

# Transparent, Fluorescent, and Mechanical Enhanced Elastomeric Composites Formed with Poly(styrene-butadiene-styrene) and SiO<sub>2</sub>-Hybridized CdTe Quantum Dots

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**ABSTRACT:** Transparent poly(styrene-butadiene-styrene) (SBS)-quantum dots (QDs) composites (SBS/CdTe QDs) that simultaneously possess strong photoluminescence (PL) and enhanced mechanical properties are presented for the first time based on the facile blending of SiO<sub>2</sub>-hybridized CdTe QDs with SBS. UV-vis spectrum and fluorescence measurement show that SBS/CdTe QDs composites exhibit good optical properties. The results of transmission electron microscopy show good dispersion of CdTe QDs in the SBS matrix. The results of dynamic mechanical thermal analysis indicate that the micro-phase separated structure of the SBS is exist in the composites,

and the presence of CdTe QDs can lead to an decrease of glass transition temperatures of polybutadiene (PB) and polystyrene(PS) domains. In addition, mechanical tests reveal that the addition of CdTe QDs is a useful approach to improve the mechanical properties of SBS. Meanwhile, the fluorescent photographs taken under ultraviolet light prove that SBS/CdTe QDs composites possess strong PL. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 122: 2325–2330, 2011

**Key words:** CdTe quantum dots; SBS; composites; optical properties; mechanical properties

## INTRODUCTION

Semiconductor quantum dots (QDs) have attracted increasing attention within recent years for their promising applications in lasers,<sup>1–3</sup> biological labeling,<sup>4–7</sup> light-emitting diodes (LEDs),<sup>8–10</sup> and devices.<sup>11–13</sup> The organic-inorganic polymer composites combining both the properties of the inorganic and organic materials offer unique mechanical, thermal, and optical features, and can apply in versatile areas.<sup>14,15</sup> To allow QDs embed in the polymer matrix, many research groups have reported on the preparation and properties of QDs/polymer composites.<sup>16–21</sup> QDs/polymer composites films were prepared by solvent casting suspensions of CdSe/ZnS semiconductor nanoparticles in cellulose triacetate solution.<sup>16</sup> Kostić et al.<sup>17</sup> prepared PbS QDs/Polyvinyl alcohol (PVA) composites by a solution method, and the composites was characterized using photoluminescence (PL) and far-infrared spectroscopy.

Suo et al.<sup>18</sup> prepared a PVA thin film filled with core/shell CdSe/ZnS QDs by a drop-casting method. Li et al.<sup>19</sup> synthesized transparent and homogeneous polymeric hybrid materials with ZnO QDs and poly(methyl methacrylate) (PMMA) via conventional *in situ* sol-gel polymerization techniques. Chen and co-workers<sup>20,21</sup> reported controllable synthesis of ZnS and CdS QDs-polymer transparent hybrids by using PMMA as a polymer matrix. Specially, CdTe QDs/polymer composites are studied actively now. The fluorescent quantum dot-polymer composites were fabricated by incorporating thioglycolic acid capped CdTe QDs into polyacrylamide via crosslinking agents.<sup>22</sup> Li et al.<sup>23</sup> prepared a series of positively charged polystyrene (PS) nanosphere emulsions by copolymerization of quaternary ammonium chloride cationic monomer with styrene via emulsifier-free emulsion polymerization, and they found that the CdTe-PS/PVA composite solution has potential applications in light emitting devices by inkjet printing. In our laboratory's previous studies, we have successfully synthesized poly(2-(dimethylamino)ethyl methacrylate) (PDMAEMA)/CdTe QDs composites<sup>24</sup> and hyperbranched polyglycerol (HPG)/CdTe QDs composites.<sup>25</sup>

Poly(styrene-butadiene-styrene) (SBS), one of the most productive and important thermoplastic elastomer

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(TPE), is widely used in various domains, such as footwear, impact modifiers in engineering plastics and adhesives, because of its high flexibility and elasticity. To obtain higher performance material, many researchers have prepared composites using SBS as a polymer matrix and montmorillonite,<sup>26,27</sup> carbon nanotubes,<sup>28–30</sup> alumina,<sup>31</sup> calcium carbonate,<sup>32</sup> and others as fillers. These composites have better tensile property, thermal resistance, and other properties compared with pure SBS. However, these inorganic materials can not endow other especial properties to SBS such as luminescence property.

From above, we know that no research was reported about using SiO<sub>2</sub>-hybridized CdTe QDs to prepare of SBS composites. Therefore, in this article, the transparent SBS composites filled with CdTe QDs were fabricated by a direct dispersion method. The composites were then thoroughly characterized by fourier transform infrared (FTIR) spectroscopy, ultraviolet-visible (UV-vis), PL, transmission electron microscopy (TEM), dynamic mechanical thermal analysis (DMTA), and instron mechanical tester. Furthermore, the luminescence property of composites was also studied.

## EXPERIMENTAL

### Materials

The triblock polymer SBS was provided by Baling Oil Chemical Industry Company (Yueyang, China) and used without further purification. Gel permeation chromatography analysis of the SBS in tetrahydrofuran gave  $M_n$  fractions of 22,800 (PB) and 68,400 (PS) with polydispersity of 1.15. SiO<sub>2</sub>-hybridized CdTe QDs was synthesized in our laboratory.<sup>33</sup> All the other chemicals were used as received without further treatment.

### Synthesis of SBS/CdTe QDs composites

The SBS/CdTe QDs composites were prepared as follows. White SBS grain (3 g) was dissolved in toluene (30 mL). After SBS was dissolved, a certain amount of QDs solution (30 mg QDs were dispersed in 100 mL toluene under ultrasonication) was added into SBS solution, and the mixture was stirred for 2 h at 50°C under the protection of nitrogen. To investigate the effect of diameter of QDs on the properties of the composites, two kinds of SiO<sub>2</sub>-hybridized QDs were used, the diameter for the first one is 3 nm (CdTe QDs-3), and the diameter for the other is 4 nm (CdTe QDs-4). Subsequently, the mixture was poured into a petri dish, the toluene was allowed to freely evaporate. Thus, the homogeneous SBS/CdTe QDs composites were obtained after complete evaporation of the toluene. The typical film thickness

was measured to be 300–500 μm (measured by micrometer). For comparison, the pure SBS thin film was made following the same procedures as those used for fabrication of SBS/CdTe QDs composites.

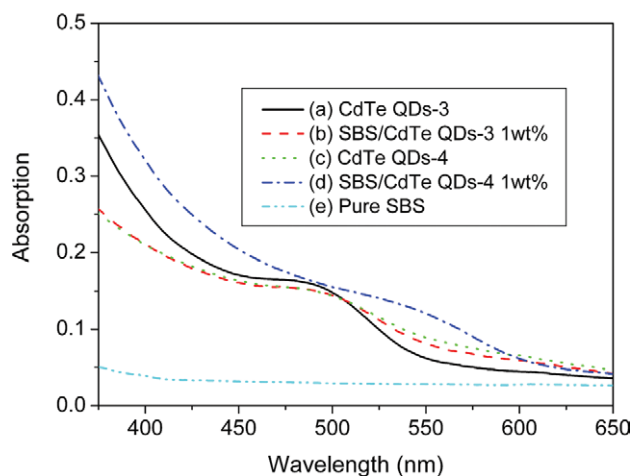
### Characterization

Ultraviolet-visible (UV-vis) absorption spectra were taken onto a Perkin-Elmer Lambda 900 UV-vis spectrometer with the scan range of 350–650 nm using toluene as the solvent, and all the UV-vis samples were diluted 60 times with toluene for analysis. All samples are measured three times and the average values have been presented. PL spectra were measured on a VARIAN Cary Eclipse fluorescence spectrophotometer operating with a 380 nm laser beam as a light source and with Xe lamp as excited source, tube voltage was 800 V, and the excitation and emission slits were both 5 nm. All the PL samples were diluted 60 times with toluene for analysis. All samples are measured three times and the average values have been presented. The dispersion of the CdTe QDs in the composites was examined using a TEM from JEM-3010, operating at 200 kV. Sample for the TEM analysis was prepared from the central cross section of the extruded pellets normal to the flow direction. Ultrathin sections of ~ 50 nm in thickness were cryo-cut using a Reichert-Jung Ultracut E microtome and a diamond knife at –50°C. DMTA was carried out on the TA Instruments DMTA-V with a tensional module at the frequency of 1 Hz and at the heating rate of 5°C/min from –120 to 110°C. The specimens (size 1.5 × 6.5 × 50 mm<sup>3</sup>) were cut from the center of the samples. The tensile properties of the samples were measured in accordance with ISO 527 at room temperature (about 20°C) using a tensile tester (Instron-4302). The stress-strain experiments have been done three times for each sample and the average values have been presented.

## RESULTS AND DISCUSSION

### UV-vis absorption spectra of SBS, SiO<sub>2</sub>-hybridized CdTe QDs, and SBS/CdTe QDs composites

To confirm that the CdTe QDs are well dispersed in the SBS matrix, the UV-vis absorption spectra of SBS/CdTe QDs composites with different diameter of CdTe QDs were measured. Figure 1 shows UV-vis absorption spectra of pure SBS, SiO<sub>2</sub>-hybridized CdTe QDs, and SBS/CdTe QDs composites diluted with toluene. As seen in Figure 1, there is no absorptions peak in pure SBS, and the absorptions peaks at 480 nm can be observed in pure CdTe QDs-3 and SBS/CdTe QDs-3 composites, according to Brus's mass model,<sup>34</sup> the particle size of CdTe QDs in the



**Figure 1** UV-vis absorption spectra of (a) pure CdTe QDs-3; (b) SBS/CdTe QDs-3 composites, CdTe QDs-3 wt % = 1.0 wt %; (c) pure CdTe QDs-4; (d) SBS/CdTe QDs-4 composites, CdTe QDs-4 wt % = 1.0 wt %; (e) pure SBS. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

composites is 3.2 nm, which indicates that the particle size of CdTe QDs in the composites is still kept at about 3 nm.<sup>33</sup> However, UV-vis absorption spectra of the SBS/CdTe QDs-4 composites display an obvious red shift comparing with the pure CdTe QDs-4 whose peak is at 500 nm. This is because the size of QDs-4 is bigger compared with QDs-3, the solubility of QDs-4 is bad, and so the QDs-4 in the SBS matrix is easy agglomeration. The other reason may be explained that the effect of band broadening is that the surface of CdTe QDs and the particle size of CdTe QDs change when CdTe QDs are embedded in SBS matrix. The result indicates that CdTe QDs-3 is more suitable for preparation of SBS composites compared with CdTe QDs-4.

#### Fluorescence emission spectra of SBS, SiO<sub>2</sub>-hybridized CdTe QDs, and SBS/CdTe QDs composites

As a typical semiconductor, CdTe QDs exhibit interesting optical properties. With the CdTe QDs embedded in the SBS matrix, the SBS/CdTe QDs composites also shows excellent optical characteristic property. Figure 2 shows the fluorescence spectra of CdTe QDs, pure SBS, and SBS/CdTe QDs composites with excitation at 380 nm, respectively. A peak at 535 nm was observed in both pure CdTe QDs-3 and SBS/CdTe QDs-3 composites, which was characteristic peak for CdTe QDs.<sup>33</sup> No fluorescence phenomenon was observed in pure SBS in the observed wavelength ranging from 500 to 700 nm. At the same time, the effect of different diameter of CdTe QDs on fluorescence property was studied. Fluorescence spectra of the SBS/CdTe QDs-4 composites display an obvious shift comparing with the pure CdTe QDs-4 whose peak is at 586 nm. Usually, the

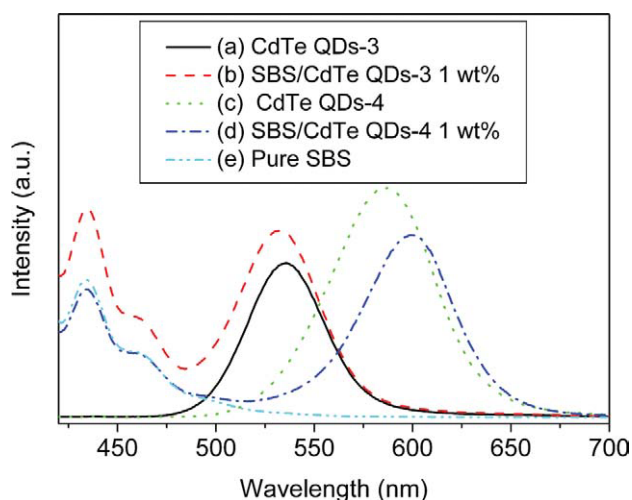
location and bandwidth of CdTe fluorescent bands are related to the size and the nature of carrier trap states located at the surface of QDs.<sup>20</sup> It may be explained that an effect of band broadening is that the surface of CdTe QDs and the particle size of CdTe QDs change when CdTe QDs are embedded in SBS matrix. Furthermore, the size of QDs-4 is bigger compared with QDs-3, the solubility of QDs-4 is bad, and so the QDs-4 in the SBS matrix is easy agglomeration. The spectrum difference indicates that CdTe QDs-3 is more suitable for preparation of SBS composites compared with CdTe QDs-4, consistent with the UV-vis absorption spectra analysis.

#### Morphologies of the SBS/CdTe QDs composites

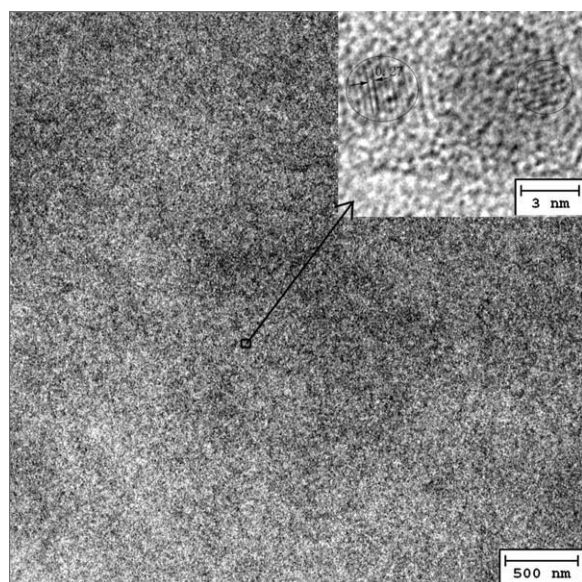
Figure 3 demonstrates the typical TEM and HRTEM images of the SBS/CdTe QDs composites. In Figure 3, no obvious aggregation was observed in the SBS polymer matrix. From the inset of Figure 3 (with the scale bar of 3 nm), it confirms the size of the CdTe QDs is about 3 nm, which is in agreement with the size of material and the calculated result by Brus's mass model. The inset of Figure 3 also shows the presence of microstructures in the coarsened CdTe QDs, and the existence of lattice planes on the magnified CdTe QDs may further confirm the crystallinity of CdTe QDs. The above results show that CdTe QDs as-prepared still behave QDs in the SBS matrix.

#### Mechanical properties of the SBS/CdTe QDs composites

Figure 4 and Table I show the representative tensile properties of SBS and its composites prepared by

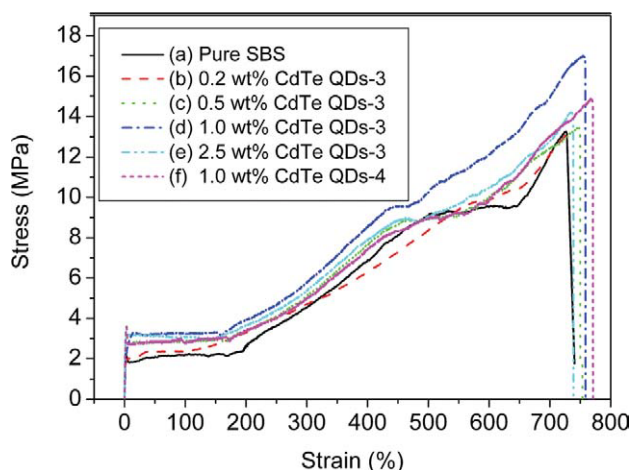


**Figure 2** Fluorescence emission spectra of (a) pure CdTe QDs-3; (b) SBS/CdTe QDs-3 composites, CdTe QDs-3 wt % = 1.0 wt %; (c) pure CdTe QDs-4; (d) SBS/CdTe QDs-4 composites, CdTe QDs-4 wt % = 1.0 wt %; (e) pure SBS, dispersed in toluene with excitation at 380 nm. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



**Figure 3** TEM images of CdTe QDs dispersed in SBS. Insert is HR TEM of SBS/CdTe QDs composites

using different amounts and sizes of CdTe QDs. The addition of CdTe QDs can reinforce the composites significantly without sacrificing their deformability. As seen in Figure 4, the tensile strength of composites increases gradually with increasing CdTe QDs-3 content before 1 wt % CdTe QDs-3. When 1 wt % CdTe QDs-3 is added, the tensile strength of composites reaches to the maximum about 16.96 MPa, however, with sequentially increase in the content of the CdTe QDs-3, the tensile strength of composites becomes to decrease. This may be attributed to excessive CdTe QDs-3 resulted in phase separation. A similar phenomenon is observed in Table I for elongation at break and tensile strength at 300%. This indicates that the addition of CdTe QDs produces a significant effect on the tensile strength and elasticity



**Figure 4** Strain-stress curves of the SBS and SBS/CdTe QDs composites. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

**TABLE I**  
Mechanical Property of the SBS and SBS/CdTe QDs Nanocomposites

Samples	Tensile strength		Elongation at break (% $\pm$ 10)
	(MPa $\pm$ 1)	at 300% (MPa $\pm$ 1)	
Pure SBS	13.26	4.55	728
0.2 wt % CdTe QDs-3	13.38	4.76	734
0.5 wt % CdTe QDs-3	13.47	5.12	747
1.0 wt % CdTe QDs-3	16.96	5.78	756
2.5 wt % CdTe QDs-3	14.20	5.36	736
1.0 wt % CdTe QDs-4	14.87	5.04	765

of SBS. The mechanism of how the addition of CdTe QDs affects mechanical properties of composites needs to be studied further.

#### DMTA analysis of the SBS/CdTe QDs composites

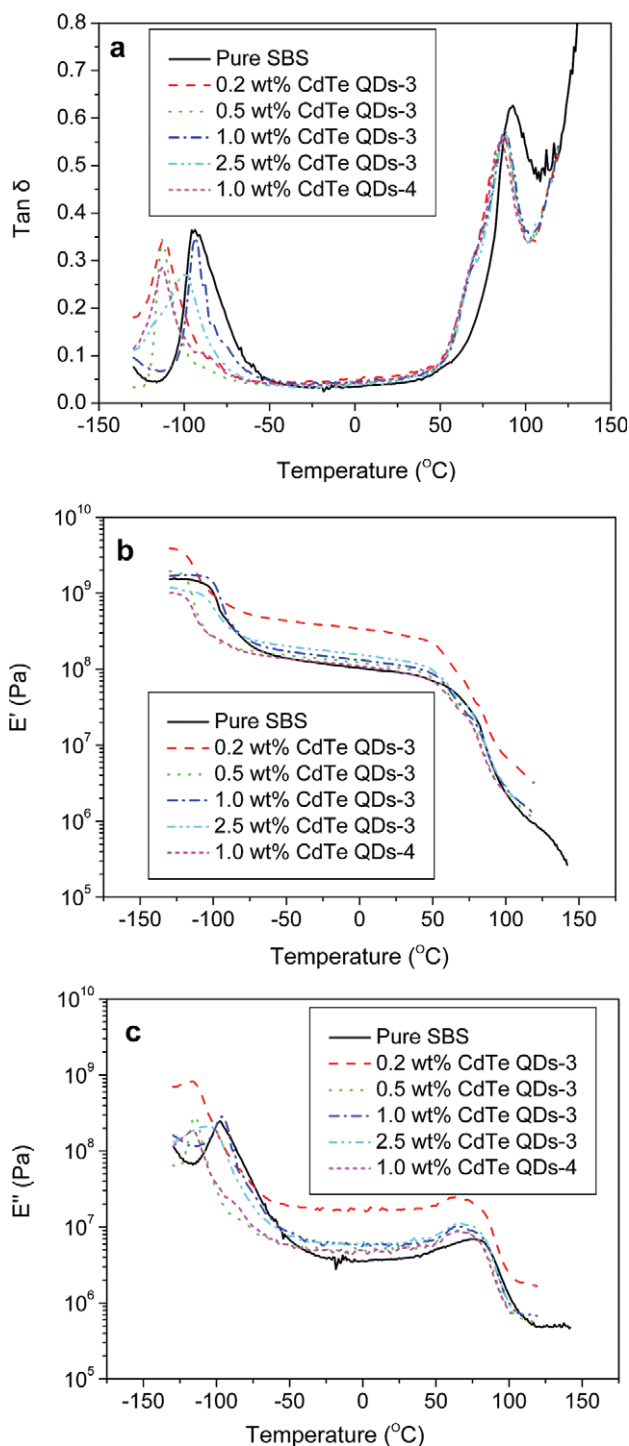
The loss factor ( $\tan \delta$ ), dynamic storage modulus ( $E'$ ), and dynamic loss modulus ( $E''$ ) versus temperature for the pure SBS and SBS/CdTe QDs composites are plotted in Figure 5. As shown in Figure 5(a), in all cases, two-glass transition temperatures are observed, which shows that the structure of microphase separation is in existence. The lower temperature transition at  $-100^\circ\text{C}$  is characteristic of the polybutadiene domains, whereas the higher temperature transition, at about  $95^\circ\text{C}$ , is characteristic of the polystyrene domains. As the addition of CdTe QDs and its content increase, the value of  $\tan \delta$  relevant to the PB and PS domains decreases. This may be attributed to the interaction between the QDs and polymer matrix. Furthermore, there is no obvious effect of size of CdTe QDs on  $T_g$  of SBS.

In the  $E'$  and  $E''$  curves [Fig. 5(b,c)], the variations of dynamic storage modulus and dynamic loss modulus as a function of temperature for the pure SBS and SBS/CdTe QDs composites containing different CdTe QDs amounts can be clearly observed. The dynamic moduli of the composites are much higher than that of pure SBS, and a considerable increase is observed in the range of temperature from  $-50$  to  $100^\circ\text{C}$ . It is probable that the addition of CdTe QDs could induce the reinforcement effect on SBS. The increasing trend becomes more significant when 1.0 wt % CdTe QDs-3 are added, but it is less with increasing CdTe QDs-3 content further. In addition, the size of CdTe QDs has almost no effect on  $E'$  and  $E''$ . This is consistent with the increase of the mechanical properties of composites.

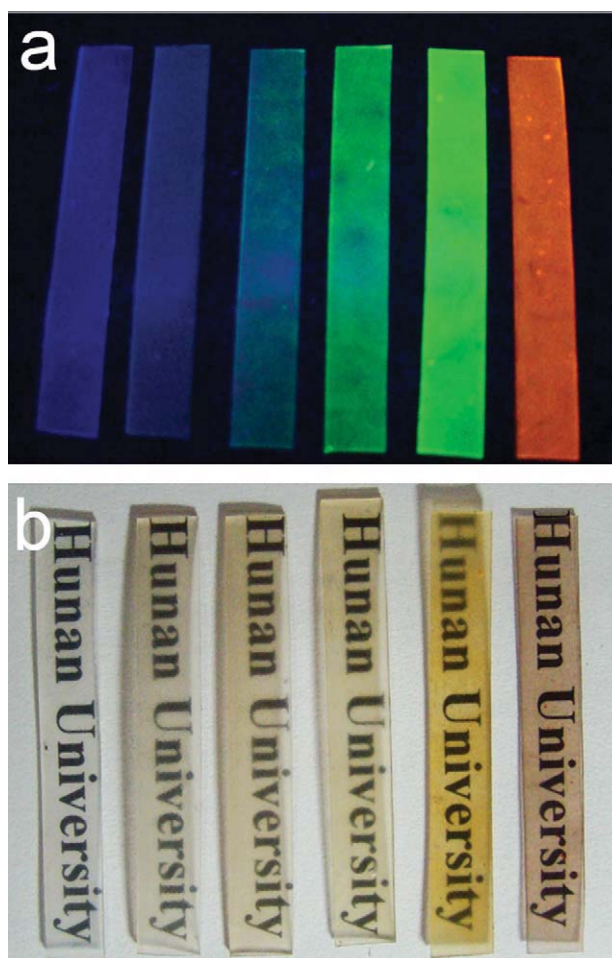
#### Photographs of the SBS/CdTe QDs composites

Figure 6 shows the photographs of the pure SBS and SBS/CdTe QDs composites taken under daylight

and ultraviolet light. Pure SBS is colorless and transparent under daylight, and SBS/CdTe QDs composites is transparent and light color taken under daylight. In addition, with the content of CdTe QDs increase the color of the composites increased.



**Figure 5** Dynamic mechanical thermal spectra of the SBS and SBS/CdTe QDs composites: (a) temperature dependence of  $\tan \delta$ ; (b) temperature dependence of dynamic storage modulus ( $E'$ ); (c) temperature dependence of dynamic loss modulus ( $E''$ ). [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



**Figure 6** Photographs of pure SBS and CdTe QDs in composites under UV light, 365 nm, (a) and daylight (b). From left to right the sample is pure SBS, 0.2 wt % CdTe QDs-3, 0.5 wt % CdTe QDs-3, 1 wt % CdTe QDs-3, 2.5 wt % CdTe QDs-3, and 1 wt % CdTe QDs-4, respectively. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

Furthermore, when taken under 365 nm ultraviolet light the composites show light green and red for SBS/CdTe QDs-3 composites and SBS/CdTe QDs-4 composites, respectively. Also, the luminescent strength of composites enhanced with the increase of CdTe QDs concentration. In conclusion, SBS/CdTe QDs composites also exhibit good optical characteristic property, and the color of the composites can be facilely controlled by adjusting the size of the CdTe QDs.

## CONCLUSIONS

In summary, SiO<sub>2</sub>-hybridized CdTe QDs were used to prepare transparent SBS/CdTe QDs composites thin film by a direct dispersion method. The FTIR spectra of the composites indicated there was interaction between the CdTe QDs and the polymer matrix. With the aid of UV-vis spectrophotometer, the

composites also showed absorption, similar to the pure CdTe QDs solution. The composites showed a compromised absorption of the polymer and the nanosized fillers. During fluorescence test, an intense emission peak for SBS/CdTe QDs composites was observed. The results of TEM revealed CdTe QDs well dispersed in the SBS matrix, and the lattice planes of CdTe QDs were still existence. The experimental results of DMTA showed that the addition of CdTe QDs had an important effect on the thermal property of SBS. In addition, mechanical tests revealed that SBS/CdTe QDs composites had better mechanical properties compared with that of pure SBS. Also, the luminescent photographs taken under ultraviolet light proved that these composites possessed good luminescence property. With the fluorescent SiO<sub>2</sub>-hybridized CdTe QDs as nanofillers, these elastomeric composites have great potential applications in the biomedical and optical area.

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