductor metals and elements restrict the sustainable development of the inorganic TE materials in the future. The inorganic TE antimony, selenium and their nanostructures have reached high ZT values above unity at room temperature. However, the problems including the rigidity, scarcity, high costs of the raw materials, the toxicity of elements restrict the sustainable development of the inorganic TE materials in the future. The inorganic TE modules currently on the market are made of scarce semiconductor metals and/or harmful metals to the environment. Because of the disadvantages of inorganic TE materials, a great effort has been made in the development of polymeric TE materials. Instead of ZT, the power factor is used as a metric of efficiency to be improved in the polymeric TE materials since measurement of thermal conductivity can be challenging.

Furthermore, polymers are generally poor thermal conductors, and have similar thermal conductivity (0.1–0.3 Wm\(^{-1}\)K\(^{-1}\)) [13–15]. Among the conductive polymers, the PEDOT:PSS was the most promising polymer as a flexible and safe TE material since Kim et al achieved the highest reported ZT of 0.42 (power factor of 469 \(\mu\)W m\(^{-1}\)K\(^{-2}\)) for a commercial PEDOT:PSS films at room temperature [16]. The PSS is present in excess and behaves as an insulator which reduces the conductivity of the PEDOT:PSS. The PEDOT chains are probably attached ionically along with PSS random coils consisting of a few hundred monomer units [17]. The conducting PEDOT domains are possibly wrapped up by nonconducting PSS chains. Selective removal of PSS is one of the mechanisms to enhance conductivity. Kim et al reported a dipping method for removing PSS from the PEDOT:PSS film using EG and DMSO to achieve the record value [16].

Lee et al reported a chemical doping/de-doping method on PEDOT:PSS films [18]. Doping with DMSO/TSA increases the electrical conductivity and de-doping with DMSO/hydrazine selectively removes the insulating barrier of PSS and controls the oxidation level of PEDOT. It was found that the electrical conductivity increased by three orders of magnitude through PEDOT chain alignment, depletion of insulating PSS shell and overall growth of the conducting domains.

Bae et al reported significant enhancement of the electrical conductivity of PEDOT:PSS from 787.99 to 4839.92 S cm\(^{-1}\) by simple chemical treatment with \(H_2SO_4\) [6]. The increase was attributed to the increasing carrier concentration and improved crystallinity of PEDOT due to the removal of PSS, which induces the formation of a more crystalline structure. The depletion of PSS by treatment of \(H_2SO_4\) in various concentrations was shown by XPS analysis of the PEDOT:PSS films.

On the other hand, it was known that the properties of conducting polymers synthesized by electropolymerization can be changed due to the alignment of the molecule structures in a specific direction depending on the applied external magnetic field (MF) [19]. Ma et al showed that the electrical conductivity of polyaniline increased from 0.14 to 0.28 S cm\(^{-1}\) when synthesized in the presence of 0.4 T MF [20]. Cho et al observed that the polymerization rate and yield increased if an external MF is applied during electropolymerization of PEDOT [21]. The Raman spectroscopy provided that conformation of polymer became more extended in the presence of the parallel MF. In the SEM images, the PEDOT samples synthesized under the MF have sharp crystalline structured morphology. Guziak et al reported that PEDOT:PSS films prepared from commercial dispersions under the MF showed clear anisotropy of DC resistivity resulting from the alignment of nano-crystals of PEDOT films by the MF [22]. It was believed that MF increases single-triple transitions in favor of triple, and triple radicals are expected to be less willing to reunite on pairing in the presence of MF [23–25]. Since the mean lifetime of radicals in polymerization reactions becomes longer and termination reactions are delayed, the chain length and orientation of the polymer chains synthesized under MF are expected to increase. It has been explained theoretically as well as experimentally that carrier mobility and consequently electrical conductivity is increased by molecular weights of the conductive polymers [14, 15, 26]. It was reported that the polymers synthesized in an external MF demonstrate increased thermal stability and lower swelling due to their regulated structures. It was found that imprinted polymers synthesized under MF were more rigid and ordered [27].

Recently, we studied the effect of the external MF on thermoelectric properties of polythiophene synthesized by chemical oxidation [28]. It was observed that the conductivity increased slightly, however, Seebeck coefficient increased considerably by the increase of flux density of the constant MF. Furthermore, the conductivity and power factor of the samples synthesized under the MF increased significantly by secondary doping with various substances.

In this study, we aimed to synthesize a new aqueous PEDOT dispersion by using moderately conductive polymer, namely, poly (sulfonic acid diphenyl aniline) (PSDA) instead of non-conductive PSS. The PSDA is a water-soluble polymer which the film conductivity is between 2.3 \(\times\) 10\(^{-2}\) and 1.3 S cm\(^{-1}\) [29]. However, since the conductivities of PEDOT:PSDA films are not high enough, we tried to increase their conductivities by...
applying an external MF during synthesis. In addition, we exposed a secondary doping to the PEDOT:PSDA composite with various substances to enhance its TE performance.

2. Materials and methods

2.1. Materials

3,4-Ethylenedioxythiophene (EDOT) was purchased from Acros Chemicals. Sodium diphenylamine 4-sulfonate (SDAS) used in PSDA synthesis, PEDOT:PSS (1.3 wt% dispersion in H2O, conductive grade, #483095), ethylene glycol (EG), and sodium dodecyl sulphate (SDS) were purchased from Sigma Aldrich. Anhydrous iron (III) chloride (FeCl3), hydrochloric acid (HCl)(37%) and analytical grade ethanol, dimethyl sulfoxide (DMSO) were purchased from Merck. All chemicals were used without purification.

2.2. Synthesis of PSDA

As described in the literature [29, 30], PSDA was synthesized by oxidative chemical polymerization of sodium diphenyl amino-4-sulfonate with ammonium persulfate at a ratio of Oxidant/Monomer: 1:2 in a medium of 1.2 N HCl at 4 °C. The reaction proceeded for 16 h and was ended by the addition of ethanol.

2.3. Synthesis of PEDOT:PSDA composites

PEDOT was synthesized by the chemical polymerization reaction of EDOT in the presence of PSDA which was used as both templating and dispersing agent. The masses of PSDA per mass of PEDOT were varied such as 0.5, 1.0, 1.5 and 2.0. Briefly, 0.25 ml of EDOT was added into the 20 ml aqueous solution including appropriate amounts of PSDA, and then the solution was stirred for 1 h. The oxidative polymerization reaction was started by the dropwise addition of FeCl3 (3 moles per mole of EDOT) in 5 ml of deionized water within 20 min, then by stirring for 16 h at ambient conditions. The same reactions were also conducted by exposing the reaction vessel to external MF which flux density was set at 3 kGa. The polymerization reaction was briefly presented in figure 1. The dispersions of PEDOT:PSDA synthesized either presence or absence of MF were dark greenish blue and more viscous compared to PEDOT:PSS dispersion.

2.4. Secondary doping

The PEDOT:PSDA composite was exposed to a secondary doping by adding sodium dodecyl benzene sulfonate (SDS), ethylene glycol (EG) and dimethyl sulfoxide (DMSO) into the dispersion. The concentration of PEDOT:PSDA in the dispersion was 1.0 mg ml⁻¹ and the concentration of dopants were 5% by mass [28]. The film of the doped dispersion was obtained by drop casting on the cleaned glass substrate and then by drying in the oven at 100 °C for 12 h.

2.5. Characterization

The synthesized composites were characterized by UV–vis, Attenuated total reflection accessory attached Fourier-Transform Infrared (FTIR-ATR) and x-ray diffraction (XRD) spectroscopy techniques. UV–vis
absorption measurements were carried out on a Shimadzu UVmini-1240 UV–vis spectrophotometer. FTIR measurements were carried out on a Thermo Fisher Scientific Nicolet IS10 FTIR-ATR Spectrometer. XRD patterns of the polymer powder were recorded on a T&T TT-90 x-ray diffractometer, operated at 30 MA and 40 KVP MAX. Elemental mapping of composite films was carried out on a scanning electron microscopy with energy dispersive x-ray spectrometry (SEM-EDX) using scanning electron microscope (Zeiss EVO® LS 10). Conductivity and Seebeck coefficient were measured by Entek Electronic FPP 470 Four Point Probe and Seebeck Coefficient Measuring System, respectively.

3. Results and discussion

3.1. UV–vis analysis
UV–vis absorption spectra of the composites are illustrated in the figure 2. The numbers 1, 1.5, and 2 indicate the mass of PSDA per mass of PEDOT while the tag MF indicates the corresponding composition synthesized in the presence of magnetic field. All of the composites display a strong absorption peak around 400 nm which is assigned as $\pi \rightarrow \pi^*$ electronic transition. The samples synthesized under MF display free carrier tailing beyond the 600 nm which is assigned as delocalized polaron-bipolaron transition [31]. It can be seen that the free carrier tail absorptions of the composites synthesized under MF are higher than their counterparts and tends to extend to the IR region by monotonically increasing. The highest absorbtion among free carrier tails is seen in the composite having a ratio of PEDOT:PSDA $= 1:1$ which is synthesized both in the absence and in the presence of MF. However, it is obvious that the amount of free charge carrier in the composites considerably increases in the presence of MF. This suggests that the amount of delocalized polarons and bipolarons is increased considerably if an external MF is exposed during their synthesis.

3.2. FTIR-ATR analysis
In the figure 3, the FTIR spectra of the composite PEDOT:PSDA $= 1:1$ synthesized in the presence (1MF) and absence (1) of MF together with pristine PSDA were given as an example since the highest UV–vis. absorption of free carrying tail was displayed for this composition (figure 2). For PSDA, absorption bands at 1610 cm$^{-1}$ and 1430 cm$^{-1}$ are related to quinoid and benzenoid ring vibrations, respectively. The peaks at 1185 cm$^{-1}$ and 1030 cm$^{-1}$ are associated with S=O symmetric and asymmetric vibrations. The ratio of quinoid and benzenoid bands indicates the degree of oxidation of the polymer. The spectrum of PSDA suggests that the polymer is in the form of pernigraniline since the intensity of quinoid band at 1610 cm$^{-1}$ is higher than that of benzenoid band at 1430 cm$^{-1}$ [29, 30]. For the composite 1, the peak area of quinoid becomes close to that of benzenoid suggesting the formation of emeraldine form of PSDA. Also, C=O asymmetric stretching and C–C inter-ring stretching bands belonging to PEDOT appeared at 1495 cm$^{-1}$ and 1380 cm$^{-1}$ respectively. The peak at around 900 cm$^{-1}$ corresponds to C–S stretching in the thiophene ring [32, 33].
It can be seen that the vibration bands of the composite 1 between 1000 cm$^{-1}$ and 1500 cm$^{-1}$ become broader if it is synthesized under magnetic field (1MF). This situation might indicate more intensive attractive intermolecular interactions between the chain segments of the sample 1MF compared to the sample 1. It can be stated that thiophene rings become a dense structure in the sample 1MF. It can be seen that some peaks of sample 1 were disappeared in the sample 1MF, i.e. the peaks at 1495 cm$^{-1}$ and 750 cm$^{-1}$ belonging to C$=$C and C–S stretching vibrations of of thiophene ring. This may also indicate strong favorable interaction between the components.

3.3. XRD analysis

XRD spectra of the composite PEDOT:PSDA = 1:1 synthesized in the absence (1) and the presence (1MF) of MF are given in figure 4. In the patterns, broad peaks at 25° are assigned to the amorphous origin of PEDOT [34]. The peak intensity becomes higher in the sample 1MF. However the significant difference between the spectra is the sharp peak at 10.81°. This sharp peak at 10.81° can be attributed to the alignment of the polymer chains in such a way that the regular sequence of repeating units can enter a crystalline network [35]. The crystalline structure of 1MF explains the broader IR bands of the sample 1MF compared to the sample 1 in the figure 3. It is obvious that the external MF causes crystallinity of PEDOT:PSDA.
3.4. SEM-EDX analysis

Figure 5 illustrates the SEM and energy dispersive x-ray (EDX) mapping images of the elements in thin composite films of 1MF and 1. SEM images reveal that the composite 1MF displays more rigid and well-stacked spherulite-like morphology compared to the composite 1. In considering the XRD spectra of the samples, it can be stated that the spherulitic morphology exhibits the crystallinity of the sample 1MF. It was found that the polymers synthesized under MF are more rigid and ordered structure in the literature [27]. EDX mapping images were given to reveal the distribution of N, O and S atoms in the composites. The images show the homogeneously distribution of the elements in elements in both of the samples. Meanwhile, the homogeneously distribution of N reveals that the polymers of the composites is mixed very well since N is included only in PSDA.

3.5. Thermoelectric properties

The conductivity and Seebeck coefficient measurements were carried out on the films of pristine PSDA and its composites with PEDOT in different ratios at the ambient conditions. Then, the power factor of the samples were calculated by multiplying the conductivity with the square of Seebeck coefficient. The values of measured conductivity and Seebeck coefficient and the calculated power factor were given in the figure 6. For comparison, the thermoelectric properties of the film sample of a commercial product of PEDOT:PSS which is purchased from Sigma Aldrich (Product # 483095) were also measured in our laboratory and given as a star symbol in the figure 6. The conductivity values in the figure 6(a) were plotted in a logarithmic scale since the differences were high. It can be seen that the conductivities of PEDOT:PSDA composites are considerably low from the commercial on average. The highest conductivity was measured as 0.13 S cm\(^{-1}\) for the sample 1MF while that of commercial product is 1.38 S cm\(^{-1}\). It can be stated that the rigid main chain of PSDA reduces the conductivity of PEDOT. However, it can be seen from figure 6(b) that the Seebeck coefficients of PEDOT:PSDA are...
considerably larger than that of the commercial PEDOT:PSS, which is 40 μV K⁻¹. In addition MF causes approximately 2 times increase of Seebeck coefficients of PEDOT:PSDA composites. It can be seen that the power factor of the composites synthesized in the presence of MF are considerably higher compared to their counterparts, especially for the composites of PEDOT:PSDA = 1:1 (i.e. 32 μW m⁻¹K⁻² and 1.8 μW m⁻¹K⁻² for...
1MF and 1, respectively). In the literature the power factor of pristine PEDOT:PSS was reported as 0.016 μW m⁻¹K⁻². By secondary doping, the power factor of the PEDOT:PSS was increased to the values changing from 7.0 μW m⁻¹K⁻² to 469 μW m⁻¹K⁻² (16). In table 1, our results were compared with the power factors reported in the literature given by Bharti et al[15].

3.6. Effect of secondary doping on thermoelectric properties

Since the electrical conductivity of PEDOT:PSS increase considerably by a secondary doping with high boiling point solvents such as DMSO and EG and surfactants [8], the composite 1MF was subjected to secondary doping by using sodium dodecyl benzene sulfonate (SDS), ethylene glycole (EG) and dimethyl sulfoxide (DMSO) and the results were given in figure 7. It was observed that the conductivity of the composite 1MF changed from 0.13 S cm⁻¹ to 1.42 S cm⁻¹, 0.12 S cm⁻¹ and 0.024 S cm⁻¹, whereas the Seebeck coefficient changed from 1700 μV K⁻¹ to 1150 μV K⁻¹, 1240 μV K⁻¹ and 1400 μV K⁻¹ by doping with SDS, EG and DMSO, respectively. Then, power factor of the composite 1MF was calculated as 185 μW m⁻¹K⁻², 18.45 μW m⁻¹K⁻² and 4.7 μW m⁻¹K⁻² by doping with SDS, EG and DMSO, respectively. The results show that the power factor was enhanced from 52 μW m⁻¹K⁻² to 185 μW m⁻¹K⁻² by subjecting the composite 1MF to a secondary doping with SDS. The conductivity of PEDOT:PSDA did not increase by doping with DMSO and EG while increased ten times by doping with SDS. In the case of PEDOT:PSS; it is believed that the increase in conductivity by a secondary doping with high boiling point solvents is due to the partial removal of PSS from PEDOT:PSS and the fact that PEDOT chains reach an extended conformation with solvent effect [8]. It can be stated that PSDA cannot be removed by DMSO and EG and also PEDOT cannot be reached an extended conformation since PSDA cannot be solved in DMSO and EG. In addition, it may be considered that the PEDOT chains cannot be

Table 1. Comparison the power factors of PEDOT:PSDA obtained in this study with those of PEDOT: PSS selected from the literature.

| Polymer          | Dopant          | Power Factor (μW m⁻¹K⁻²) | References |
|------------------|-----------------|-------------------------|------------|
| PEDOT:PSDA 1:1   | —               | 1.8                     | this study |
| PEDOT:PSDA 1:1   | SDS             | 184.54                  | This study |
| PEDOT:PSS        | DMSO            | 12.5                    | [36]       |
| PEDOT:PSS        | DMSO + EG       | 469                     | [16]       |
| PEDOT:PSDA 1:1   | EG              | 351                     | [16]       |
| PEDOT:PSDA 1:1   | Sorbitol        | 22.28                   | [37]       |
| PEDOT:PSDA 1:1   | Formic acid     | 80.16                   | [39]       |
| PEDOT:PSDA 1:1   | Methanol        | 58                      | [39]       |

Figure 7. Conductivity (a), Seebeck coefficient (b) and power factor (c) values of 1MF doped with SDS, EG, and DMSO.
reach a more extended conformation since the PSDA has a rigid aromatic backbone and can undergo self-doping. In the SDS doping, the mechanism might be different [40]. It is possible that the ten-fold increase in conductivity is due to the fact that SDS dopped PEDOT as counterion instead of PSDA.

4. Conclusions

The enhancement of thermoelectric properties of the PEDOT:PSDA composites in various ratios was investigated by an exposing the composite to external magnetic field and then secondary doping. The following results are reached.

- The stable and viscous aqueous dispersions were synthesized by oxidative chemical polymerization of EDOT in the presence of PSDA.
- The films of aqueous dispersions of PEDOT:PSDA are transparent but darker than that of PEDOT:PSS.
- The free carrier tailing in the UV–vis region of polaron-bipolaron transition in the composites synthesized under magnetic field considerably higher than their counterparts.
- FTIR and XRD spectra suggest that magnetic field cause a crystallinity in the PEDOT:PSDA composite.
- Both the conductivity and the Seebeck coefficient of the PEDOT:PSDA composites enhance by magnetic field.
- The power factors of the composites synthesized under magnetic field are considerably higher than those of their counterparts as well as the commercial PEDOT:PSS.
- The power factor of the composite PEDOT:PSDA  = 1:1 synthesized under magnetic field was enhanced from 32 $\mu$W m$^{-1}$K$^{-2}$ to 185 $\mu$W m$^{-1}$K$^{-2}$ by a secondary doping with SDS.

We are planning to prepare hybrid composites of the PEDOT:PSDA with some inorganic thermoelectric nanoparticles in the future works.

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