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Cyclotriveratrylene–carbazole cage for self-assembly of C_{60}

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ABSTRACT

A novel open-bowl pyramidal compound cyclotriveratrylene–carbazole (CTV–CZ) was designed and synthesized. Its absorption and emission spectroscopic properties in different organic solvents were characterized. Three absorption peaks of CTV–CZ around 235 nm, 260 nm and 290 nm were observed. The fluorescence emission peaks were at about 353 nm and 368 nm using a solution of $\sim 10^{-7}$ M CTV–CZ. The intermolecular interaction between CTV–CZ and fullerene (C_{60}) was studied in detail by UV–vis absorption and fluorescence spectra. CTV–CZ could associate with C_{60} to form supramolecular complex with 1:1 molar ratio and the binding constant was estimated to be $5.0(6) \times 10^4\ M^{-1}$ by fluorimetry. The formation of inclusion complex for CTV–CZ and C_{60} was further confirmed by ^1H NMR and cyclic voltammetry. The downfield shift of protons of CTV–CZ in its ^1H NMR spectrum and the decrease of redox currents on addition of C_{60} showed that photo induced electron transfer (PET) occurred from the electron donor CTV and CZ units to the electron acceptor C_{60}.

1. Introduction

Fullerenes as the third carbon allotrope have attracted increasing attention in chemical research since discovered [1]. Fullerenes' exciting physical and chemical properties have arguably centered most of the research and application fields, such as superconductors, photoconductors, catalysts, and molecular recognition. C_{60}, the most representative member of fullerenes, has been demonstrated excellent development of organic electrical and optical devices [2–5]. For example, C_{60} is an excellent electrical conductor as good as copper metal and is one of the strongest materials known, being 100 times stronger than steel [6–8]. Moreover, C_{60} as an electron acceptor played an important role in intermolecular photoinduced electron transfer (PET) [9]. Hence, searching supramolecular scaffolds as receptors for C_{60} in order to fulfill the goals of affinity and selectivity has been of great importance. Many other receptors including calixarenes [10], calixpyrroles [11], corannulenes [12], cryptophane [13] or tribenzotriquinacenes [14] have been employed as scaffolds for the recognition of fullerene. However, relatively few receptors such as cyclotriveratrylene (CTV) and its derivatives as supramolecular scaffolds for fullerene were involved [15,18].

Atwood et al. [16,17] first reported the bowl-shaped crown pattern CTV and C_{60} could form inclusion complex with a 'ball-and-socket' feature structure. Since then, template CTV as a new supramolecular host has opened avenue for recognizing C_{60}. CTV possesses the unique structure with a pyramidal shape and rigid electron-rich molecular cavity and is able to accommodate size suitable molecules including neutral or ionic polyhedral C_{60} and *o*-carborane derivatives [18,19]. It has received great interest due to its good chemical stability, stable conformation, and facile modification [20–24]. However, the further application of CTV platform is limited due to its shallow cavity. Matsubara et al. [15,25] tested associating two or more electron rich units could improve the affinity and selectivity of the CTV unit receptor for C_{60}. Various CTV derivatives with extended cavities have been also developed for recognizing C_{60}. Modification with appending functional groups can extend side-arms of CTV, which expands the cavity effectively and enhances the binding ability to C_{60} in solution. De Mendoza [26] introduced a fully conjugated unit 2-[9-(1,3-dithiol-2-ylidene)-anthracen-10(9*H*)-ylidene]-1,3-dithiole (exTTF) to CTV forming the host exTTF-CTV with effective association with C_{60} due to the combination of shape and electronic complementary between exTTF and C_{60}. ExTTF-CTV could be used to purify C_{60} efficiently from crude soot or fullerite mixtures based on the formation of stable complex with C_{60} in organic solvent [27,28]. Han [29] proposed that CTV functionalized by glucose and lactose residues (CTV-G and CTV-L) could form water-soluble supramolecular complexes with C_{60} and the corresponding binding constants were $1.38 \times 10^5\ M^{-1}$ and $5.09 \times 10^4\ M^{-1}$, respectively.

The preparation of CTV derivatives is of great interest due to their unique bowl structure. They might be good candidates for the biological activity applications of fullerenes both in vitro and

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in vivo [30]. we designed an electron-rich compound carbazole to modify CTV to obtain cyclotriveratrylene–carbazole (CTV–CZ) as carbazole owed the same characters as exTTF. The concave surface of both CTV and the carbazole subunits will nicely wrap around the entrapped fullerene guest. In addition, carbazole possesses a fully conjugated system with the great ability to donate electron. Photoinduced electron transfer (PET) from the electron donor carbazole to the electron acceptor C_{60} will occur in the excited state. In this article, CTV–CZ was first synthesized and its interaction with C_{60} was investigated in detail by absorption and fluorescence spectroscopy. The formation of inclusion complex between CTV–CZ and C_{60} was further confirmed by cyclic voltammetry and ^{1}H NMR spectroscopy.

2. Experimental

2.1. Materials and chemicals

1,3-dibromopropane, acetonitrile, dichloromethane (CH_2Cl_2), cyclohexane, diethyl ethanol, ether, methanol, sodium borohydride ($NaBH_4$), ethyl acetate, and vanillin were purchased from Beijing Chemical Plant (Beijing, China). Fullerene (C_{60}) was purchased from Aladdin, without further purification. All chemicals solvents and reagents were used as received unless otherwise stated.

2.2. Synthesis of CTV–CZ

The synthetic routes to CTV–CZ are outlined in Scheme 1. 4-(3-bromopropoxy)-3-methoxybenzaldehyde (compound 1) was prepared from the reaction of vanillin with 1,3-dibromopropane. The bis-aldehyde derivative was reduced to its corresponding bis-vanillyl alcohol (compound 2) by using $NaBH_4$ in methanol. The cyclocondensation reaction of benzylic alcohol (compound 2) was performed in a perchloric acid solution to obtain compound 3 according to the literature procedure [31]. Cyclotriveratrylene–carbazole (CTV–CZ) was synthesized by the reaction of compound 3 and carbazole. The synthetic steps are as follows: To a solution of compound 3 (0.1 g, 0.124 mmol) and carbazole (0.079 g, 0.37 mmol) in 5 mL of dry DMF were added KOH (0.145 g, 2.59 mmol), and the reaction mixture was stirred and followed by reflux for 48 h before it was poured into ice–cold water and extracted twice with 20 mL portions of dichloromethane. The organic layer was then dried over anhydrous sodium sulfate and evaporated in vacuum. The crude product was purified by column chromatography using dichloromethane/diethyl ether (10/0.2, v/v) as the eluent to obtain CTV–CZ as a pale yellow solid (20 mg, 16% yield, mp 188–189 °C). ^{1}H NMR((300 MHz, $CDCl_3$)): δ (ppm) 6.55(s, 3H, Ar), 6.61 (s, 3H, Ar), 7.13(t, 3H, Ar),7.36 (t, 3H, Ar),7.37 (d, 3H, Ar),8.01(d, 3H, Ar), 3.58 (s, 9H, OCH_3), 4.51 (d, 3H, CHa), 3.37 (d, 3H, CHe), 3.85(m, 6H, NCH_2), 3.86 (m, 6H, OCH_2), and 2.30 (m, 6H, CH_2). MALDI–TOF–MS: calcd. for $C_{69}H_{63}N_3O_6$, 1030.26 (m/z); found 1029.47.

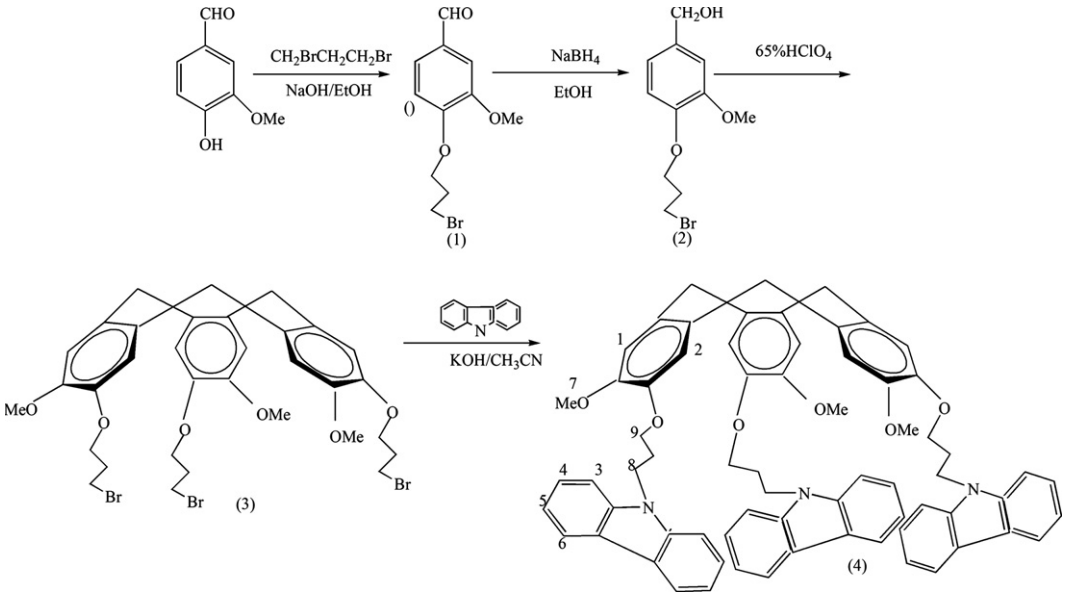
2.3. Instrument

UV absorption spectra were recorded on a Shimadzu UV-2450 spectrophotometer (Tokyo, Japan). Fluorescence spectra were taken on a Hitachi F4500 spectrofluorometer (Tokyo, Japan). Both excitation and emission slits were set at 5 nm. Cyclic voltammetric measurements were carried out on a CHI660C electrochemical workstation (Shanghai, China). ^{1}H NMR spectra were done on a Bruker Avance DRX 300 MHz nuclear magnetic resonance spectrometer (Fällanden, Switzerland). All the experiments were carried out at 20 ± 1 °C.

3. Results and discussion

3.1. Absorption and fluorescence spectral characteristics of CTV–CZ

The UV–vis absorption and fluorescence emission spectral maxima of CTV–CZ (2.92×10^{-6} M) in different solvents including cyclohexane, diethyl ether, dichloromethane (CH_2Cl_2), methanol, ethanol and acetonitrile were examined and shown in Table 1. CTV–CZ had three absorption peaks around 235 nm, 260 nm and 290 nm, respectively. The fluorescence emission maxima were at about 353 nm and 368 nm when the concentration of CTV–CZ was 1.73×10^{-7} M. The absorption and emission maxima of CTV–CZ did not change significantly with the solvent polarity possibly attributing to the rigid molecular structure.



Scheme 1. The synthesis routes of CTV–CZ.

Table 1
The absorption and fluorescence emission maxima of CTV–CZ in various organic solvents.

Solvent	Cyclohexane	CH_2Cl_2	Diethylether	Acetonitrile	Methanol	Ethanol
λ_{abs} (nm)	235/260/293	237/263/294	234/259/293	235/261/293	234/256/292	234/260/293
λ_{em} (nm)	350/365	353/369	351/365	350/365	352/366	351/365

λ_{abs} (nm): absorption peak maximum, λ_{em} (nm): emission maximum.

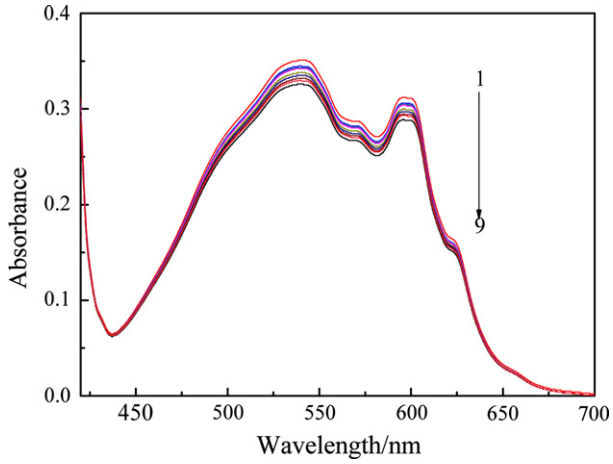


Fig. 1. The absorption spectra of C_{60} (3.90×10^{-4} M) in the absence and presence of various concentrations of CTV–CZ in toluene at room temperature. The concentrations of CTV–CZ ($\times10^{-5}$ M) were 0.0, 1.85, 2.5, 3.05, 5.0, 6.25, 7.4, 8.18 and 10.0, respectively.

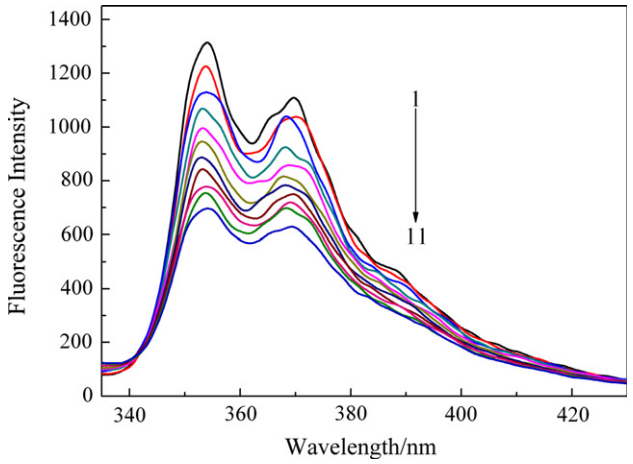


Fig. 2. Fluorescence quenching effect of C_{60} on the emission of 2.79×10^{-7} M CTV–CZ in CH_2Cl_2, (λ_{ex}=265 nm). The concentrations of C_{60} ($\times10^{-6}$ M) were 0.0, 0.92, 1.84, 2.76, 3.68, 4.6, 5.52, 6.44, 7.36, 8.28 and 9.2, respectively.

3.2. The absorption spectra of inclusion complex of CTV–CZ and C_{60}

CTV and its derivatives were able to bind C_{60} in solution. The binding ability to C_{60} was enhanced by extending the cavity of CTV derivatives [32]. The absorption spectrum of C_{60} possessed a strong absorption band at about 450–650 nm in the visible light region, deriving from the symmetry-forbidden electronic transitions and the symmetry-allowed vibronic transition [33]. Since CTV–CZ did not absorb at wavelengths longer than ~400 nm, the C_{60} absorption band at 410–650 nm was chosen to investigate the interaction between CTV–CZ and C_{60}.

Intermolecular interaction between CTV–CZ and C_{60} was investigated by absorption spectra. Fig. 1 showed the absorption spectra of C_{60} in the absence and presence of various concentrations of CTV–CZ in toluene. On addition of CTV–CZ to C_{60}, the maximum absorption intensity of C_{60} at 538 nm and 596 nm gradually dropped and the decrease was proportional to the concentration of CTV–CZ. This phenomenon might be attributed to the fact that three carbazoles directly attaching to the CTV phenol groups to form deep hole host, which could nicely wrap around the entrapped fullerene guest C_{60}. Thus, when CTV–CZ interacted with C_{60}, the photoinduced electron transfer (PET) from CTV–CZ to C_{60} might take place.

3.3. Fluorescence quenching of C_{60} on CTV–CZ

The interaction between CTV–CZ and C_{60} was also studied by spectrofluorometric titrations. The fluorescence quenching effect of C_{60} on CTV–CZ in CH_2Cl_2 was depicted in Fig. 2. CTV–CZ displayed strong fluorescence with emission maximum at 353 nm and 368 nm. Upon the addition of C_{60} solution of CH_2Cl_2, the fluorescence intensity of CTV–CZ obviously decreased. The phenomenon further indicated that the cavity of CTV–CZ not only accommodated size-suitable spherical C_{60} molecule but also provided microenvironment for the photoinduced intermolecular electron transfer process between CTV–CZ and C_{60}.

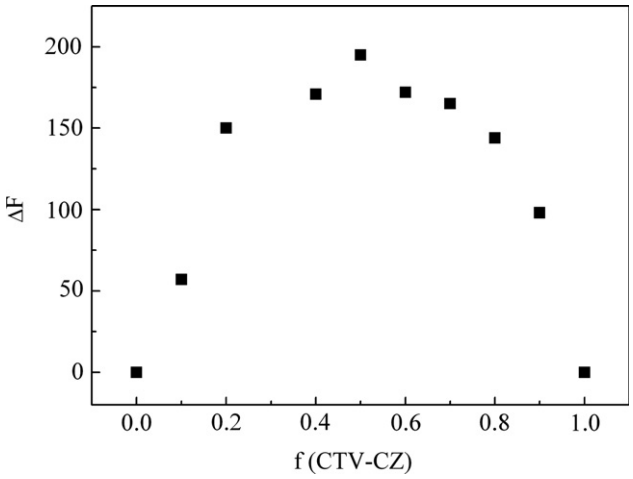


Fig. 3. Job's plot for the complexation of CTV–CZ with C_{60} in CH_2Cl_2 (λ_{em}=353 nm). The sum of the total concentration was constant ([CTV–CZ]+[C_{60}]=3.0×10^{-6} M) and f is the mole fraction of CTV–CZ added.

Job's plot is used to determine the stoichiometry of a binding event. Fig. 3 showed Job's plot of CTV–CZ and C_{60}. The maximum at 0.5 M concentrations indicated the formation of a 1:1 ratio host–guest complex in solution [34].

3.4. Binding constants

The binding constant (K) is an important parameter, indicating the inclusion capacity of the host–guest complex. The binding constant can be estimated by the least squares fit to the experimental data obtained from the fluorescence titrations as

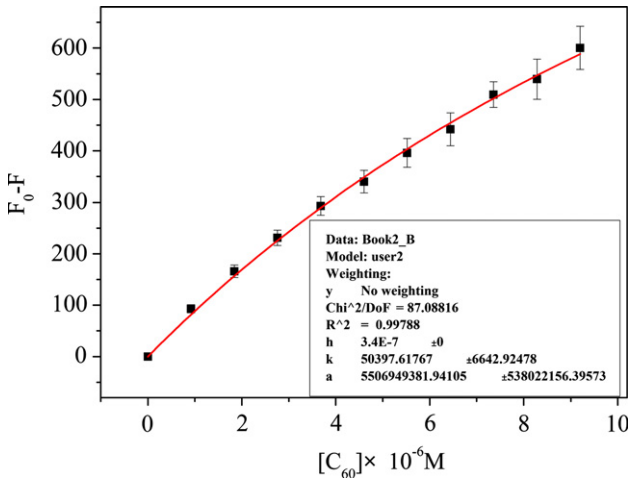


Fig. 4. Nonlinear curve fits for the complex of CTV–CZ and C_{60} in CH_2Cl_2.

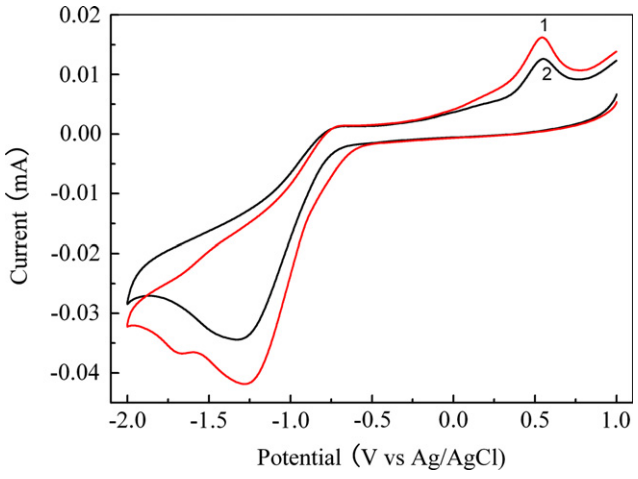


Fig. 5. Cyclic voltammograms of CTV–CZ (1) before and (2) after addition of C_{60} (5.5×10^{-6} M). The supporting electrolyte is 0.10 M Bu_4NClO_4 in acetonitrile and the scan rate is 0.10 V/s.

follows [35]:

$$\Delta F=\frac{1}{2}\left\{\alpha\left([H]_0+[G]_0+\frac{1}{K}\right)-\sqrt{\alpha^2\left([H]_0+[G]_0+\frac{1}{K}\right)^2-4[H]_0+[G]_0\alpha^2}\right\}$$

where $[H]_0$ and $[G]_0$ are the initial concentrations of host CTV–CZ and the guest C_{60}, respectively. ΔF denotes the change of the fluorescence intensity of CTV–CZ with the addition of C_{60}. α is a sensitive factor of the structure change of the CTV–CZ complex at the interactive course. As shown in Fig. 4, the binding constant (K) of CTV–CZ with C_{60} in CH_2Cl_2 was $5.0(6)\times10^4\ M^{-1}$ and the correlation coefficients R of the curve was 0.9989. The good result of nonlinear fitting indicated that the inclusion complex was formed with a stoichiometric ratio of 1:1 and the large binding constant attributed to better shape match and effective PET between CTV–CZ and C_{60}.

3.5. Cyclic voltammetry

Cyclic voltammetry is a very powerful electrochemical tool used in intermolecular donor–acceptor system. The electrochemical behavior of CTV–CZ and its fullerene complex were investigated by cyclic voltammetry (CV) in toluene/acetonitrile (5:1, v/v). During the CVs, 0.1 M of tetrabutylammonium hexafluorophosphate (Bu_4NPF_6) in acetonitrile was used as the supporting electrolyte, glassy carbon as a working electrode, *Pt* wire as a counter electrode, Ag/AgCl as the reference electrode, and the scan rate was 0.1 v/s, working electrode was polished between each measurement with 3 μm silica. From Fig. 5 we observed that CTV–CZ had oxidation peaks at 0.54 V, and reduction peaks at −1.33 V and −1.67 V, respectively. Upon addition of C_{60}, the redox current intensity decreased, and the peak potentials were slightly shifted. Fortunately the reduction potential at −1.67 V disappeared. The changes in the reduction potential gave the evidence of complex formation and meanwhile showed the interaction between CTV–CZ and C_{60} dependent on their conjugated π systems and the electron transfer from CTV–CZ to C_{60}.

3.6. ^1H NMR spectra studies

^1H NMR was used to characterize the formation of host–guest inclusion complex [36–38]. Fig. 6 exhibited that the ^1H NMR spectra of CTV–CZ in CS_2 with a $CDCl_3$ external lock (1:1, v/v) in the absence and presence of C_{60}. The ^1H chemical shifts ($\Delta\delta=\delta_{(complex)}-\delta_{(free)}$) of CTV–CZ were listed in Table 2. Addition of C_{60} induced the signals of H (1–10) to shift slightly. These shifts

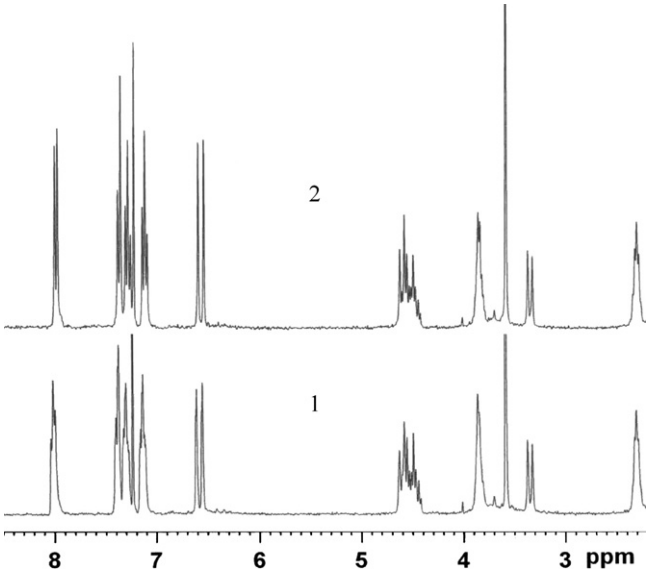


Fig.6. ^1H NMR spectra of CTV–CZ (1) before and (2) after forming inclusion complex.

Table 2
^1H chemical shifts of CTV–CZ in the presence and absence of C_{60}.

CTV–CZ proton	$\delta_{(free)}$	$\delta_{(complex)}$	$\Delta\delta$
H-1	6.549	6.543	−0.006
H-2	6.605	6.598	−0.007
H-3	7.131	7.125	−0.006
H-4	7.365	7.372	−0.007
H-5	7.372	7.365	−0.006
H-6	8.012	8.008	−0.004
H-7	3.579	3.576	−0.003
H-8,9	3.856	3.852	−0.004
H-10	2.302	2.299	−0.003

$\Delta\delta=\delta_{(complex)}-\delta_{(free)}$.

were small, but distinct and repeatable [39]. The shifts of protons in aromatic ring of CTV–CZ (H1–H2) and carbazole (H3–H6) (see the structure for numbering) were relatively obvious and could be explained by the fact that C_{60} was trapped in the hole of CTV–CZ and led to PET process between C_{60} and CTV–CZ occur. The slight

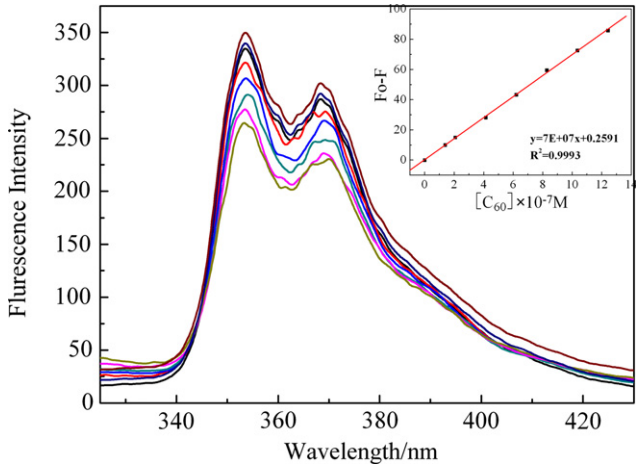


Fig. 7. The fluorescence spectra of CTV–CZ (7.2×10^{-8} M) and the solution of C_{60} in CH_2Cl_2 with the concentration range of 1.38×10^{-7}–1.24×10^{-6} M, Inset: the standard curve of C_{60} in CH_2Cl_2.

shift of H7–H10 might be from the change in the surrounding environment caused by mutual interaction between CTV–CZ and C_{60}.

3.7. Analytical merits of C_{60} based on CTV–CZ

The high binding affinity made CTV–CZ an effective fullerene receptor. In order to extend its analytical application, the calibration curve of C_{60} (λ_{em}=353 nm) in the presence of CTV–CZ was made by quenched fluorescence spectra in Fig. 7 with the linear dynamic range of 1.38×10^{-7}–1.24×10^{-6} M and a linear regression equation: $y=7E+07x+0.2591$ ($r^2=0.9993$) as an inset in Fig. 7. When added the known concentration of 6.21×10^{-7} C_{60}, the recovery of C_{60} was 99.0%, 102.2%, 96.1%, and the relative standard deviation was 2.1%. It revealed that potentially analytical application to determination of C_{60} was available.

4. Conclusions

In summary, CTV–CZ host molecule was synthesized and photophysical properties of the supramolecular complex between CTV–CZ and C_{60} were characterized in detail by UV–vis and fluorescence spectra. The results implied that the complex of CTV–CZ and C_{60} with 1:1 molar ratio was formed and ascribed to their size-match and PET interaction. ^{1}H NMR and cyclic voltammetry also further confirmed the interaction. CTV–CZ with inclusion interaction will be potentially served as a promising candidate for the purification and extraction of C_{60}.

Acknowledgements

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