

Research Article

Multifunctional Composites Obtained by Incorporating Nanocrystals into Decorated PVK Polymers

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Poly(vinylcarbazole) (PVK) was decorated with surfactant group to achieve amphiphilic polymer with luminescent property. The composition and properties of the polymers were systematically investigated using FTIR, EA, TGA, UV-Vis, and PL characterizations. Different CdTe nanocrystals (NCs) prepared in aqueous medium were directly transferred to organic phase using the PVK-based polymers. The quantum yield of NCs in the composites had been improved by 50% compared with their parent aqueous solution due to the short distance from carbazole moieties to NCs, which facilitated the Förster resonant energy transfer (FRET) between them. Moreover, efficient electron transfer at the interface of NCs and polymers had been confirmed which also indicated the application in photovoltaic cell for such composites.

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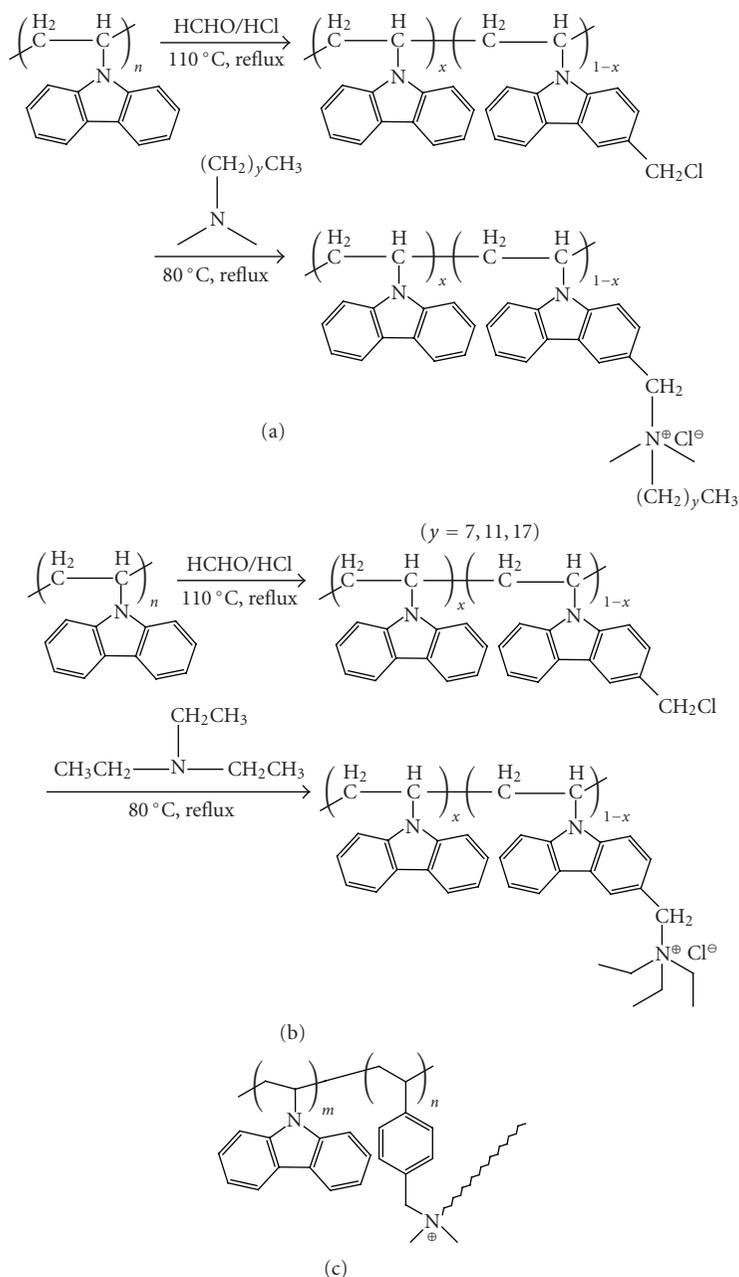
1. INTRODUCTION

Nowadays, a novel kind of luminescent polymers, namely, polymer with amphiphilic groups, has been extensively studied because such kind of polymers possesses several advantages. (1) They can offer particular properties compared with the normal neutral polymers due to their ionic side groups attached to the main chain. (2) Most amphiphilic polymers are typically soluble in water or alcohol, which are environment-friendly solvents. (3) Amphiphilic polymers are important building blocks for nanocrystal assembly via electrostatic interaction [1]. These advantages donate amphiphilic polymers with much more extensive applications; they can be used as the active layer in light-emitting diodes (LEDs) through layer-by-layer self-assembly method [2]; they are considered as highly sensitive materials in biological [3] and chemical sensors as well [4].

Semiconductor nanocrystals (NCs) directly synthesized in aqueous solution using water-soluble thio-compounds as ligands have intrigued much interests in recent years, because they are cheaper, less toxic, and biocompatible in comparison with the ones prepared in nonaqueous synthesis [5–8]. Many researchers pay attention to how to incorporate NCs into the polymers, because people expect that the resulted NC-polymer composites will combine the ad-

vantages of both NCs and polymers. Luminescent polymers with amphiphilic groups naturally become one of the best choices to realize this aim. In our recent work [9, 10], we have developed a novel method to incorporate NCs into functional amphiphilic polymers. First, we designed a novel amphiphilic copolymer of poly(9-vinylcarbazole-co-octadecyl-4-vinylbenzyl-dimethyl-ammonium chloride) (CPVKOVDAC) with blue fluorescence. Then, NCs in aqueous phase were transferred to organic phase using the polymers to achieve NC-polymer multifunctional composites. Moreover, this method provided a chance of incorporating several different color-emitting NCs into a functional polymer. Therefore, by incorporating two types of CdTe NCs having two distinct PL emissions at 524 and 650 nm into the polymer, white-light PL and electroluminescence (EL) have been obtained from the solution processable NC-polymer composite. However, the quantum yield (QY) of the composites was low and hence we could not get the EL devices with high efficiency.

In this paper, we directly grafted surfactant group on poly(vinylcarbazole) (PVK) (see Scheme 1) in order to achieve PVK-based amphiphilic polymer. Different CdTe NCs prepared in aqueous medium were directly transferred to the organic phase using the PVK-based polymers. Compared with the CPVKOVDAC copolymer which we used in



SCHEME 1: (a), (b) Synthesis route of PVK-N⁺ polymers. (c) The structure of CPVKOVDAC.

previous studies, the novel polymer had several advantages. It was (1) simple to characterize and had (2) better property of carrier transport. (3) The length of alkyl chains could be changed easily, which allowed us to investigate the effect of alkyl chain length on the PL properties of NCs. (4) The quantum yield of NCs had been improved by 50% due to the shorter distance between carbazole groups and NCs. The results of interaction between polymers and CdTe NCs indicated that the CdTe NC-PVK-based composites would possess good luminescent or photovoltaic properties just by varying the content of CdTe NCs in the composites.

2. EXPERIMENTAL

2.1. Materials

Poly(9-vinylcarbazole) (M_w 90 000) and tellurium powder (−200 mesh, 99.8%) were purchased from Sigma-Aldrich Chemical Corporation (Mo, USA). 3-Mercaptopropionic acid (MPA), thioglycolic acid (TGA), N, N-dimethyl octadecylamine, N, N-dimethyl dodecyl amine, N, N-dimethyl octyl amine, and 4-vinylbenzyl chloride were purchased from Acros Organics (UK). CdCl₂ (99+%), NaBH₄ (99%), and

triethylamine were commercially available products. All of the solvents had analytical grade and were used as received.

2.2. Synthesis of decorated PVK polymer

Concentrated hydrochloric acid (2.3 mL, 36.7%) and aqueous formaldehyde (1.25 mL, 30%) were added to a stirred solution (0–5°C) of PVK (1 g) in 1,4-dioxane (100 mL). The reaction mixture was allowed to warm to room temperature and was stirred for 1 hour. The reaction mixture was then heated to reflux for 4 hours. Cooling back to 80°C, tertiary amines were added to the reactant mixture and reacted for 4 hours. The mixtures were poured into a large amount of methanol to remove the unreacted small molecules, and the polymers were obtained by centrifugation. The polymer was dissolved in chloroform and precipitated in methanol several times to achieve pure decorated PVK polymer. Then, the product was kept at 40°C in vacuum overnight (FTIR (KBr): 2923, 2851 (ν , C–H stretch vibrations in octadecyl), 1624, 1595 (two aromatic vibrations in carbazole moieties), 1482, 1449 (vibrations in five-membered ring of carbazole), 1325, 1227 (ν , C–N stretch vibrations)).

2.3. Preparation of CdTe NC-PVK-based polymer composites

A series of aqueous solutions of CdTe nanocrystals capped with MPA or TGA was prepared according to previous reports [11]. The polymer was dissolved in chloroform at some concentration. The polymer solution was added to an aqueous CdTe solution (0.00125 N according to Cd^{2+}) with vigorous stirring. The organic phase was then separated to yield the CdTe NC-composite solution. The chloroform solvent was removed under reduced pressure and the resulting composite solids were kept in vacuum overnight. The composite solid can be dissolved again in chloroform for characterization.

2.4. Characterizations

FTIR spectra were recorded from a KBr window on a Nicolet AVATAR 360 FTIR spectrophotometer. UV-Vis spectra were acquired using a Shimadzu 3100 UV-Vis spectrophotometer. Fluorescence experiments were performed on a Shimadzu RF-5301 PC spectrofluorimeter. NETZSCH STA 449C thermogravimetric analyzer (TGA) with a heating rate of 10°C/min, up to 800°C, was used for the thermal degradation of the polymers under nitrogen. Element analysis was performed on Elementar Analysensysteme GmbH VarioEL. The quantum yields (QYs) of CdTe NC-polymer composites were determined using quinine sulfate (10^{-5} M in 0.5 mol/L H_2SO_4) as reference. The absorbance at the excitation wavelength was below 0.1 in order to avoid any significant reabsorption.

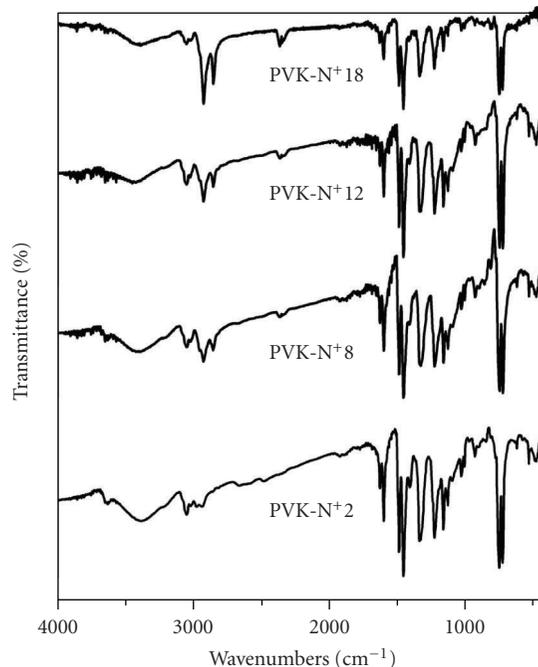


FIGURE 1: FTIR spectra of PVK-N⁺ polymers.

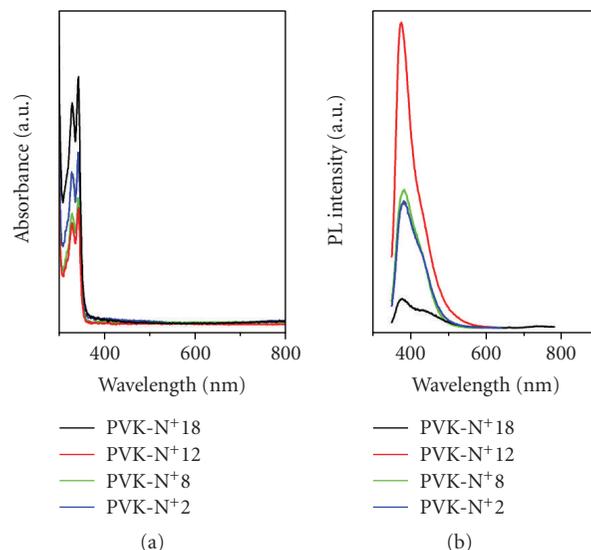


FIGURE 2: (a) UV-Vis absorption spectra and (b) PL spectra excited at 325 nm of the PVK-N⁺ polymers in chloroform.

3. RESULTS AND DISCUSSION

3.1. Synthesis and properties of PVK-based polymer

PVKs have been considered as potential polymer hosts for high-efficiency blue light-emitting diodes, and they found applications in photovoltaics, photoconductive materials, electroluminescence (EL) devices, photorefractive materials, nonlinear optical devices, and so forth [12–16]. It has been reported that chloromethylated PVK was functionalized with

TABLE 1: Basic information of the PVK-N⁺ polymers.

Abbreviations of the polymers	Tertiary amine used in the second step of the reaction	Numbers of quaternary amine decorated on the polymer chain per 100 carbazole moieties	Decomposition temperature (°C) (5% weight loss)
PVK-N ⁺ 18	N, N-dimethyl octadecylamine	25	215
PVK-N ⁺ 12	N, N-dimethyl dodecylamine	18	201
PVK-N ⁺ 8	N, N-dimethyl octylamine	21	211
PVK-N ⁺ 2	Triethylamine	15	209

Disperse Red 1 (DR-1) by Williamson ether synthesis [17]. Because of the higher reactivity of the carbazole ring toward electrophilic substitution, a serious cross-linking reaction occurred in preparation of chloromethylated PVK using chloromethyl methyl ether and zinc chloride, leading to the low content of chloromethyl in the soluble part of chloromethylated PVK. Consequently, further chromophoric functionalization was not easy to complete. In our studies, it was also found that the molecular weight, temperature of reaction, time of reflux, and so forth had an important influence on the resulted polymers. The formation of cross-linking polymers would occur if the experiment conditions were not well controlled. Scheme 1 gives the synthesis route of PVK-based polymer and Table 1 shows the basic information of the polymers. For a given molecular weight of PVK, for example, $M_w = 90\,000$, the concentration of PVK in 1, 4-dioxane should be controlled below 10 mg/mL. The temperature of reaction should be controlled at 80–110°C. Temperature below this will result in low degree of chloromethylation and low yield of PVK-based polymer at last. The reflux time must be shorter than 4 hours in the first step of the reaction in order to avoid the formation of cross-linking polymers. In the second step of the reaction, the tertiary amine probably reacts with surplus hydrochloric acid in the first step to form small quaternary amine molecules. Therefore, we added excessive tertiary amine to insure that there was enough tertiary amine to react with the chloromethylated PVK polymers. The PVK-based polymer was dissolved in chloroform and precipitated in methanol to delete the small quaternary amine molecules. Figure 1 shows the FTIR spectra of the PVK-based polymers. We can clearly observe strong C–H stretch vibration peaks at 2923 and 2851 cm^{-1} , which resulted from alkyl in the amine moieties. The peaks at 1624 and 1595 cm^{-1} are attributed to the two aromatic vibrations in carbazole moieties. The peaks at 1482 and 1449 cm^{-1} resulted from the five-membered ring of carbazole. The peaks at 1325 and 1227 cm^{-1} are attributed to C–N stretch vibration. These characteristic peaks have verified the formation of PVK-based polymers. The numbers of quaternary amine decorated on the polymer chain are also listed in Table 1, which are estimated from element analysis (see Table 2). The decomposition temperature of the polymers was about 200°C, which could satisfy the requirement of fabricating device using spin-coating method.

Figure 2 shows the UV-Vis absorption and PL spectra of the polymers. The electron transition within carbazole moieties leads to the peaks at 343 and 330 nm of absorption spectra. The peaks at 365 nm of PL spectrum are attributed to

TABLE 2: Element analysis of the PVK-N⁺ polymers.

	C (%)	H (%)	N (%)	Cl (%)
PVK-N ⁺ 18	82.415	7.415	6.291	1.640
PVK-N ⁺ 12	83.310	6.379	6.936	1.000
PVK-N ⁺ 8	83.145	6.352	7.074	1.600
PVK-N ⁺ 2	83.260	5.760	7.201	1.700

the high-energy excimer radiation of carbazole moieties. The UV-Vis and PL spectra indicate that the resultant polymers retain the photophysical properties of PVK.

3.2. Composite of semiconductor Nanocrystals and PVK-N⁺

The PVK-N⁺ polymers are readily soluble in common organic solvents such as chloroform, dichloromethane, N, N-dimethylformamide, and so forth. Different CdTe NCs as prepared in aqueous medium were directly transferred to the organic phase using the PVK-N⁺18 polymers to achieve composites 1–5, respectively (see Table 3). In this process, the electrostatic interaction between negatively charged CdTe NCs and positively charged moieties in polymers was the driving force for the formation of composite. Figures 3(a) and 3(b) show the UV-Vis absorption and PL spectra of the resultant composites 1–5 in chloroform. Absorption and PL spectra at different wavelengths correspond to distinct CdTe NCs, which confirms the presence of NCs in the polymer and the successful transfer of NCs from the aqueous phase to the organic phase. Furthermore, the full width at half maximum (FWHM) of PL peaks of the composite is similar to that of the parent NCs as prepared in aqueous medium, which indicates that the excellent properties of CdTe NCs were retained after they were capped with polymers. A clear solution of NCs in organic solvent strongly indicates a homogeneous distribution of NCs in the composites (see Figure 3(c)). The QYs of CdTe NCs in the composites 1–5 are 0.612, 0.476, 0.168, 0.368, and 0.146, respectively. For composites 1, 2, 3, and 5, the QYs are higher than their parent NC aqueous solutions. When we used polymer CPVKOVDAC to obtain NC composites, the QYs of CdTe NCs in the composites were always lower than parent NC aqueous solutions [9, 10]. The improvement of QY in this study is believed to be brought by the shorter distance from carbazole moieties to CdTe NCs in PVK-N⁺ system, which facilitates the Förster resonant energy transfer (FRET) between them.

TABLE 3: Component and quantum yields (QYs) of the composites.

Abbreviations of the composites	CdTe NCs used	QYs ^(b) of CdTe NCs in aqueous solution	QYs of CdTe NCs in the composites
Composite 1	TGA (530 nm) ^(a)	0.400	0.612
Composite 2	TGA (540 nm)	0.462	0.476
Composite 3	MPA (490 nm)	0.141	0.168
Composite 4	MPA (540 nm)	0.395	0.368
Composite 5	MPA (600 nm)	0.108	0.146

^(a) Reference to the CdTe NCs capping with TGA ligand and absorption of exciton at 530 nm. ^(b) Using quinine sulfate (10^{-5}) as PL reference.

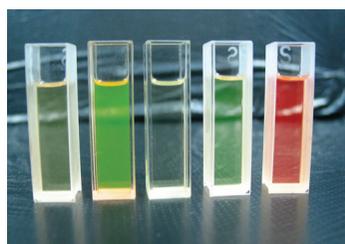
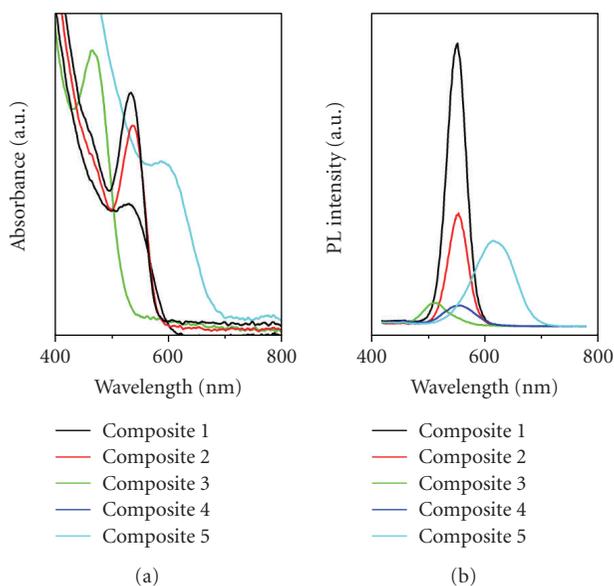


FIGURE 3: (a) UV-Vis absorption and (b) PL spectra excited at 400 nm of CdTe NC-polymer composites 1–5 in chloroform containing different CdTe NCs. (c) Photographs of composites 1–5 in chloroform.

3.3. Interaction between CdTe NCs and PVK-N⁺

Several methods have been developed to obtain the composites such as directly mixing the NCs into the polymers, in situ synthesis of NCs in polymer matrix, layer-by-layer assembly to achieve NC-polymer composite, and so forth [18–24]. Although some composites have been obtained using these methods, new problems also confronted chemists, such as aggregation of NCs in polymer matrix and PL quenching due to the different kinds of interaction between polymers and NCs. In our system, the process of composition of poly-

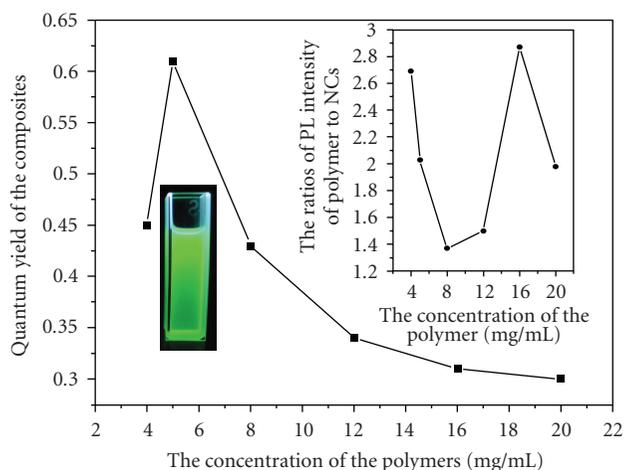


FIGURE 4: The curves of QYs versus concentration of polymer. Inset is the photograph of the composites with QY of 0.612, and the curves of the ratios of PL intensity of polymer to NCs versus concentration of polymer.

mer and CdTe NCs overcomes the major problems associated with the miscibility of NCs with polymer matrix and PL quenching of NCs in the polymer. However, we did find that the photophysical properties of NCs were influenced by the electrostatic interaction between polymers and NCs. Figure 4 shows that the QYs of the composites increase with increasing polymer concentration when the concentration of the polymer is below 5 mg/mL (the contents of CdTe were kept constant and the CdTe NCs were completely transferred from aqueous phase to organic phase). We conclude that the interaction between carbazole and CdTe NCs is mainly FRET whose energy is transferred by the excited carbazole moieties to CdTe NCs via dipole-dipole interaction at such concentrations. The highest QY was 0.612 when the concentration of the polymer was equal to 5 mg/mL. Inset of Figure 4 is the photo of the composite with the highest QY under UV illumination. The QY of CdTe NCs decreases with increasing polymer concentration when the concentration of the polymer is higher than 5 mg/mL. We suppose that electron transfer from carbazole moieties and CdTe NCs occurred, which quenched the PL of the resultant composites. For the PVK-N⁺ polymer, the PL intensity increases with increasing polymer concentration from 5 mg/mL to 20 mg/mL. However, from the inset curve of Figure 4, it can be observed that

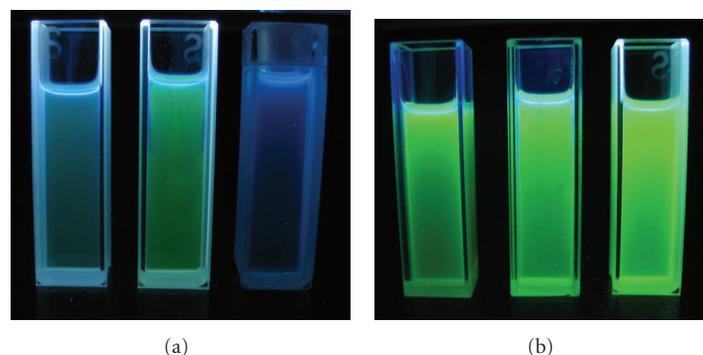


FIGURE 5: The photographs of (a) the composites with low-content CdTe NCs (the QYs of the NCs in the composite were 0.043, 0.107, and 0.061 from left to right) and (b) high-content CdTe NCs (the QYs of the NCs in the composite were 0.489, 0.571, and 0.612 from left to right). The photographs of (a) and (b) were taken under the same condition. The composites with low content of CdTe NCs refer to the composites obtained via 10 mL (5 mg/mL) PVK-N⁺ polymer solution adding 10, 20, 40 mL CdTe NC aqueous solutions, while the composites with high content of CdTe NCs refer to the composites obtained via 10 mL (5 mg/mL) PVK-N⁺ polymer solution adding 60, 80, 100 mL CdTe NC aqueous solutions.

the ratios of PL intensity of polymer to NCs decrease first and then increase when the polymer concentration increases continuously. These results indicate that the most efficient FRET occurred at the point at which the concentration of the polymer was about 8 mg/mL.

The tendency of PL changes of the composites became much more obvious when the concentration of the polymer was set constant, namely, 5 mg/mL, and the content of NCs was varied at some range. In CdTe NC aqueous solution, the QYs of the NCs increase with decreasing NC concentration because the self-quenching occurs at higher concentration. In the composite system, however, when the concentration of the polymer was about 5 mg/mL, the QYs of the NCs were higher in the composites with high content of CdTe NCs than those of the composites with low content of CdTe NCs. Figure 5 gives the photos of the composites with different contents of CdTe NCs under UV illumination. It can be clearly observed that the composites with low content of CdTe NCs show weak PL intensity (see Figure 5(a); the QYs of the NCs in the composite are 0.043, 0.107, 0.061 from left to right) while the ones with high content of CdTe NCs show strong PL intensity (see Figure 5(b); the QYs of the NCs in the composite are 0.489, 0.571, 0.612 from left to right). This phenomenon verifies the conclusion we have drawn above. We have ever systematically studied in our recent work [25] the mechanism of interaction between carbazole moieties and CdTe NCs. It is believed that the excited carbazole moieties could form carbazole anions and carbazole cations via resonance with carbazole moieties at the ground state under UV illumination. The carbazole anions migrated to the positively charged surface of CdTe NC-polymer composite and then shortened the distance between carbazole moieties and CdTe NCs. Carbazole anions donated their electrons to CdTe NCs, and then the donated electrons recombined with the holes in CdTe NCs. The result of electron transfer was that holes were confined to polymers while electrons were limited within CdTe NCs. The more the polymer was, the more positive net charges and carbazole moieties were. Therefore, the electron transfer became efficient with decreasing CdTe NCs.

4. CONCLUSION

In summary, we prepared a novel kind of PVK-based amphiphilic polymer with luminescent properties. CdTe NCs were directly transferred to the organic phase using the PVK-based polymers to achieve composites. The quantum yield of NCs had been improved by 50% compared with their parent aqueous solution due to the shorter distance from carbazole groups to NCs which facilitated the FRET between them. More efficient electron transfer at the interface of NCs and polymers was confirmed for composites by decreasing NC content. The results indicated that the composites could find applications in LEDs or photovoltaic devices.

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