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Photoinduced electron transfer between C_{60} and carbazole dimer compounds in a polar solvent

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Abstract

Photoinduced electron transfer processes between carbazole (Cz) dimer compounds and C_{60} in benzonitrile have been investigated by observing the transient absorption spectra in the visible and near-IR regions. By excitation of C_{60} with 532 nm-laser light, electron transfer occurred via the triplet excited state of C_{60} ($^{T}C_{60}^{*}$) from the Cz-dimer compounds, which was confirmed by observing characteristic absorption bands due to $C_{60}^{\bullet-}$ and the Cz-dimer radical cation. The Cz-dimer compounds, forming sandwich-like conformation of radical cation, showed the charge resonance (CR) bands in the near-IR region longer than 1200 nm. Rate constants for electron transfer seem to depend on the conformation of the Cz-dimer radical cation. The Cz-dimer compounds taking sandwich conformation donate the electron easily to $^{T}C_{60}^{*}$, because of the stabilization energies of the sandwich dimer radical cations of the Cz-dimer compounds. By excitation of the Cz-dimer compounds with 355 nm-laser light, the absorption bands due to the Cz-dimer radical cations appeared immediately after the laser irradiation and the absorption of $C_{60}^{\bullet-}$ is formed at diffusion controlled rate. This observation suggests that direct photoejection from the Cz-dimer compounds occurs in benzonitrile, yielding the Cz radical cations and solvated electron, in which the latter was trapped with C_{60}. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Fullerene; Carbazole dimer compounds; Electron transfer; Transient absorption spectra

1. Introduction

Fullerenes such as C_{60} and C_{70} have been known to enhance the photoinduced electric conductivity when they are doped into some organic conductors. For example, an enhancement of the photoconductivity of poly(*N*-vinylcarbazole) (PVCz) film by doping with fullerenes has been reported by Wang et al. [1,2]. Similar doping effects of fullerene on photoconductivity have also been reported on poly(*p*-phenylene vinylene) [3], poly(2,5-dialkoxy-*p*-phenylene vinylene) [4], and poly(3-alkylthiophene) [5]. These results show possibilities for the development of fullerene-based polymeric photoconductors. The photoconductivity is attributed to the roles of the fullerene to generate photocarrier in the polymeric system. These mechanisms have been investigated by photochemical techniques such as laser flash photolysis; for C_{60}-doped PVCz film, the charge-separated state ($C_{60}^{\bullet-}$ PVCz$^{\bullet+}$) is produced immediately after irradiation of a picosecond 532 nm-laser pulse, which excites mainly the charge-transfer (CT) absorption band [6].

In our previous paper, we reported that photoinduced electron transfer processes occur in the mixture of C_{60} and PVCz in polar solvent using nanosecond laser flash photolysis method [7,8]. The electron transfer mechanism depends on excitation wavelength; when C_{60} was excited selectively, electron transfer takes place via the triplet state of C_{60} ($^{T}C_{60}^{*}$). By the excitation of PVCz, direct photoejection occurs from PVCz yielding PVCz$^{\bullet+}$ and solvated electron, in which the latter was captured by C_{60}.

In the present paper, we report photoinduced electron transfer between C_{60} and the dimer model compounds of PVCz. We examine the following compounds (Scheme 1); di(*N*-carbazolyl)methane (DCzMe), 1,3-di(*N*-carbazolyl)-propane (DCzPr), 1,4-di(*N*-carbazolyl)butane (DCzBu), *trans*-1,2-di(*N*-carbazolyl)cyclobutane (DCzCBu), and *meso*-2,4-di(*N*-carbazolyl)-pentane (*m*-DCzPe). *N*-ethylcarbazole (EtCz) was used as a monomer model compound.

2. Experimental

C_{60} (>99.5%) was obtained from Term USA and used as received. EtCz was obtained from Tokyo Kasei Kogyo.

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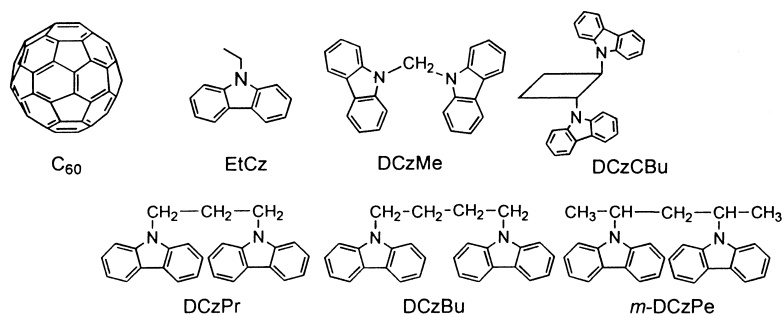
Scheme 1.

Carbazole (Cz) dimers were synthesized by the method described in the literature [9–11]. Benzonitrile (BN) was purchased from Kanto Kagaku and used without further purification.

Steady-state absorption spectra were recorded on a JASCO V-530 UV/VIS spectrophotometer. The sample solutions were contained in a 5 mm quartz cell.

Nanosecond transient absorption spectra were measured using SHG (532 nm) or THG (355 nm) of a Nd:YAG laser (Spectra-Physics, Quanta-Ray, GCR-130, FWHM 6 ns) as an excitation source. For measurements in the near-IR region, a Ge avalanche photodiode (Hamamatsu Photonics, B2834) was used as a detector for monitoring light from a pulsed Xe-lamp. For measuring sub-millisecond phenomena, an InGaAs-PIN photodiode (Hamamatsu Photonics, G5125-10) was used as the detector of a continuous Xe lamp. The sample solutions contained in a 10 mm quartz cell were deaerated with Ar bubbling before measurements at 298 K.

Oxidation potentials ($E(D^+/D)$) of the Cz compounds in BN were measured by cyclic voltammetry using a potentiostat (Hokuto Denko, HAB-151) with a conventional three-electrode-cell equipped with Pt working and counter electrodes and an Ag/AgCl reference electrode at scan rates of 100 mV s^{-1}. In each case, solution contains 2.0–5.0 mM of a sample with 0.10 M of tetrabutylammonium perchlorate (Nakarai Tesque) and deaerated with Ar bubbling before measurements.

3. Results and discussion

3.1. Steady-state absorption spectra

Fig. 1 shows steady-state absorption spectra of C_{60} (0.1 mM) and m-DCzPe (5.1 mM) in BN, which is typical experimental condition of the present laser flash photolysis study. For the other Cz compounds, the similar types of absorption spectra to m-DCzPe were observed. The absorption spectrum of the mixture of C_{60} and m-DCzPe in BN is essentially the same with a superposition of the components, suggesting that the CT interaction is negligible in ground state when the concentration of m-DCzPe is diluted

in the sample. Similarly, no CT interaction between other Cz compounds and C_{60} were observed.

C_{60} can be selectively excited by the 532 nm-laser light, because all Cz compounds used in the present study have no appreciable absorbance at 532 nm. On the other hand, the Cz-dimer compounds or EtCz are also excited with the 355 nm-laser light.

3.2. Excitation of C_{60} at 532 nm

Fig. 2(A) shows the transient absorption spectra obtained by 532 nm-laser light exposure on C_{60} in the presence of m-DCzPe in BN. A sharp absorption band at 740 nm appeared immediately after the laser exposure was attributed to an absorption band of triplet excited state of C_{60} ($^{T}C_{60}^{*}$) [12]. After the decay of $^{T}C_{60}^{*}$, new absorption bands appeared at 780, 1070, and 1200–1600 nm. Absorption band at 1070 nm was attributed to the radical anion of C_{60} ($C_{60}^{\bullet -}$) [13,14]. The absorption band at 780 nm observed at 4 μs was attributed to the $(m$-DCzPe$)^{\bullet +}$ [15–17]. Broad absorption band at 1200–1600 nm was assigned to the charge resonance (CR) band of $(m$-DCzPe$)^{\bullet +}$, which indicates the delocalization of the radical cation between two Cz units [18,19]. Both rise time-profiles of $C_{60}^{\bullet -}$ and CR band are mirror
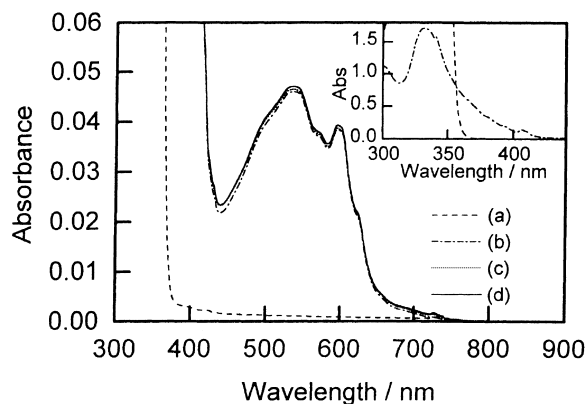

Fig. 1. Steady-state absorption spectra in the UV and visible regions in benzonitrile: (a) m-DCzPe (5.1 mM), (b) C_{60} (0.1 mM), (c) mixture of C_{60} (0.1 mM) and m-DCzPe (5.1 mM) in 5 mm cell, and (d) superposition of (a) and (b).
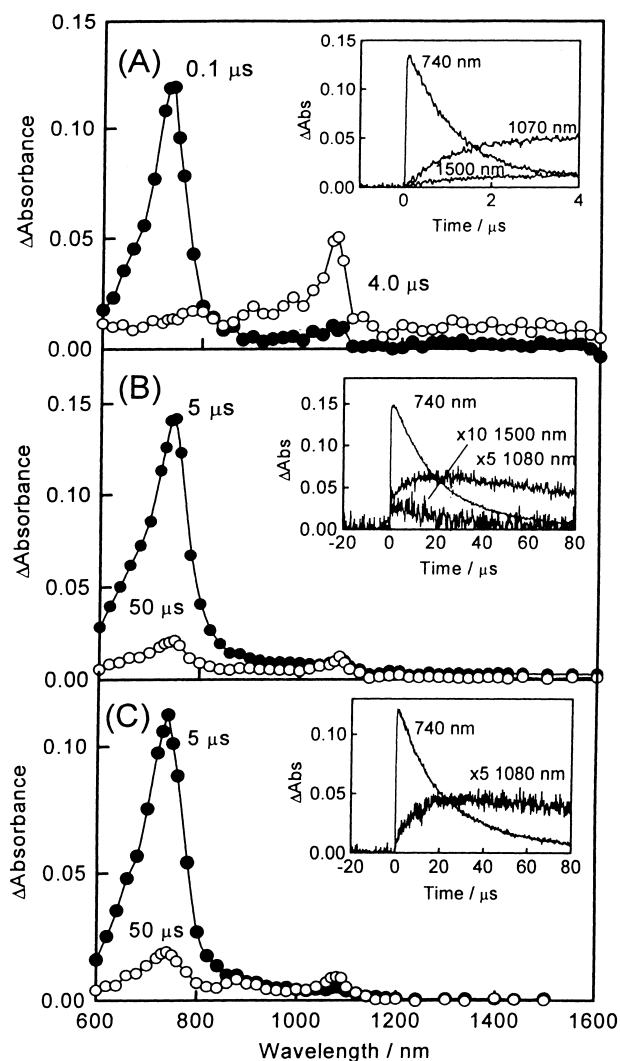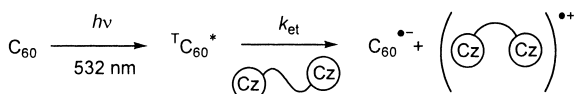
Fig. 2. Transient absorption spectra obtained by 532 nm-laser photolysis of C_{60} (0.1 mM) and (A) m-DCzPe (5.0 mM), (B) DCzCBu (30 mM), and (C) EtCz (30 mM) in benzonitrile. Inset: time profiles.

[20]. When $(m\text{-DCzPe})^{\bullet+}$ and $(\text{DCzPr})^{\bullet+}$ are produced, two Cz units take the sandwich-like conformation of the dimer radical cation, showing the CR band.

The transient absorption spectra of C_{60} in the presence of DCzCBu (Fig. 2(B)) also showed the generation of $C_{60}^{\bullet-}$ after decay of $^{T}C_{60}^{*}$; however, the absorption intensity of $C_{60}^{\bullet-}$ was weak in comparison with that of m-DCzPe by a factor of 1/5. The CR band was also very weak, indicating that two Cz units are difficult to overlap with each other in $(\text{DCzCBu})^{\bullet+}$, because of $trans$-position of two Cz units. The CR band reflects directly the degree of the overlap of Cz units; i.e. the lower degree of the overlap of Cz unit, the longer and weaker of the CR band [19]. The transient absorption spectra of C_{60} and DCzMe or DCzBu also show very weak CR bands, because Cz units are at 1,1- and 1,4-position, respectively.

Fig. 2(C) shows the transient absorption spectra of C_{60} in the presence of EtCz in BN. No absorption was observed in the near-IR region (1200–1600 nm). At this concentration of EtCz, intermolecular dimer radical cation is not formed after electron transfer [19].

In the presence of excess m-DCzPe with respect to concentration of $^{T}C_{60}^{*}$, the first-order decay rate of $^{T}C_{60}^{*}$ (k_{obs}) increased with concentration of m-DCzPe (Fig. 3). The absorption intensity at 740 nm did not decrease to zero within 4 μs because of spectral overlap with the absorption band due to $(m\text{-DCzPe})^{\bullet+}$ at 780 nm. The decay rate of $^{T}C_{60}^{*}$ was estimated by curve fitting assuming rise and decay functions (curve c in Fig. 3). In the case of DCzPr, the same method was used. On the other hand, in the case of DCzBu, DCzCBu, and DCzMe, the absorbance of the dimer radical cations in 700–800 nm region were very low; therefore, the decay curves of $^{T}C_{60}^{*}$ were fitted by one decay component. The pseudo first-order plot of k_{obs} versus [m-DCzPe] gives the second-order rate constant (k_{q}^{T}) as shown in inset
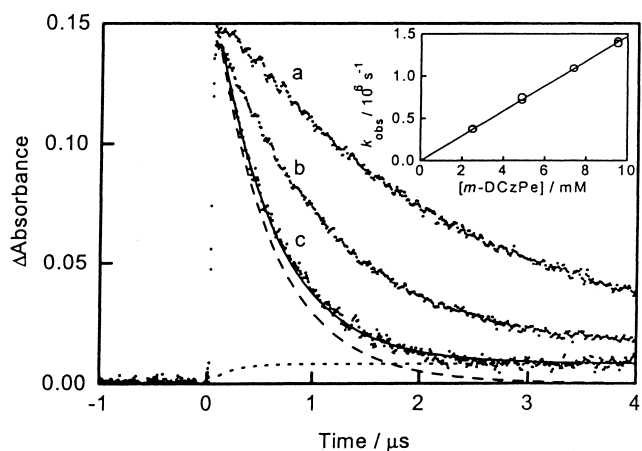
image with respect to the decay time-profile of $^{T}C_{60}^{*}$ (inset of Fig. 2(A)), indicating that both $(m\text{-DCzPe})^{\bullet+}$ and $C_{60}^{\bullet-}$ are produced via $^{T}C_{60}^{*}$. These findings show that electron transfer from m-DCzPe to $^{T}C_{60}^{*}$ generates $(m\text{-DCzPe})^{\bullet+}$ and $C_{60}^{\bullet-}$ as shown in Scheme 2.

In the case of mixture of DCzPr and C_{60}, the similar transient spectrum to the mixture of m-DCzPe and C_{60} was observed, indicating the same electron transfer process as m-DCzPe and the generation of the CR band of $(\text{DCzPr})^{\bullet+}$. In the ground state, the Cz-dimer model compounds take a conformation in which the two Cz units are farthest apart

Scheme 2.

Fig. 3. Decay time-profiles obtained by 532 nm-laser photolysis of $^{T}C_{60}^{*}$ at 740 nm with changing [m-DCzPe]: (a) 2.5, (b) 4.9, and (c) 9.5 mM and fitting curve (solid line) of (c) by assuming two components (rise and decay curve, dash and dot line, respectively). Inset: pseudo first-order plot.

Table 1
Oxidation potentials of the Cz compounds ($E(D^+/D)$), free-energy changes (ΔG_{et}) and rate constants (k_{et}) of photoinduced electron transfers from the Cz compounds to $^TC_{60}^*$, triplet quenching rate constants (k_q^T), and quantum yields (Φ_{et}^T) in benzonitrile

Cz [a]	$E(D^+/D)$ [b] (V vs. Ag/AgCl)	ΔG_{et} [c] (kcal mol^{-1})	k_q^T [d] (M^{-1} s^{-1})	Φ_{et}^T [e]	k_{et} [f] (M^{-1} s^{-1})
EtCz	+1.24	+0.23	3.8×10^6	0.45	1.7×10^6
DCzMe	+1.46	+5.2	$\sim 2 \times 10^5$ [g]	~ 0.1 [g]	$\sim 2 \times 10^4$ [g]
DCzPr	+1.22	−0.23	9.4×10^5	0.23	2.2×10^5
DCzBu	+1.23	+0.0	2.7×10^5	0.096	2.6×10^4
DCzCBu	+1.32	+2.1	3.1×10^5	0.082	2.5×10^4
m-DCzPe	+1.02	−4.8	1.8×10^8	0.54	8.1×10^7
PVCz	+1.04 [h]	−3.9	9.3×10^6 [i,j]	0.55 [i]	5.1×10^6 [i,j]

[a] Abbreviations are shown in Scheme 1.
[b] Values in benzonitrile.
[c] Evaluated by using Eq. (1).
[d] Experimental error is ±5%.
[e] Φ_{et}^T was evaluated from the plateau of the plot $[C_{60}^{\bullet-}]/[^TC_{60}^*]$ vs. [Cz].
[f] $k_{et} = \Phi_{et}^T k_q^T$.
[g] The values were estimated in the low concentration of DCzMe, because of poor solubility; hence, these value contain much estimation error.
[h] The oxidation potential of PVCz was evaluated to be +0.86 V vs. SCE in acetonitrile. This value was converted to the value vs. Ag/AgCl in BN.
[i] From [8].
[j] Calculated as monomer unit.

of Fig. 3. The values of k_q^T for the Cz-dimer compounds and EtCz are listed in Table 1.

An efficiency for the formation of $C_{60}^{\bullet-}$ via $^TC_{60}^*$ can be evaluated by the quantity of $[C_{60}^{\bullet-}]/[^TC_{60}^*]$ on substituting the observed absorption intensities and reported molar absorption coefficients for $^TC_{60}^*$ (16 100 M^{-1} cm^{-1} at 740 nm) and $C_{60}^{\bullet-}$ (12 000 M^{-1} cm^{-1} at 1070 nm) [21,22]. On plotting $[C_{60}^{\bullet-}]/[^TC_{60}^*]$ against the concentrations of the Cz-dimer compounds, $[C_{60}^{\bullet-}]/[^TC_{60}^*]$ increases and reaches a plateau, yielding a quantum yield (Φ_{et}^T) for electron transfer via $^TC_{60}^*$ [23,24]. Rate constants for electron transfer (k_{et}) can be evaluated by a relation $k_{et} = \Phi_{et}^T k_q^T$ [23,24]. The values of Φ_{et}^T and k_{et} for the Cz-dimer compounds and EtCz in BN are summarized in Table 1.

According to Rehm–Weller relation, free-energy change of the electron transfer (ΔG_{et}) can be calculated from Eq. (1) [25]

$$\Delta G_{et} = 23.06 \left[E\left(\frac{D^+}{D}\right) - E\left(\frac{A^-}{A}\right) - \Delta E_{0,0} - \frac{e^2}{a\varepsilon} \right] \quad (1)$$

where $E(D^+/D)$ is the oxidation potential of a donor (the Cz compound), $E(A^-/A)$ the reduction potential of acceptor (−0.33 V versus Ag/AgCl in BN for C_{60}) [26], $\Delta E_{0,0}$ the lowest energy of $^TC_{60}^*$ (1.53 eV) [27], and $e^2/a\varepsilon$ is a Coulomb term. The oxidation potentials and ΔG_{et} are summarized in Table 1. In Fig. 4, the evaluated k_{et} values of the Cz compounds and some amines [28] in BN are plotted against the ΔG_{et} values. The plot is in good agreement with a curve calculated from semi-empirical Rehm–Weller equation [25].

The variation for k_{et} between C_{60} and the Cz-dimer compounds is considered to depend on the conformation of the Cz-dimer radical cations. $(m\text{-DCzPe})^{\bullet+}$ and $(\text{DCzPr})^{\bullet+}$ can take a sandwich-like conformation, in which two Cz units

overlap completely; on the other hand, DCzBu, DCzCBu, and DCzMe seem to form partially overlapping or open conformation [19,20]. The Cz-dimer radical cations with sandwich conformation are more stable than that with partially overlapping or open one [19,20]; hence, m-DCzPe and DCzPr donate the electron to $^TC_{60}^*$ easily. As for the EtCz, which does not form the sandwich dimer radical cations even in high concentration, the electron transfer rate is faster than some Cz-dimer compounds having no sandwich dimer radical cations. This reason is presumably due to less steric hindrance of EtCz than the Cz-dimer compounds on the approach of $^TC_{60}^*$.

After reaching maximum concentration, the absorption intensity of $C_{60}^{\bullet-}$ began to decay as shown in Fig. 5. The decay time-profile obeys second-order kinetics, which suggests that $C_{60}^{\bullet-}$ and $(m\text{-DCzPe})^{\bullet+}$ recombine by the back electron transfer. From the slopes of the second-order plots, the ratios of k_{bet}/ε_A (k_{bet} and ε_A refer to the rate constant for
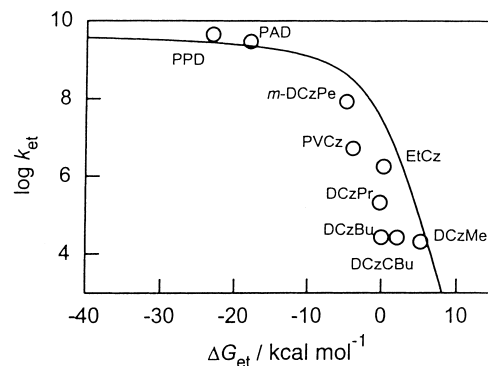

Fig. 4. Plots of log k_{et} vs. ΔG_{et} for the Cz-dimer compounds, EtCz, PVCz, and some amines (PPD, *p*-phenylenediamine; PAD, *p*-anisidine) in benzonitrile. The solid line was calculated from semi-empirical Rehm–Weller equation [25].
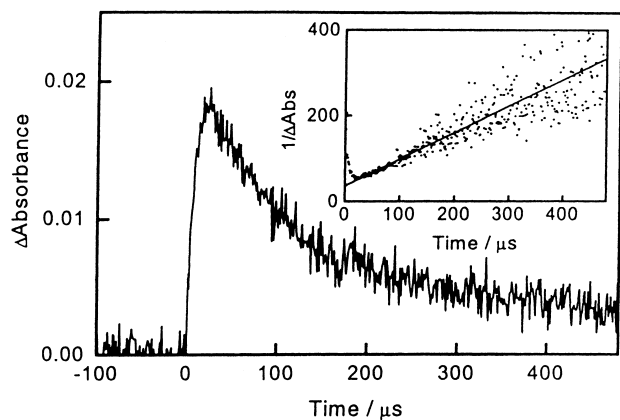
Fig. 5. Decay time-profile of $C_{60}^{\bullet-}$ at 1070 nm with m-DCzPe in benzonitrile. Inset: second-order plot.

back electron transfer and the molar absorption coefficient of $C_{60}^{\bullet-}$, respectively) were evaluated as listed in Table 2. Upon substitution of ε_A, one can obtained the k_{bet} values which are also listed in Table 2. Free-energy changes for the back electron transfer reactions (ΔG_{bet}) [29,30] range from -41.3 to -31.1 kcal mol^{-1}. The sufficiently negative ΔG_{bet} suggests that the back electron transfer processes are exothermic and close to the diffusion controlled rate ($k_{diff}=5.6\times10^9$ M^{-1} s^{-1} in BN) [31]. Tsujii et al. [20] reported that stabilization energies were estimated to be 7 and 9 kcal mol^{-1} for the sandwich dimer radical cations of m-DCzPe and DCzPr, respectively. After taking these values into consideration, the values of ΔG_{bet} are still sufficiently negative, which support k_{bet} close to k_{diff}.

3.3. Excitation at 355 nm

The transient absorption spectra observed by the laser photolysis of the Cz-compounds in the presence of C_{60} in BN with 355 nm-light are shown in Fig. 6. In the case of Cz-dimer compounds, the CR band appeared immediately
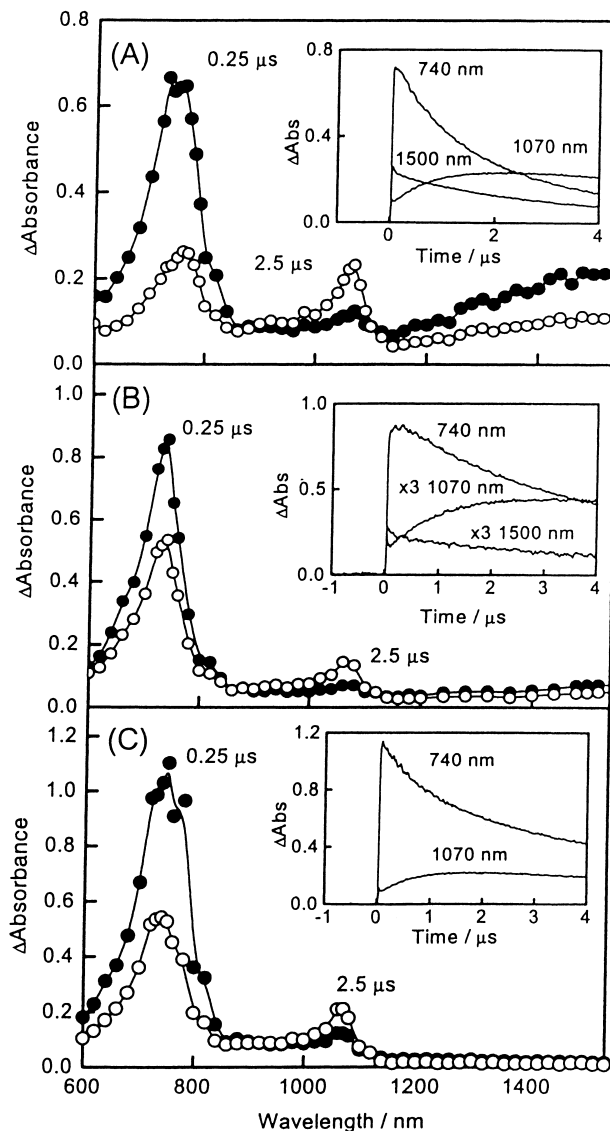

Fig. 6. Transient absorption spectra obtained by 355 nm-laser photolysis of (A) m-DCzPe (3.0 mM), (B) DCzCBu (3.0 mM), and (C) EtCz (3.0 mM) in the presence of C_{60} (0.1 mM) in benzonitrile. Inset: time profiles.

after the laser irradiation (Fig. 6(A) and (B)). Comparison with decay of $^TC_{60}^*$, rise of $C_{60}^{\bullet-}$ was rather faster. In the case of DCzCBu, for example, decay rate constant of $^TC_{60}^*$ and rise rate constant of $C_{60}^{\bullet-}$ were 1.5×10^5 and 9.4×10^5 s^{-1}, respectively. This indicates that the electron transfer occurred not only via $^TC_{60}^*$ but also by another reaction. This electron transfer process was also observed between C_{60} and the other Cz compounds.

The generation of the Cz-dimer radical cations can be attributed to the photoejection from the excited state of the Cz compounds, because the Cz-dimer compounds showed the absorption bands upon 355 nm-excitation without C_{60}. Fig. 7 shows that photoejection occurs from the Cz compounds, yielding the radical cations and solvated electron in BN. The lifetime of the radical cations are relatively long

Table 2
Free-energy changes (ΔG_{bet}) and rate constants (k_{bet}) of back electron transfers from $C_{60}^{\bullet-}$ to $Cz^{\bullet+}$ calculated from the slope of second-order plots (k_{bet}/ε_A) in benzonitrile

Cz [a]	ΔG_{bet} [b] (kcal mol^{-1})	k_{bet}/ε_A [c] (cm s^{-1})	k_{bet} (M^{-1} s^{-1})
EtCz	-36	4.7×10^5	5.6×10^9
DCzMe	-41	7.2×10^5	8.6×10^9
DCzPr	-35	6.6×10^5	7.9×10^9
DCzBu	-36	5.7×10^5	6.8×10^9
DCzCBu	-38	1.1×10^6	1.3×10^{10}
m-DCzPe	-31	7.4×10^5	8.8×10^9
PVCz	-32	2.7×10^5 [d]	4.4×10^9 [d]

[a] Abbreviations are shown in Scheme 1.

[b] Evaluated by using $23.06[E(A^-/A)-E(D^+/D)]$ [29,30].

[c] ε_A: Molar absorption coefficient for $C_{60}^{\bullet-}$ (12 000 M^{-1} cm^{-1} at 1070 nm) [21,22].
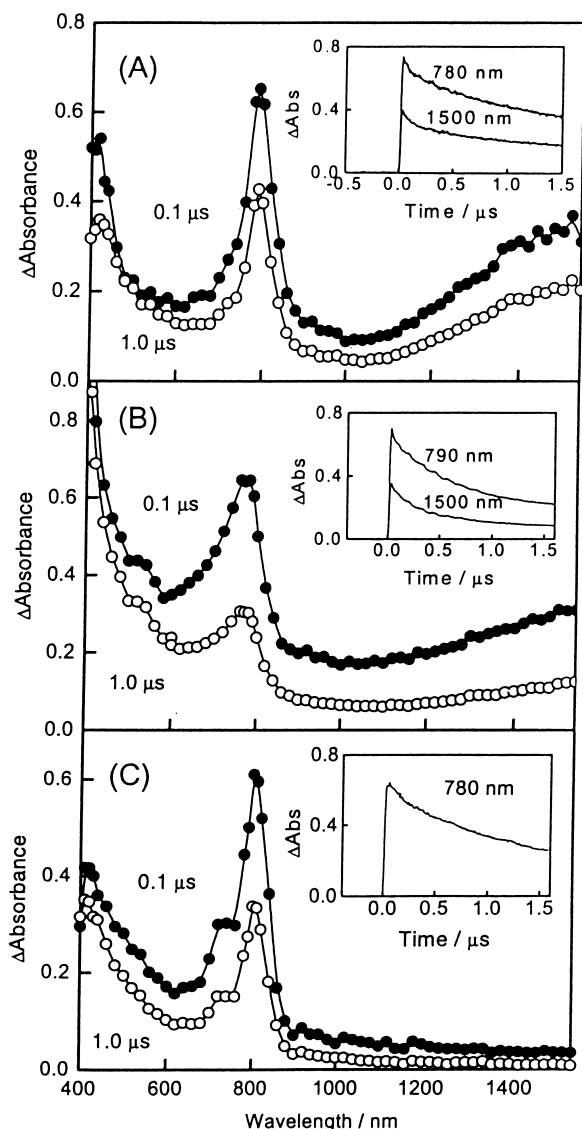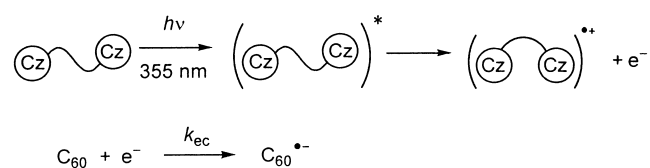
[d] From [8].

Fig. 7. Transient absorption spectra obtained by 355 nm-laser photolysis of (A) *m*-DCzPe (3.0 mM), (B) DCzCBu (3.0 mM), and (C) EtCz (3.0 mM) in benzonitrile. Inset: time profiles.
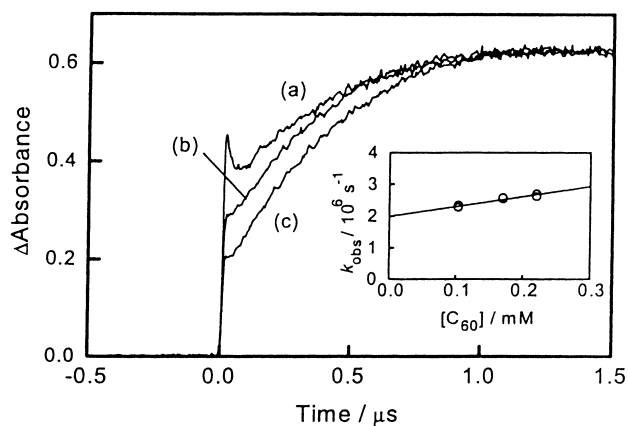

Fig. 8. Time profiles of $C_{60}^{\bullet-}$ at 1070 nm observed by 355 nm-laser photolysis of *m*-DCzPe (5.0 mM) with changing $[C_{60}]$; (a) 1.0, (b) 1.7, and (c) 9.5 mM in benzonitrile. The profiles are normalized. Inset: pseudo first-order plot.

Table 3
Rate constants (k_{ec}) for electron capture by C_{60} in benzonitrile

Cz [a]	k_{ec} ($M^{-1} s^{-1}$)
EtCz	6.9×10^9
DCzMe	4.5×10^9
DCzPr	4.6×10^9
DCzBu	3.5×10^9
m-DCzPe	3.1×10^9
PVCz	8.3×10^9 [b]

[a] Abbreviations are shown in Scheme 1.
[b] From [8].

(inset of Fig. 7), indicating that the solvated electron exists long time without recombination with the Cz radical cations. Therefore, the lifetime of the solvated electron is sufficiently long to react with C_{60} (Scheme 3).

The generation rate of $C_{60}^{\bullet-}$ was accelerated with the concentration of C_{60} in the presence of *m*-DCzPe as shown in Fig. 8. Furthermore, a linear relationship was observed between the generation rate (k_{obs}) and the concentration of C_{60}, indicating the pseudo first-order reaction between the ground state of C_{60} and the solvated electron. From the slope of inset of Fig. 8, the reaction rate for electron capture by C_{60} (k_{ec}) was estimated to be $3.1 \times 10^9 M^{-1} s^{-1}$. The values of k_{ec} for the other Cz compounds are evaluated as listed in Table 3. The k_{ec} values are almost same for all Cz-dimer compounds and closed to k_{diff} in BN. These findings are reasonable for the electron capture reaction between the solvated electron in BN and C_{60}.

4. Conclusions

Photoinduced electron transfer mechanism between the Cz-dimer compounds and C_{60} in BN depends on excitation wavelength. By excitation of C_{60} at 532 nm, the electron transfer reactions occurred via $^{T}C_{60}^{*}$, generating $C_{60}^{\bullet-}$ and the Cz-dimer radical cations. The Cz-dimer compounds donate the electron to $^{T}C_{60}^{*}$ easily, when they form the sandwich-like conformation of the Cz-dimer radical cations, because of their stabilized energy.

By excitation at 355 nm, the Cz-dimer radical cations were produced immediately after the laser irradiation and $C_{60}^{\bullet-}$ was formed rather slowly, which suggests that direct

Scheme 3.

photoejection occurs from the Cz unit, yielding the solvated electron, which was captured by C_{60}. The rate constants for electron capture are almost same for the Cz-dimer compounds, indicating the absence of steric effect in this process.

Acknowledgements

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