Preparation of polyaniline/graphene coated wearable thermoelectric fabric using ultrasonic-assisted dip-coating method

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Abstract

The use of thermoelectric fabrics for powering wearable devices is expected to become widespread soon. A thermoelectric fabric was prepared by coating nanocomposite of polyaniline/graphene nanosheets (PANI/GNS) on a fabric. Four samples of the fabric containing different wt% of GNS (0.5, 2.5, 5, and 10) were prepared. To characterize the samples, Fourier transform infrared (FTIR) spectra, attenuated total reflectance-Fourier transform infrared (AT-FTIR) spectra, field-emission scanning electron microscopy (FE-SEM), electrical conductivity and Seebeck coefficient measurements were used. The electrical conductivity increased from 0.0188 to 0.277 S cm⁻¹ (from 0.5 to 10 wt% of the GNS in PANI/GNS nanocomposite). The maximum coefficient of Seebeck was 18 μV K⁻¹ with 2.5 wt% GNS at 338 °C. The power factor improvement was from 2.047 to 3.084 μW m⁻¹ K⁻² (0.5–2.5 wt% GNS).

Graphic abstract

Keywords Thermoelectric · Nanocomposite · Graphene · Conductive polymer · Polyaniline

Extended author information available on the last page of the article

Introduction

Considering the population growth and significant increases in welfare of most populated countries, the energy crisis has become an ever more crucial phenomenon. There are several solutions to this crisis such as solar cells, biomass, wind, and so on [1]. Another solution involves recycling of waste energies and inventing energy-harvesting technologies that can harvest various forms of energy from the environment dynamically and convert it to electricity. Recently, piezoelectric and thermoelectric materials have been used in this regard. For instance, as the Internet of Things (IoT) becomes more popular, the need for advance sensors will also become more and more crucial [2–4]. Most of the waste energy is basically the thermal energy from factories, transportation vehicles and residential units. Thermoelectric materials can convert heat to electricity directly. This phenomenon was first observed by Seebeck in 1821 [5], and is characterized by the formula $ZT = S^2\sigma T/\kappa$, where S , σ , κ and T , stand for Seebeck coefficient, electrical conductivity, thermal conductivity, and absolute temperature, respectively [6]. Another method to describe thermoelectric property is the power factor ($PF = S^2\sigma$) [7].

As in semiconductors, the thermoelectric materials are also divided into two types; n-type and p-type. In the n-type thermoelectrics, the current generation results as the electrons move to the cold side, while in the p-type, the current generation takes place as the holes move to the cold side. Among the most important inorganic materials that have ever been used in thermoelectrics are Bi_2Te_3 , PbTe , GeTe , and Sb_2Te_3 [8–13]. However, the use of these materials has its own limitations such as high cost and low flexibility as well as the complexity of the production methods [14–18].

In contrast, the organic thermoelectric materials have special properties such as low density, low thermal conductivity, easy to formulate, etc. [19–21]. The main conductive organic polymers with high potential for thermoelectric use include poly (3,4-ethylenedioxythiophene) (PEDOT), polyaniline (PANI), polythiophene (PTh), polypyrrole (PPy), polyacetylene (PA), and polycarbazole (PC). Different thermoelectric composites of these polymers reinforced with particles have been constructed in recent years [22]. In 2013, Zhang et al. [23] worked on PEDOT:poly(styrenesulfonate) (PEDOT:PSS) reinforced with graphene oxide particles and obtained power factor of $32.4 \mu\text{W m}^{-1} \text{K}^{-2}$ (300 K) at 21 wt% of graphene oxide, although in 2012 Kim et al. [24] had achieved power factor of $11.09 \mu\text{W m}^{-1} \text{K}^{-2}$ (300 K)

with only 2 wt% graphene particles. In 2013, Lu et al. [25], using PANI and 30 wt% graphene nanosheets (GNS), achieved power factor of $2.6 \mu\text{W m}^{-1} \text{K}^{-2}$ (453 K).

The aforementioned cases focused on construction of thermoelectric composites, but recently, researchers have been studying the topic of wearable thermoelectric materials. In 2015, Du et al. [26] coated PEDOT:PSS on polyester fabric and in 2017, Yong Du et al. [27] coated PEDOT:PSS and 15 wt% graphite on polyester fabric achieving $0.025 \mu\text{W m}^{-1} \text{K}^{-2}$ power factor.

In this research work, we fabricated PANI/GNS coated polyester/linen fabric and studied its characteristics. Four samples of the fabric containing different wt% of GNS (0.5, 2.5, 5, and 10) were prepared. The materials and methods used in this work are scalable and can easily be commercialized. Besides the advantage of affordable materials and methods, the results also showed that the Seebeck values for the produced samples are comparable with those of other works reported in the literature.

Materials and methods

Synthesis grade aniline monomer double distilled under vacuum and stored at 0 °C from Sigma-Aldrich, ammonium peroxydisulfate (APS) from Sigma-Aldrich, GNS from Daejeon, South Korea, hydrochloric acid (HCl) and acetone from Merck, deionized (DI) water, commercial polyester and yarn fabric were used in this study.

Preparation of PANI/GNS composites

Polymerization

Initially GNS was dispersed in 1 M HCl by ultrasonic horn for 30 min at room temperature. Then aniline monomer was added to the HCl/GNS suspension and was stirred for 1 h under nitrogen atmosphere at 0–4 °C. APS was dissolved in 1 M HCl and was added to the HCl/GNS/aniline suspension slowly (to prevent flash polymerization) and was continuously stirred for 6 h in nitrogen atmosphere at 0–4 °C. Finally, it was kept at 0–4 °C for 24 h without any movement to complete polymerization. The completely polymerized product was centrifuged for 15 min to obtain pure PANI/GNS composite nanoparticles and eliminate the undesirable aniline oligomer. Then acetone was added to the composite nanoparticles and centrifuged for 15 min again, and the extra

Fig. 1 **a** Schematic of nano-composite synthesis process. **b** Schematic of PANI/GNS composite nanoparticles synthesis

diluent was removed. This was repeated twice. Finally, a suspension was made by dispersing PANI/GNS composite nanoparticles (PANI/GNS suspension) in acetone by stirring. Schematic of nanocomposite synthesis process and schematic of PANI/GNS composite nanoparticles synthesis are shown in Fig. 1a and b, respectively.

Preparation of PANI/GNS coated fabric

The as-received fabric was cut into several strips (20 mm × 10 mm). All strips were washed in DI water to remove any contamination. For coating, the fabric strips were dropped in PANI/GNS suspension and sonicated for 1 h. Then the coated fabric strips were removed from the PANI/GNS suspension and dried in an oven at 60–70 °C for 15 min. These steps were repeated twice for achieving the final product (thermoelectric fabric nanocomposite).

Characterization

The surface morphology of the thermoelectric fabric samples was studied by field emission scanning electron microscopy (FE-SEM) model TEScan-Mira III. To verify the desired synthesis of nanocomposites, Fourier-transform infrared spectroscopy (FTIR) was used. Attenuated total reflection (ATR) test was used to demonstrate the presence of nanocomposite on the fabric. A standard four-probe method was used for the electrical conductivity measurements. A Keithley 487 picoammeter/voltage source was used for the measurements. Seebeck coefficient of the samples was measured by an automatic apparatus described comprehensively in [28, 29].

Fig. 2 FTIR spectra of PANI and PANI/GNS nanocomposites: **a** 0.5 wt% GNS, **b** 0 wt% GNS, **c** 2.5 wt% GNS, **d** 5 wt% GNS, **e** 10 wt% GNS

Results and discussion

Weight measurement revealed that the percentage of PANI/GNS on the fabric surface was proportionate in all four samples containing GNS. The FTIR spectra of PANI and PANI/GNS are shown in Fig. 2. In the region 400–4000 cm^{-1} , there are five main peaks at wavelengths of 802, 1145, 1303, 1508, and 1597 cm^{-1} indicating good agreement with those reported in other works [16, 17]. The peaks appearing in the 1500–1600 cm^{-1} range correspond to the C–N bond of quinoid ring (1597 cm^{-1}) and benzenoid ring (1508 cm^{-1}) vibrations in the emeraldine salt. The intensity ratio of these two peaks quinone/benzene for synthesized PANI is less than unity. The peaks observed at 1303 and 802 cm^{-1} are related to the C–N bond of secondary aromatic amines and the aromatic C–H flexion graft [13]. The peak appearing at 1145 cm^{-1} is related to the protonation of nitrogen atoms in the imine ring of quinones [13, 17], and is known as an electron-like band.

There are significant differences between the PANI/GNS and PANI spectra. Interestingly enough, however, there is a little difference in the peaks of those containing different wt% GNS. In the PANI/GNS spectra, the ratio of the quinone/benzene intensity is more than unity, indicating that

PANI is richer in this case than in the quinone rings. In other words, the interaction of PANI-graphene increased the quinone ring structure [19]. The position of peaks of quinone and benzene also changed. Another obvious difference in the N–H bond is in the 3402 cm^{-1} . This peak is strong in the nanocomposites, while very weak in PANI. The reason for the strength of this peak is not well known, but the interaction between PANI and GNS may seem to lead to chain transfer [20]. The intensity and position of other peaks in the PANI/GNS nanocomposites have also changed as compared to PANI. For instance, the intensity of 1145 cm^{-1} peak has increased, indicating that the interaction between PANI and GNS has facilitated the transfer process and increased the degree of electronic stability in the PANI chain, which ultimately resulted in increased electrical conductivity in the PANI/GNS [13].

To determine the presence of nanocomposite on the fabric, AT-FTIR spectrum was taken of the 2.5 wt% GNS sample. The result is shown in Fig. 3 and in Table 1.

FE-SEM images of the specimens were taken at different magnifications. Examples include impregnated fabric samples by PANI/GNS containing 0.5, 2.5, 5, 10 wt% GNS. The surface morphology and particle size on the fabrics containing varying wt% GNS are shown in Figs. 4 and

Fig. 3 AT-FTIR spectrum of PANI/2.5 wt%GNS coated fabric

Table 1 Characteristic AT-FTIR absorption frequencies of organic functional groups of PANI/2.5 wt% GNS coated fabric

Characteristic Absorptions (cm ⁻¹)	Functional Group	References
786	C–H	[30]
1097	C–H	[31]
1231	C–N	[32]
1285	C–N	[31, 32]
1470	Benzene ring	[33]
1564	Quinoid ring	[30, 34]
1680	C=C	[31, 35]
3222	O–H	[36, 37]

5, and it can be said that with increasing wt% GNS, the particle size distribution improved, but only up to 5 wt%, then a composite film formed on the fabric.

By examining Fig. 5, it is obvious that all composites are in the nano size range.

Thermoelectric properties

Electrical conductivity

The result of electrical conductivity of PANI/GNS coated fabric are shown as a function of GNS loading in Fig. 6.

As shown in Fig. 6, with increment in GNS content, electrical conductivity increased. The increase in electrical conductivity was more pronounced up to 2.5 wt% GNS. This improvement in the composite electrical conductivity can be attributed to the high electrical conductivity of GNS; their high carrier movability, as well as their uniform distribution throughout the PANI matrix [27, 38, 39]. In other words, GNS acted as bridge helping in carrier transport by π – π interactions and thus enhanced carrier movability, leading to increased electrical conductivity on the fabric surface. The optimal enhancement in electrical conductivity occurred at 2.5 wt% GNS, beyond which GNS may interrupt the carrier transportation in the PANI matrix chains [38].

Fig. 4 FE-SEM images of the surface of PANI/GNS coated fabric containing **a** 0.5 wt% GNS, **b** 2.5 wt% GNS, **c** 5 wt% GNS and **d** 10 wt% GNS

Seebeck coefficient

The Seebeck coefficient, shown in Fig. 7, was obtained by measuring the difference in voltage produced per unit of temperature difference created on both sides of the coated fabric samples. As shown in Fig. 7a, the Seebeck coefficient increased very sharply at around 2.5 wt% GNS followed by downward trend. The improvement of Seebeck coefficient could be illustrated in the sense of the energy filtering of carriers by inducing crystallization that may occur during PANI/GNS nanocomposite preparation [40,

41]. To estimate Seebeck coefficient for a p-type semiconductor, the figure of merit equation is used:

$$S = \frac{k_B}{q} \left[r + 2 + \ln \frac{2(2\pi m^* k_B T)^{3/2}}{h^3 n} \right], \quad (1)$$

$$\sigma = ne\mu_H, \quad (2)$$

where n is carrier concentration, q is charge, r is scattering parameter, m^* is effective mass, k_B is Boltzmann constant, T is temperature, and h is Planck's constant. Presented results

Fig. 5 FE-SEM images of the surface of PANI/GNS coated fabric, **a** 0.5 wt% GNS, at larger magnification, containing **a** 0.5 wt% GNS, **b** 2.5 wt% GNS, **c** 5 wt% GNS and **d** 10 wt% GNS

in literature also shows that Seebeck coefficient decreases by increasing carrier concentration, usually [42]. The increase and decrease trends in Seebeck coefficient have also been attributed to PANI-type transition from n- to p-type in the presence of GNS for around 1 wt% [40]. A study has noted that electrical conductivity and Seebeck coefficient cannot increase simultaneously due to narrow energy transport level (E_T) and Fermi level (E_F). In this study, however, both the Seebeck coefficient and electrical conductivity of PANI/GNS increased simultaneously, indicating that a conventional model based on the band theory or the electron–phonon scattering cannot explain the conduction mechanism [43]. The simultaneous increase in both properties has been reported in other studies too [39, 44, 45]. The reason might

be due to the electronic-structure of the polymers [43]. In other words, the molecular ordering of polymer chains affect their electronic-structure, causing an increase in charge carrier mobility, and leading to simultaneous increase in both Seebeck coefficient and electrical conductivity [46].

As noted above, Seebeck coefficient, according to Eq. 1, is function of temperature. In Fig. 7b, the Seebeck coefficient is shown for two samples (2.5 and 5 wt% GNS) that showed the highest Seebeck coefficient and electrical conductivity among the four samples. Figure 7b illustrates the significance of temperature on Seebeck coefficient; the Seebeck coefficient for 2.5 wt% GNS sample increased from 12 to 18 $\mu\text{V K}^{-1}$ (50%) in the temperature range of

Fig. 6 Electrical conductivity of coated fabric as a function of GNS loading measured at room temperature (300 K)

303–338 K, while for the 5 wt% GNS sample increased from 4.5 to 13.2 $\mu\text{V K}^{-1}$ (193%) in the same temperature range. The upward progression of the Seebeck coefficient with temperature for the PANI/GNS composite of p-type has been reported in another study, too [20, 47].

Power factor

The effects of GNS loading on the power factor of the PANI/GNS nanocomposite fabric are presented in Fig. 8. It is obvious that the power factor was very low up to around 1.5 wt% GNS, which is due to very low conductivity in the samples. Within the range of 1.5–2.5 wt% GNS, the power factor very sharply increased and reached its maximum value. Considering that 2.5 wt% GNS provided the

maximum power factor, it was used as the appropriate wt% GNS for coating the fabric strips and preparing the nanocomposite materials.

Conclusions

Thermoelectric flexible fabric was prepared by applying a thin layer of PANI/GNS on the surface of a woven fabric by ultrasonic assisted dipping and slurry aqueous polymerization processes. The Seebeck coefficient results revealed that PANI/GNS induced thermoelectric properties to the flexible fabric up to 193%, and over 190% enhancement in the power factor, making this a potential fabric for powering wearable devices and waste energy recycling.

Fig. 7 **a** Seebeck coefficient as a function of wt% GNS measured at average temperature (338 K). **b** Seebeck coefficient for 2.5 and 5 wt% GNS samples as a function of average temperature

Fig. 8 Power factor as a function of wt% GNS measured at average temperature (338 K)

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