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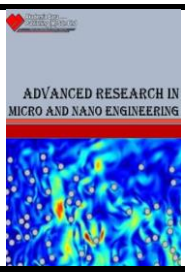

Optical Properties of Gold Nanoparticles Doped P3HT Nanowires Films

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ABSTRACT

Poly (3-hexyl-thiophene-2,5-diyl) (P3HT) material is widely used as the benchmark of p-type semiconductors in organic photovoltaics due to its high conductivity and self-assembly into nanowires. In this study, gold-doped P3HT nanowires were synthesized using o-xylene, and their optical and electronic changes were investigated. P3HT nanowires were kept in the dark for 72 hours to obtain 60 – 80 nm diameter of nanowires. 1 mM of chloroauric acid doping was used as glass-coated P3HT nanowires were dipped repetitively into the chloroauric acid solution. It was found that visible absorbance intensity reduced and the calculated optical bandgap energy of P3HT nanowires thin film decreased as much as 0.08 eV after doping. FTIR and Raman show there are no extreme changes in P3HT conjugated backbones structure after doping meaning that the gold did not completely integrate within nanowires. From this observation, we discussed the feasibility of this coating method to produce metal-doped organic film and the required improvement to suit photonic device applications.

1. Introduction

In recent years, conjugated conducting polymers have been used for organic electronic devices due to solution processability and good charge transport properties [1]. Poly (3-hexylthiophene-2, 5-diyl) (P3HT) is the most interesting candidate due to its versatile semi-crystalline properties. According to many, P3HT film transports high currents despite its limitations in optical absorption [2-4]. P3HT is also known for its good stability and higher charge mobility [5]. However, the narrow optical absorption coverage greatly affected the energy conversion of P3HT in solar cells and photovoltaic applications. Some attempts to improve the absorption are by increasing the thickness of the P3HT film to enhance maximum light absorption that will result in the coherent collection of charges following more exciton dissociations at the interface of the polymer. The formation of dense and highly oriented polymer nanowires and nanofibers was also reported to produce faster charge carriers and lower recombination rates [6]. Since the polymers are better oriented as 1-dimensional

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nanostructures, it will surely improve both their electronic and optical properties. Doping of conjugated polymer is an alternative to tailor organic electronic materials as sensor and photonic devices by enhanced charge transfer and shift of the operative wavelengths, respectively.

Chloroauric acid ($HAuCl_4$) is a p-type dopant for conjugated organic molecules and acts as a precursor for gold particles (Au) [7]. The reduction of $HAuCl_4$ produced different sizes of gold nanoparticles (AuNP) [8]. Depending on the application, AuNP could be synthesized chemically using sodium citrate or ascorbic acid reducing agents. Different sizes and shapes of AuNP are controlled by the preparation of $HAuCl_4$ using the laser ablation method or chemical means [9]. Strong optical response and electromagnetic enhancement by AuNP are strategically employed in sensor applications, optical diodes, photonic technology, optoelectronics, and many more [10].

Intrinsic electronic modification by gold in polymer host originated from their molecular interaction and changes in microscopic structures. A study by Zaminpayma *et al.,* in 2013 shows the mechanical and electrical enhancement of P3HT-Au nanowires was due to good interfacial interaction between polymer and nanowire [11]. The Au nanowires comprise of high surface area and hence have stronger interaction with the polymer backbone. The same study also showed that Au has the strongest metal interaction with P3HT followed by silver, copper, and aluminum metals [11]. In addition, a computational study of P3HT and Au nanowires was done by Mirabbaszadeh *et al.,* using different time steps of the simulation [12]. Firstly, the P3HT chain was placed near the Au nanowire. Then, it elongated and moved beside the nanowire until their surfaces were attached. The aromatic rings of P3HT orient themselves to align parallel to the Au nanowire surface according to its lowest energy formation. As a result, better charge transfer will occur from Au to P3HT, increasing device performance [12].

In this study, we present the optical and electronic properties of Au-doped P3HT nanowires prepared using simple dip coating in $HAuCl_4$ solution. The technique was previously employed by Zhao *et al.,* to fabricate a volatile organic compound (VOC) sensor device [7]. The structure of P3HT nanowires and Au-doped P3HT nanowires are discussed using Fourier Transform Infrared (FTIR) spectroscopy, Raman spectroscopy, and Field Emission Scanning Electron Microscopy (FESEM). This complementary analysis further explains the microscopic science in this P3HT film fabrication. The color changes after doping are also recorded.

2. Methodology

2.1 Preparation of P3HT Nanowire Film

Poly (3-hexylthiophene-2, 5-diyl) (P3HT) (50000 ~ 100000 M_w, 99% purity, regiorandom) and o-xylene (106.17 M_w, 97% purity) were purchased from Sigma Aldrich and were used without purification. To produce a nanowire solution, the o-xylene solvent was added to P3HT using a ratio of 1:2. The methodology was described elsewhere [13]. This is a similar method demonstrated by Zhao *et al.,* [7]. After 4 hours of stirring at 75 °C, the solution was kept in the dark for an additional 72 hours to allow P3HT molecules to self-assemble into nanowires. Meanwhile, a glass substrate was cleaned with distilled water. A 100 µL of P3HT nanowire suspension was drop-casted onto the glass until it had adequate surface coverage. The film was then dried at room temperature for 24 hours.

2.2 Preparation of Au-Doped P3HT Nanowire Film

Gold (III) chloride trihydrate, $HAuCl_{4.3}H_2O$ (383.83 M_w, 49% purity) purchased from Sigma Aldrich was used without further treatment. The powder was dissolved in distilled water to produce 1 mM of $HAuCl_4$ solution. The glass-coated P3HT nanowire film was coated with Au by alternate dipping

into $HAuCl_4$ solution for 3 s and distilled water for six consecutive times. The schematic illustration of the doping procedure is shown in Figure 1.

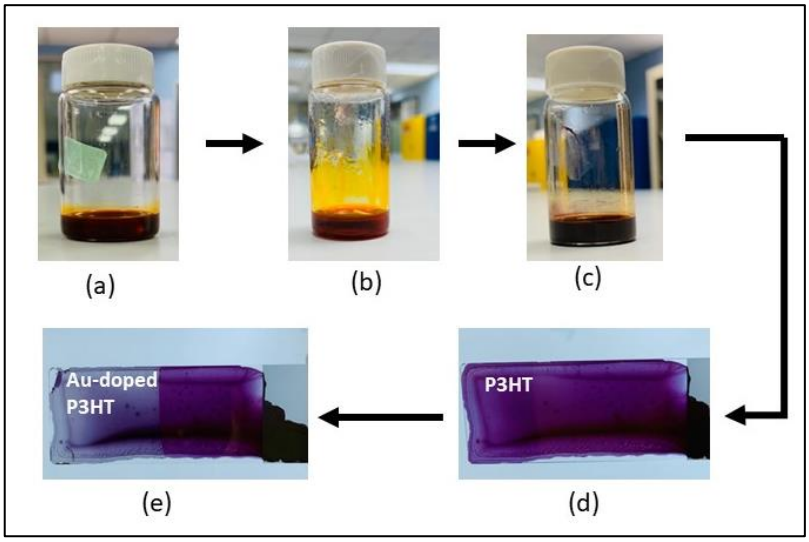


Fig. 1. Color transformation of Au-doped P3HT nanowires: (a) P3HT solution before stirring, (b) P3HT solution after stirring, (c) P3HT solution after being kept in the dark, (d) P3HT film drop casted onto a glass substrate, and (e) P3HT film after doped with $HAuCl_4$ solution

2.3 Characterization Analysis

The optical absorbance of the P3HT nanowire film was recorded with a UV/Vis spectrophotometer (Perkin Elmer UV, Winlab). Vibrational spectroscopies by FTIR spectrometer by Perkin Elmer and Raman spectrometer by Renishaw were used to study the change in polymer structure. The morphology of the P3HT nanowire film was analyzed by the FESEM machine model Gemini SEM 500.

The optical bandgap was plotted using the Tauc plot equation. The optical absorption coefficient, α was gained from UV-Vis data. The Tauc equation can be expressed as

$$\alpha h\nu = A\left(h\nu - E_g\right)^{1/2} \quad (1)$$

where $h\nu$ is a photon energy, E_g is an energy gap of the nanomaterial, and A is a proportionality constant [14].

3. Results

As shown in Figure 1, the initial state of the P3HT mixture has a dark brown color. It will change into a lighter yellowish-brown color after a continuous heat-stirring process. The technique to produce self-assembled polymer using choice solvent in the solution processing method is notably easy and low cost [15]. Here, the P3HT solution was also kept away from light exposure so the polymer chains entangled due to strong Van der Walls force between themselves. The force is stronger without light interruption which usually causes the polymer to oxidize and become charged. The aggregated P3HT solution became dark purple after 72 hours left in the dark indicating the formation of denser molecules. Further drying allowed the solvent to evaporate or migrate from the drop-cast P3HT film. Finally, the color of the Au-doped P3HT nanowire changed from dark purple to

dark blue after it was dipped into the $HAuCl_4$ solution. A similar observation was shown by Zhao *et al.,* signifying successful Au doping [7]. The color changes of P3HT nanowire films were further analyzed by UV-Vis spectrometer and the result is shown in Figure 2.

Comparison of UV-Vis spectra obtained from pristine P3HT nanowires film, Au-doped P3HT nanowires film (1x), and Au-doped P3HT film (6x) clearly showed a drastic decrease of absorption across P3HT absorption bands around visible range of 400 – 650 nm. The three bumps located around 530 nm, 575 nm, and 615 nm represent ground to first excited state transition that is quite sensitive to polymer conformation that takes place in the P3HT conjugation system. The least intense peak of 615 nm belonged to the interchain transition of P3HT, and its intensity is correlated to the degree of interchain order [16]. The transformation of these peaks usually elucidates the state of P3HT aggregation both in amorphous P3HT and dense P3HT nanowires as discussed in our previous report [13]. Au nanostructures can also be identified for the UV-Vis spectrum, for example, citrate reduced AuNPs by Kasim *et al.,* have surface plasmon resonance (SPR) peaks located around 560 nm [17]. In our study, this peak can be easily consumed by the large absorption around 400 – 600 nm of P3HT.

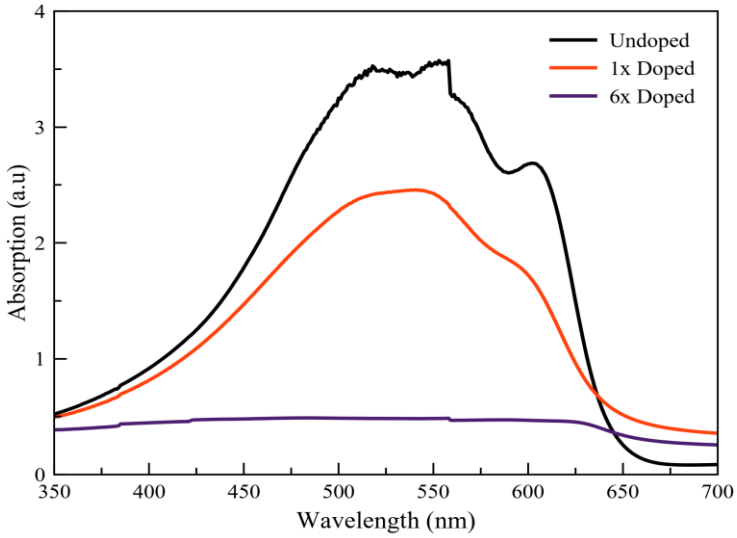
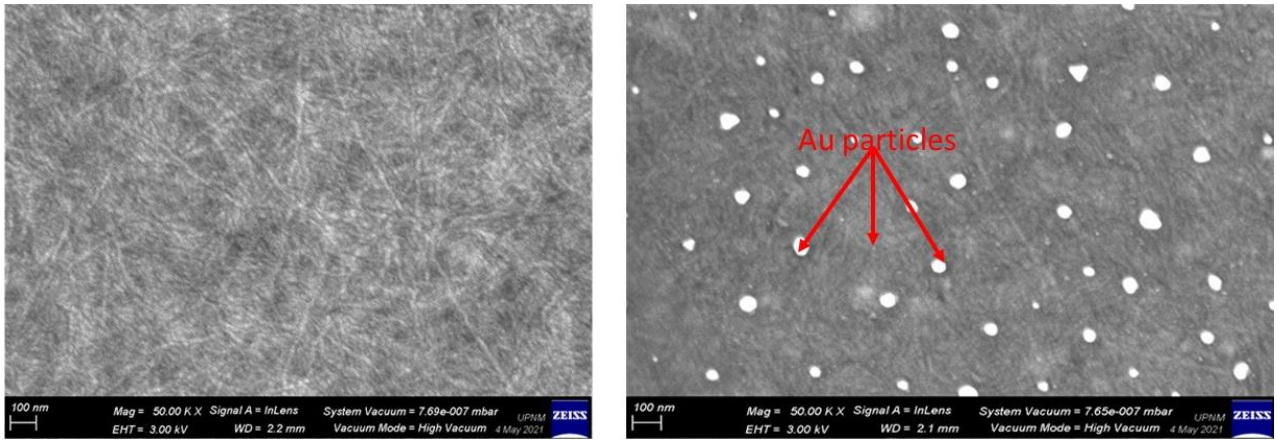


Fig. 2. UV-Vis spectra of P3HT nanowires: undoped, doped at one and six times

Figure 3 shows electron images of undoped P3HT nanowires film and Au-doped P3HT nanowires film (1x) which present nanowires of random orientation measured from the surface of glass substrates. In the 50K magnification images, we found the average diameter of P3HT nanowires is around 60 - 80 nm. The wire diameter and number of P3HT aggregates implied an increase in the semi-crystallinity of the polymer. This also indicates that the P3HT nanowires have been successfully fabricated. On the other hand, randomly sized irregular-shaped particles were observed in Figure 3(b) that can only belong to Au particles. The sizes of particles are widely dispersed with the largest Au particles being 100 - 120 nm in diameter. Random smaller particles were also seen which pose the possibility of manifesting localized SPR from such a sample. This doping technique is shown to be quite efficient in producing Au-decorated surfaces as the Au^+ ions in $HAuCl_4$ are completely reduced into elemental gold, Au^0 by surface reactants.

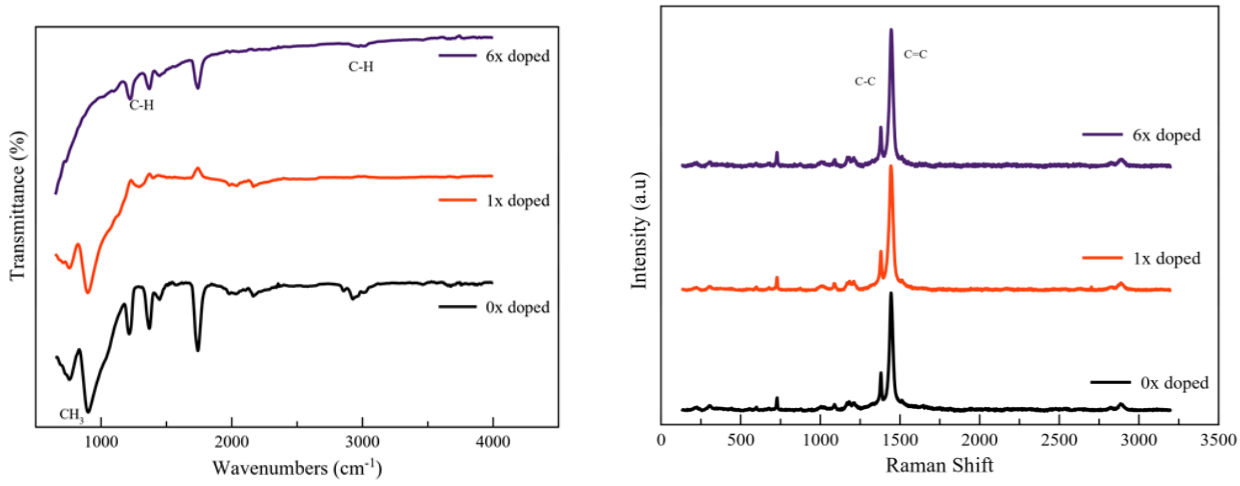


(a) (b)

Fig. 3. FESEM images of (a) P3HT nanowires and (b) Au-doped P3HT nanowires at 50kV magnification

Structural characterizations using Raman and FTIR were used to study the effect of Au doping on carbon interaction in polymer chains. Based on FTIR spectra in Figure 4(a), similar vibrational peaks belonging to CH_3 and CH groups were observed in undoped and doped P3HT nanowires films. The peak at 2850 cm^{-1} belonged to the C-H bond from the hexyl side chain [18]. Two peaks from the P3HT fingerprint are 902 cm^{-1} and 762 cm^{-1} associated with CH_3 and CH bonds respectively [18]. The decrease in peak intensities after doping was observed because of P3HT layer depletion after dipping into an acidic solution. This correlates with the observation in Figure 1 where the P3HT layer was seen to become thinner after six consecutive dips. The reduction of these peaks might also relate to the pack arrangement of the nanowire or mechanical suppression by Au particles.

The change of bulk crystallinity of P3HT nanowires is best studied using Raman spectra (Figure 4(b)) because of its sensitivity to the thiophene rings on the conjugated backbones [19]. Furthermore, the Raman cross-section increased under a laser wavelength of 633 nm. From this measurement, it was observed that two strong peaks belonging to C=C are symmetric at 1445 cm^{-1} and C–C stretching at 1380 cm^{-1} vibrational modes of the thiophene ring [20]. Moreover, there were few weak peaks at 1208 cm^{-1}, 1182 cm^{-1}, 1089 cm^{-1}, 1000 cm^{-1}, and 728 cm^{-1} were referred to as C–C stretch mode, C–H bending mode with C–C inter-ring stretch mode, C–H bending, stretching deformation between carbon and carbon of the hexyl substituent and C–S–C deformation mode, respectively [21]. Noticeably, the peak Raman intensity at 1x dipping cycle slightly did not change, however, after a 6x dipping cycle, the Raman intensity was reduced by 64%. Interestingly, there were no changes at the peak C=C position, indicating crystallinity and conjugated length of P3HT nanowires remained undisturbed after gold doping [20].



(a) (b)

Fig. 4. (a) FTIR transmittance of P3HT and Au-doped nanowires at different times doped with Au, and the complementary (b) Raman spectra of P3HT and Au-doped P3HT nanowires

Using the UV-vis data, we produced a Tauc plot for both undoped and Au-doped P3HT nanowires as shown in Figure 5. Previously, the red-shifted absorption edge was due to the optical bandgap of Au-doped P3HT that was investigated using the Tauc plot. The Tauc plot is useful in analyzing the optical band gap of semiconductor material. By definition, the optical bandgap is the threshold for photons to be absorbed and located at the lower side of the energy level [22]. The optical bandgap of organic semiconductors is a key factor in their performance, mostly in optoelectronic applications such as solar cells [23]. Recently, modification of bandgap was performed by coherent doping with plasmonic materials and the usage of organic ligands in silicon nanocrystals [24,25]. These studies have shown the significance lowering of the optical bandgap and the potential to enhance the performance of organic semiconductors. In our previous publication, the optical band gap of the P3HT nanowire was 1.89 eV, slightly lower in comparison to the P3HT film synthesized using tetrahydrofuran which is 1.9 eV [13]. The bandgap variation is due to electron delocalization induced by P3HT interaction with the solvent. After being doped with gold chloride, the optical band gap got smaller; 1.84 eV and 1.81 eV after 1x doped and 6x doped respectively. Table 1 shows the reading of the optical band gap in each film.

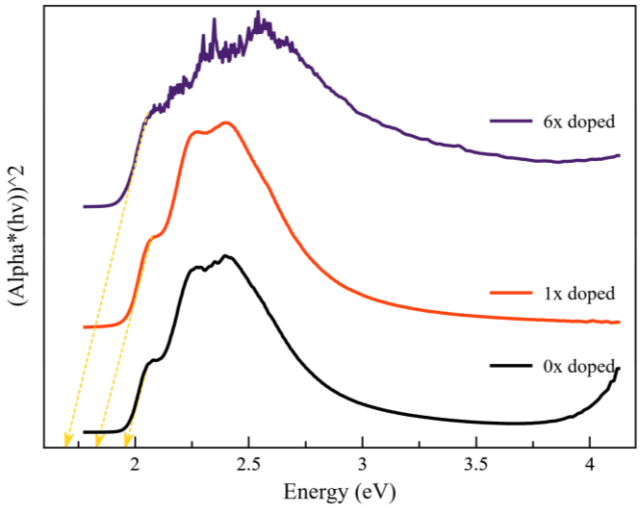


Fig. 5. Tauc plot of P3HT nanowire and Au-doped P3HT from 1.9 eV to 1.81 eV

Table 1
The optical band gap of P3HT nanowire

Sample	Optical bandgap, eV
Undoped P3HT	1.89 eV
1x Au-doped P3HT	1.84 eV
6x Au-doped P3HT	1.81 eV

Figure 6 shows an illustration of Au-doped P3HT nanowires produced from this method where the AuNP mostly resided on the surface of P3HT film instead of intercalating within the nanowire structure. This deposition strategy is suitable for layer-by-layer film devices. In realizing this experiment, we discover several weaknesses of this technique such as the handling of acidic chloroauric acid that can deplete the preceding polymer layer, in our case the P3HT nanowires film. We also encountered the difficulty of controlling the size and shape of the Au particles, hence the need to find the correct environment for homogenous deposition. Nevertheless, Au-surface modified P3HT nanowire film should be reliable as optical substrates in surface-enhanced Raman spectroscopy (SERS) applications and fiber optics applications [26]. Since the P3HT nanowires lie horizontally, the orientation of the electrodes should be thoroughly considered to provide gain in electrical conductivity-related mechanisms.

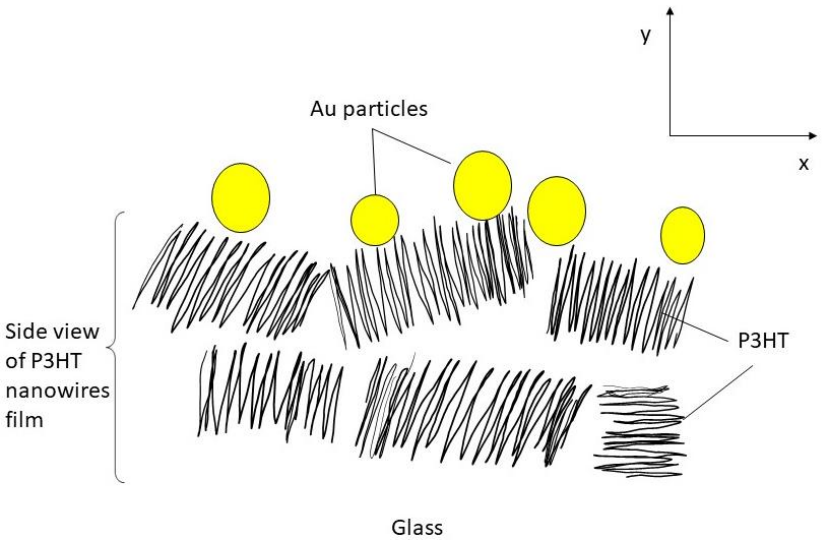


Fig. 6. Surface modified Au-doped P3HT

4. Conclusion

In conclusion, Au-doped P3HT nanowires were successfully produced by the drop-cast method and showed a red-shifted absorption edge that indicated a reduction of P3HT's optical bandgap. This study of controlling optical properties through the doping of gold precursors on P3HT nanowire films has shown an interesting understanding of nanomaterial interactions and their potential applications. Throughout this study, we have examined the benefits and weaknesses of the dip coating technique to incorporate metal nanoparticles onto polymer film coating to optimize its feasibility and functionality in various fields, including optoelectronics, sensing, and photovoltaics.

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