

# Electrochromic Properties of Poly(3,4-ethylenedioxythiophene) Nanocomposite Film Containing SiO<sub>2</sub> Nanoparticles

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**ABSTRACT:** Through incorporation of SiO<sub>2</sub> nanoparticles (NPs) into poly(3,4-ethylenedioxythiophene) (PEDOT) films, the physical volume effect of nanoparticles on the electrochromic properties of PEDOT was investigated. PEDOT-SiO<sub>2</sub> nanocomposite thin films were prepared using SiO<sub>2</sub> NPs, 3,4-ethylenedioxythiophene (EDOT), iron *p*-toluenesulfonate and an imidazole mixture. During incorporation, SiO<sub>2</sub> NPs agglomerated to form a cluster, which affected the surface roughness of PEDOT films. Photoemission spectroscopic results of C 1s and S 2p show that incorporated SiO<sub>2</sub> NPs did not chemically interact but heavily affect the conjugation length and packing density of PEDOT due to a physical volume effect.

This physical effect of SiO<sub>2</sub> NPs resulted in enhanced electrical conductivity of PEDOT and induced a blue shift of the maximum absorption peak in ultra violet-visible spectroscopic analysis. Also, a gradual enhancement in the electrochromic properties of PEDOT was obtained by increasing the amount of SiO<sub>2</sub> NPs; in particular, the switching speed of PEDOT reduced from 3.34 s to 1.28 s with incorporation of 10 wt % SiO<sub>2</sub> NPs. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 122: 3080–3085, 2011

**Key words:** SiO<sub>2</sub> nanoparticles; PEDOT film; electrochromic; switching speed; NEXAFS

## INTRODUCTION

Composite materials containing nanoparticles are of interest for various applications because of their unique chemical and physical properties. Nanoparticle/conducting polymer composites are applicable for displays, mirrors, windows, light-emitting diodes, photovoltaics, and electrochromic devices.

Electrochromism is defined as a reversible optical change or coloration under electrochemical redox reactions.<sup>1</sup> Inorganic materials like WO<sub>3</sub>, MoO<sub>3</sub>, and V<sub>2</sub>O<sub>5</sub>, and organic materials like polypyrrole, polythiophene, and polyaniline show electrochromic properties.<sup>2</sup> To improve the electrochromic properties of organic materials, many groups have studied the chemical structure of monomers.<sup>3–5</sup> On the other hand, physical approaches, such as making mesoporous electrochromic films to facilitate ion diffusion,<sup>6</sup> reducing the diffusion distance for the dopant ions through the use of conjugated polymer nanotubes<sup>7</sup> and porous microspheres<sup>8</sup> have improved the

switching speed of the devices but have had insignificant impact on the contrast. For enhancement of electrochromic-related properties of conjugated polymers, some groups have recently studied conjugated polymer hybridization with several nanoparticles, including Au, Ag, TiO<sub>2</sub>, and SiO<sub>2</sub> NPs.<sup>9–12</sup>

The effect of incorporated oxide nanoparticles on conjugated polymers has been studied by many groups. For example, PPV-SiO<sub>2</sub> composites have been characterized in terms of  $\pi$ -electron behavior, conjugation length, and hole transport properties.<sup>13,14</sup> Oxide nanoparticles added to PPV gave rise to improved current density and radiance in polymer light-emitting diodes.<sup>15</sup> Recently, organic materials like poly(3,4-ethylenedioxythiophene) (PEDOT) have received much attention for applications in electrochromic devices due to unique properties, such as high transparency, high conductivity, and good thermal stability.<sup>16,17</sup> For improvement of the electrochromic-related properties of PEDOT, some groups have recently studied PEDOT hybridization with metal nanoparticles because metal nanoparticles are known to be excellent chromophores in the visible and near-infrared ranges due to an excitation of surface plasmons.<sup>18–20</sup> Although many studies have been done on the preparation and basic physicochemical characterization of hybridized PEDOT with Ag or Au metal nanoparticles,<sup>21,22</sup> the influence the physical volume effect (steric effect) has on the electrochromic properties needs to be addressed. In this study, SiO<sub>2</sub> NPs were incorporated into PEDOT to investigate the steric effect and enhance the

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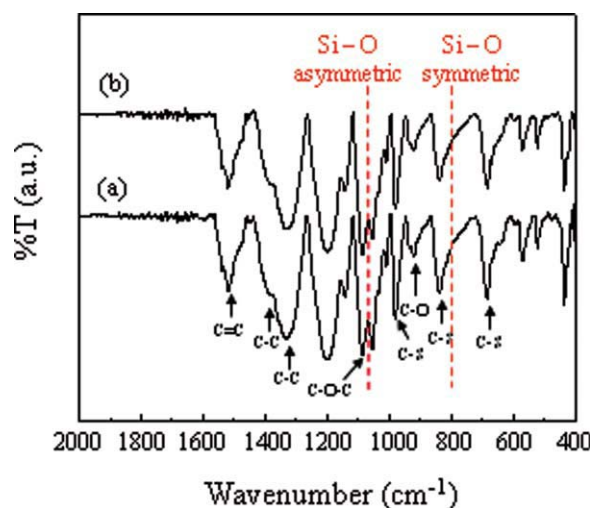
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electrochromic properties of PEDOT. The effects of SiO<sub>2</sub> NPs on the electrical, optical, and electrochromic properties of PEDOT were investigated.

### EXPERIMENTAL

3,4-ethylenedioxythiophene was used as a monomer for PEDOT and iron *p*-toluenesulfonate (Fe(OTs)<sub>3</sub>) was used as both an oxidant and dopant. Imidazole (IM) was used as a weak base to control polymerization. These materials were obtained from Aldrich and were used as received. The films of PEDOT and PEDOT-SiO<sub>2</sub> nanocomposites were prepared as follows. First, Fe(OTs)<sub>3</sub> (1.75 mmol) and IM (2 mmol) in MeOH were prepared separately and 1 mmol of EDOT was added to the IM solution. Then, the Fe(OTs)<sub>3</sub> solution was mixed with the EDOT-IM solution. The mixture color changed from yellow to dark green. Next, the mixed solution was spin-coated onto glass and indium tin oxide (ITO) coated glass (2000 rpm, 10 s), and polymerization was carried out on a hot-plate at 80°C for 10 min. Completed films were carefully washed with MeOH and DI water to remove unreacted residues containing EDOT, Fe(OTs)<sub>3</sub>, and IM. The PEDOT-SiO<sub>2</sub> nanocomposite films were synthesized by adding SiO<sub>2</sub> NPs dispersed in a MeOH solution to the mixture of EDOT, Fe(OTs)<sub>3</sub> and IM. The PEDOT-SiO<sub>2</sub> nanocomposite films with different SiO<sub>2</sub> NPs content were prepared: 1, 5, and 10 wt % of SiO<sub>2</sub> NPs. The weight percents were calculated using the weights of SiO<sub>2</sub> and EDOT.

The formation of PEDOT and PEDOT-SiO<sub>2</sub> nanocomposite films was verified using Fourier transform-infrared spectroscopy (FTIR: Jasco 300Z) and ultra violet-visible (UV-Vis: Lambda 35, Perkin-Elmer) spectroscopy. Scanning electron microscopy (SEM: Hitachi S-4200, Nissei Sangyo) was used to monitor the surface morphologies of the PEDOT and PEDOT-SiO<sub>2</sub> nanocomposite films. Energy dispersive spectroscopic (EDS) analysis was performed on the nanocomposite film with 10 wt % SiO<sub>2</sub> to check the presence of SiO<sub>2</sub> NPs. To investigate the chemical bonding states and electronic structure of PEDOT-SiO<sub>2</sub> nanocomposite films, photoemission spectroscopy (PES) and C 1s near edge X-ray absorption fine structure (NEXAFS) measurements were carried out using the 8A2 beam line at the Pohang Accelerator Laboratory (PAL). The electrochromic properties, such as switching speed and absorbance with various voltages were measured for PEDOT and PEDOT-SiO<sub>2</sub> nanocomposite films deposited on ITO glass using a galvanostat/potentiostat (Compactstat, IVIUM) in 0.1M LiClO<sub>4</sub>/acetonitrile. The reference and counter electrodes were Ag/AgCl and platinum wire, respectively. The working electrode size was 1 cm × 5 cm.

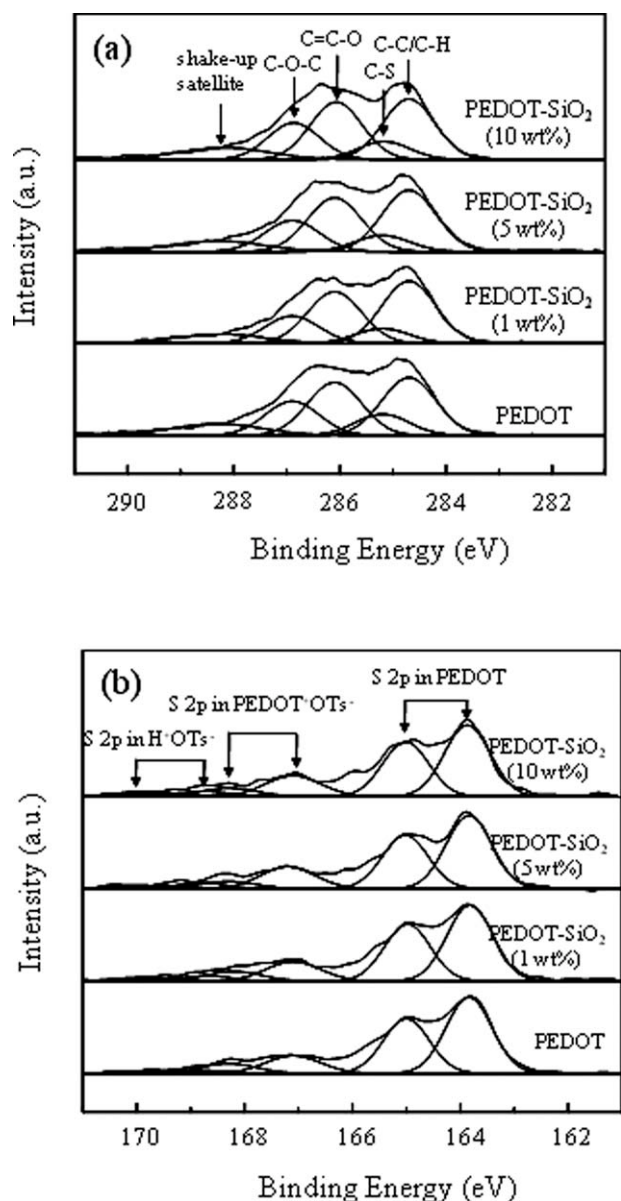


**Figure 1** FTIR spectra of (a) PEDOT and (b) PEDOT-SiO<sub>2</sub> (10 wt %) thin films. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://www.interscience.wiley.com).]

### RESULTS AND DISCUSSION

The effect of SiO<sub>2</sub> NPs on the formation of PEDOT was investigated using FTIR measurements. Figure 1 shows the FTIR spectra of (a) PEDOT and (b) PEDOT-SiO<sub>2</sub> nanocomposite films. The peak at 1517 cm<sup>-1</sup> was attributed to the stretching mode of C = C in the thiophene ring, and 1387 cm<sup>-1</sup> and 1332 cm<sup>-1</sup> were attributed to that of C—C. Furthermore, C—O—C stretching and deformation bands in the thiophene ring were observed at 1086 cm<sup>-1</sup> and 921 cm<sup>-1</sup>, and C—S vibration bands were observed at 976 cm<sup>-1</sup>, 837 cm<sup>-1</sup>, and 682 cm<sup>-1</sup>.<sup>23–25</sup> Also, concerning the bands in the counter ions (OTs), asymmetric and symmetric stretching modes of sulfonate were observed at 1198 cm<sup>-1</sup> and 1053 cm<sup>-1</sup>, while in-plane skeleton and in-plane bending vibration modes of the benzene ring were found at 1141 cm<sup>-1</sup> and 1010 cm<sup>-1</sup>, respectively.<sup>26</sup> Normally, Si—O—Si asymmetric and Si—O—Si symmetric stretching vibrational absorptions should have appeared at 1075 and 795 cm<sup>-1</sup>, respectively<sup>27,28</sup>; however, PEDOT and all PEDOT/SiO<sub>2</sub> nanocomposite films showed similar absorption behaviors. The FTIR results confirmed that the synthesized PEDOT and PEDOT-SiO<sub>2</sub> nanocomposite films were well doped by OTs, and that the addition of SiO<sub>2</sub> NPs did not affect the formation of the PEDOT film.

Figure 2 provides PES results for (a) C 1s and (b) S 2p of PEDOT and PEDOT-SiO<sub>2</sub> nanocomposite films. They showed exactly the same spectra for C 1s and S 2p. In the C 1s spectra, four different bonding states, including the C—C bond (284.7 eV), the C—S bond (285.2 eV), the C=C—O bond (286.1 eV), and the C—O—C bond (286.9 eV) were found with a shake-up satellite peak (288.2 eV); these were the same as those observed by Spanninga et al.<sup>29</sup> In the

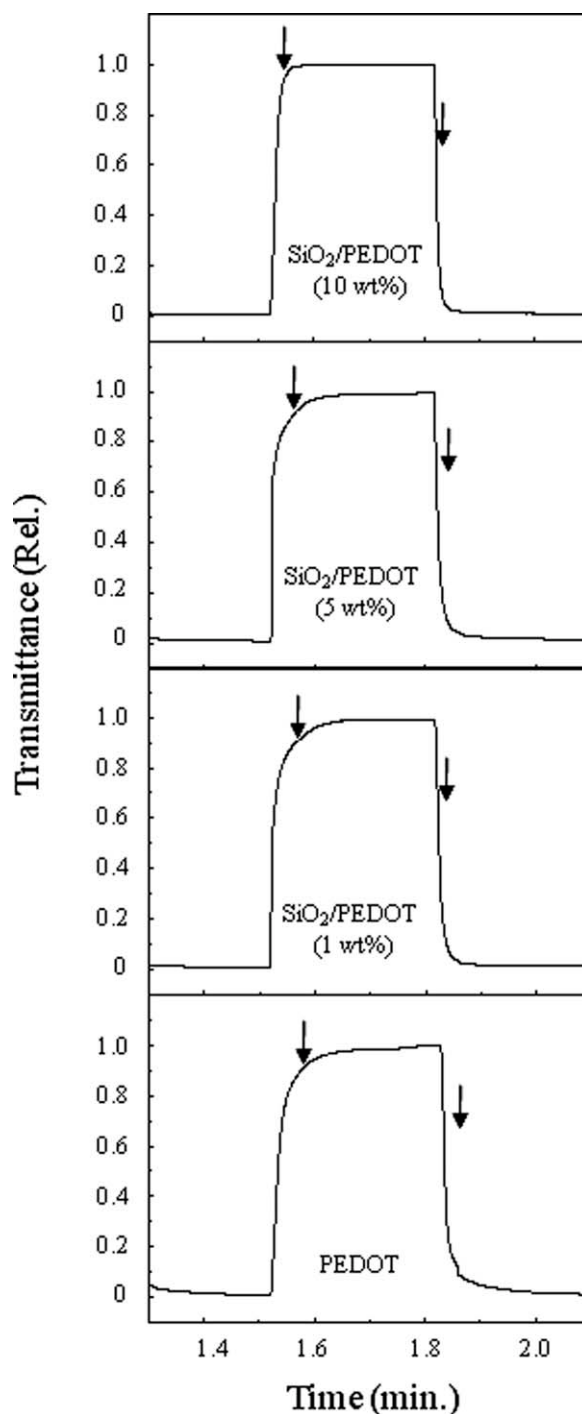


**Figure 2** PES spectra of PEDOT and PEDOT-SiO<sub>2</sub> thin films: (a) C 1s and (b) S 2p.

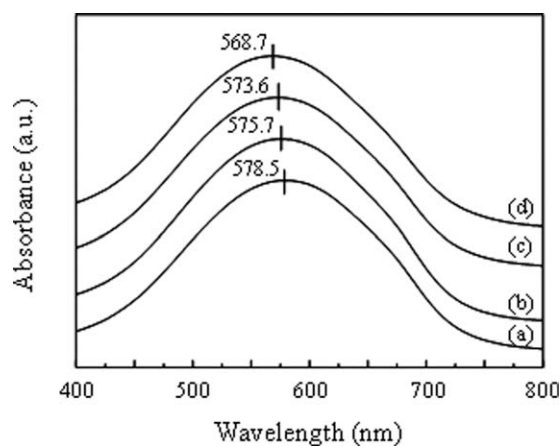
S 2p spectra, the low binding energy peaks at 163.8 eV and 165 eV were assigned to the S 2p<sub>3/2</sub> and S 2p<sub>1/2</sub> of sulfur in PEDOT, and the high binding energy peaks at 167.1 eV and 168.3 eV were assigned to S 2p<sub>3/2</sub> and S 2p<sub>1/2</sub> of sulfur in sulfonate, PEDOT<sup>+</sup>Ots.<sup>30</sup> Peaks at 168.7 eV and 170 eV corresponded to sulfur in sulfonic acid, H<sup>+</sup>Ots. The PES results of C 1s and S 2p of PEDOT and PEDOT-SiO<sub>2</sub> showed neither chemical interaction between SiO<sub>2</sub> NPs and PEDOT nor chemical modification of PEDOT; these results agreed well with the FTIR observations. The lack of interaction or modification seemed to be due to the chemical inertness of SiO<sub>2</sub> and stability of PEDOT in the doped state.<sup>31</sup>

Figure 3 shows the switching speeds of PEDOT and PEDOT-SiO<sub>2</sub> nanocomposite films. These data

were measured in the  $-0.7$  V to  $+0.3$  V (vs. Ag/AgCl) range. The switching time from dark blue ( $\Delta t = 0.9$ ) was enhanced with increasing SiO<sub>2</sub> NP content: 3.34 s for PEDOT, 2.7 s for PEDOT-SiO<sub>2</sub> (1 wt %), 2.3 s for PEDOT-SiO<sub>2</sub> (5 wt %), and 1.28 s for PEDOT-SiO<sub>2</sub> (10 wt %). Also, the switching time from pale blue to dark blue ( $\Delta t = 0.9$ ) was slightly improved, 1.83 s for PEDOT, 1.3 s



**Figure 3** Switching speeds of PEDOT and PEDOT-SiO<sub>2</sub> (1 wt %, 5 wt %, and 10 wt %) thin films from  $-0.7$  V to  $+0.3$  V (vs. Ag/AgCl).



**Figure 4** UV-Vis spectra of (a) PEDOT, (b) PEDOT-SiO<sub>2</sub> (1 wt %), (c) PEDOT-SiO<sub>2</sub> (5 wt %), and (d) PEDOT-SiO<sub>2</sub> (10 wt %) thin films at  $-0.7$  V (vs. Ag/AgCl).

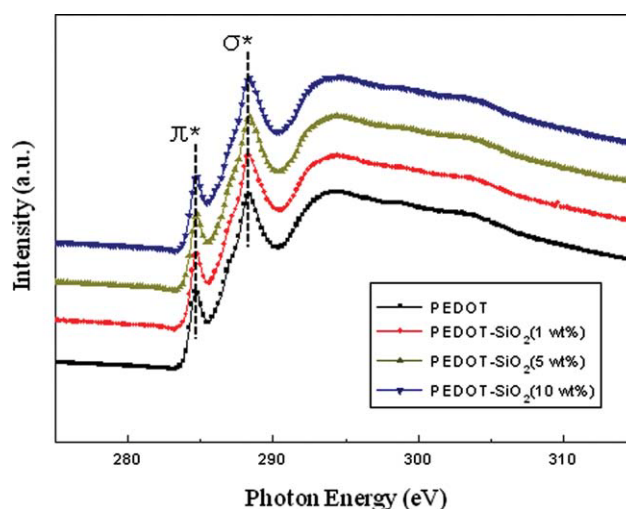
for PEDOT-SiO<sub>2</sub> (1 wt %), 1.4 s for PEDOT-SiO<sub>2</sub> (5 wt %), and 0.89 s for PEDOT-SiO<sub>2</sub> (10 wt %). Typically, maximum contrast values can be calculated from the difference in transmittance between the clear state and dark state.<sup>32</sup> The calculated contrast values were found to increase with increasing SiO<sub>2</sub> nanoparticle concentration: 34.93 for PEDOT, 36.76 for PEDOT-SiO<sub>2</sub> (1 wt %), 39.73 for PEDOT-SiO<sub>2</sub> (5 wt %), and 38.17 for PEDOT-SiO<sub>2</sub> (10 wt %). From the FTIR and PES results, there was no chemical influence of SiO<sub>2</sub> NPs on PEDOT. As such, this enhancement in the switching speed should be explained by the physical influence of SiO<sub>2</sub> NPs on PEDOT. Normally, for an improvement in the switching speed of the polymer, high ionic conductivity is required.<sup>33</sup> For example, Cho and Lee showed an enhancement in the switching speed by decreasing the diffusion distance for the counterions in the polymer.<sup>34</sup>

UV-Vis spectra were obtained in the reduced state ( $-0.7$  V versus Ag/AgCl) to check any physical effect of SiO<sub>2</sub> NPs introduction on PEDOT (Fig. 4). The band maximum, corresponding to the  $\pi$ - $\pi^*$  transition, was used instead of the absorption edge to avoid an ambiguous definition of cut-off in the inhomogeneously broadened spectra.<sup>13,35</sup> The maximum absorption peak shifted to a shorter wavelength with the incorporation of SiO<sub>2</sub> NPs: 578.5 nm for PEDOT, 575.7 nm for PEDOT-SiO<sub>2</sub> (1 wt %), and 573.6 nm and 568.7 nm for PEDOT-SiO<sub>2</sub> 5 wt % and 10 wt %, respectively. This blue shift, i.e., a shortening of conjugation length of the PEDOT chain, was induced by the steric effect.<sup>36</sup> The shortening of the conjugation length of the polymer in the conjugated polymer based-nanocomposite was induced by the increased morphological and conformational disorder due to the presence of nanoparticles. Furthermore, nanoparticles are known to interfere in the

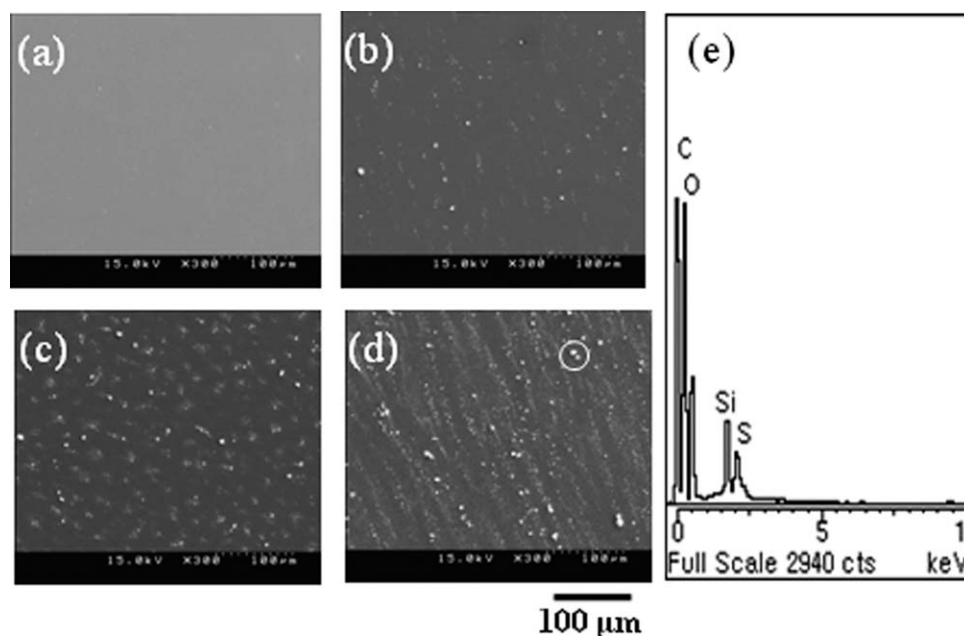
conjugated polymerization reaction and contribute to the formation of disorder and defects, which could reduce the conjugation length of the conjugated polymer.<sup>37</sup> In light of the above PES results, it can be said that PEDOT chains do not chemically interact with SiO<sub>2</sub> NPs but rather the formation of PEDOT chains is influenced by the physical volume effect of SiO<sub>2</sub> NPs.

The steric effect of SiO<sub>2</sub> NPs on PEDOT, i.e., ordering and alignment of chain polymer were investigated by using NEXAFS. In Figure 5, C 1s NEXAFS spectra showed C 1s- $\pi^*$  (284.7 eV) and  $\sigma^*$  (288.3 eV) resonances. As previously observed with PES results, no chemical modification of PEDOT due to SiO<sub>2</sub> NPs incorporation was observed from the position and shape of the absorption peaks. However, the relative intensities of  $\pi^*/\sigma^*$  resonances increased with increasing SiO<sub>2</sub> NP incorporation; for example, 0.442, 0.576, 0.64, and 0.657 for PEDOT, PEDOT-SiO<sub>2</sub> (1 wt %), PEDOT-SiO<sub>2</sub> (5 wt %), and PEDOT-SiO<sub>2</sub> (10 wt %), respectively. This increase in the ratio means that PEDOT becomes highly aligned with increased interaction between chains when incorporating with SiO<sub>2</sub> NPs.<sup>38</sup> The higher degree of interaction between chains corresponds to a shortening of interchain distance. This closer chain conformation induces facile interchain hopping of ions and results in fast switching speeds.

Figure 6 shows the planar SEM images of (a) PEDOT, (b) PEDOT-SiO<sub>2</sub> (1 wt %), (c) PEDOT-SiO<sub>2</sub> (5 wt %), and (d) PEDOT-SiO<sub>2</sub> (10 wt %) films and (e) EDS results of PEDOT-SiO<sub>2</sub> (10 wt %) films. The SEM images showed an increase in the surface roughness with increasing amounts of SiO<sub>2</sub> NPs due to agglomeration of SiO<sub>2</sub> NPs. The presence of SiO<sub>2</sub>



**Figure 5** C 1s NEXAFS spectra of PEDOT and PEDOT-SiO<sub>2</sub> (1 wt %, 5 wt %, and 10 wt %) thin films. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



**Figure 6** SEM images of (a) PEDOT, (b) PEDOT-SiO<sub>2</sub> (1 wt %), (c) PEDOT-SiO<sub>2</sub> (5 wt %), and (d) PEDOT-SiO<sub>2</sub> (10 wt %) thin films and (e) EDS result of PEDOT-SiO<sub>2</sub> (10 wt %) film.

NPs in a PEDOT matrix was confirmed by EDS analysis. In Figure 2(d), the white-circled area corresponds to the EDS analysis area. The result showed primarily the composition of PEDOT, for example, C, O, and S, and the presence of SiO<sub>2</sub> NPs. The surfaces of SiO<sub>2</sub> NPs were not capped by stabilizer, and they seemed to be easily agglomerated during the process. These agglomerated SiO<sub>2</sub> clusters were well distributed in PEDOT film, and increased the surface roughness uniformly, resulting in an increase in surface area. This increase in surface area could also contribute somewhat to an enhancement in the electrochromic properties of PEDOT films.

As a result, incorporation of SiO<sub>2</sub> NPs induced shortening of PEDOT chain conjugation length and interchain distance, and increases in ion conductivity and surface area. These further resulted in an enhancement in switching speed in PEDOT-SiO<sub>2</sub> nanocomposite films.

### CONCLUSIONS

In summary, we investigated the effect of SiO<sub>2</sub> NP incorporation on PEDOT films for electrochromic devices. The incorporated SiO<sub>2</sub> NPs were agglomerated by increasing amount of SiO<sub>2</sub> NPs and did not chemically interact with the PEDOT chains. However, the PEDOT chains were influenced by the steric effect of SiO<sub>2</sub> NPs. Also, SiO<sub>2</sub> NPs induced a decrease in the conductivity: interference polymerization of EDOT which lead to reduced conduction paths and intrachain coherence. Also, the electrochromic properties of PEDOT were changed by

incorporating SiO<sub>2</sub> NPs including a shift of maximum absorption to shorter wavelength in the reduced state, shortening of the conjugation lengths of PEDOT chains and enhancement of switching speed, and a decrease in packing density and increased surface area. On the basis of this study, the PEDOT-SiO<sub>2</sub> nanocomposite films seem capable of enhancing electrochromic device operation via improvements in switching speed and the ability to tune the maximum absorption by controlling the amount of SiO<sub>2</sub> NPs incorporated into PEDOT.

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