Magnetic field effects on photoelectrochemical reactions of modified electrodes with C60-phenothiazine nanoclusters

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Abstract

Magnetic field effects on the photoelectrochemical reactions of photosensitive electrodes modified with C\textsubscript{60} nanoclusters were examined, intended for the development of nanodevices where photofunctions were controllable with magnetic field. Mixed clusters of a C\textsubscript{60} derivative (C\textsubscript{60}N\textsuperscript{+}) containing a positive charge and methylphenothiazine (MePH) were prepared by dissolving them in the THF–H\textsubscript{2}O mixed solvent. Diameters of the clusters were estimated to be ca. 100 nm from dynamic light scattering and AFM measurements. Photofunctional electrodes with the nanoclusters of C\textsubscript{60}N\textsuperscript{+} and MePH were fabricated on the electrodes modified with a self-assembled monolayer of HS(CH\textsubscript{2})\textsubscript{2}SO\textsubscript{3}\textsuperscript{+}/Na\textsuperscript{+} on a gold substrate. The photocurrent action spectrum of the cluster-modified electrode strongly indicates that the photocurrents are ascribed to the photoexcitation of the nanoclusters of C\textsubscript{60}N\textsuperscript{+}. Furthermore, the photocurrents increased with increasing an external magnetic field, typically 3\% at 0.5 T. The profile of magnetic field dependence on the photocurrents is in good agreement with the results on the dynamics of radical pair in the nanoclusters of C\textsubscript{60}N\textsuperscript{+}. This suggests that the magnetic field effects are ascribable to the contribution of triplet radical pair that are generated by photoinduced intermolecular electron-transfer from MePH to triplet excited state of nanocluster of C\textsubscript{60}N\textsuperscript{+}, and are explained in terms of spin-lattice relaxation mechanism in radical pair mechanism.

Keywords: C\textsubscript{60}; Magnetic field effects; Radical pair; Nanocluster; Photoelectrochemical reaction

1. Introduction

Recently, photochemical and photoelectrochemical properties of fullerene (C\textsubscript{60}) have been widely investigated [1]. Photoinduced electron-transfer reactions of donor–C\textsubscript{60} linked molecules have also been reported [1–7]. In a series of donor–C\textsubscript{60} linked systems, some of the compounds show novel properties, which accelerate photoinduced charge separation and decelerate charge recombination [3–5]. Those properties have been based on the remarkably small reorganization energy in their electron-transfer reactions. The porphyrin–C\textsubscript{60} linked compounds, where the porphyrin moieties act as both donors and sensitizers, have been extensively studied [3–5].

It has also been reported that C\textsubscript{60} and its derivatives formed optically transparent microscopic clusters in some mixed solvents [8,9]. The clustering behavior of C\textsubscript{60} is associated with the strong hydrophobic interactions among the C\textsubscript{60} units. Photoinduced electron-transfer and photoelectrochemical reactions using the C\textsubscript{60} clusters have been reported because of their interesting properties [8,9].

Magnetic field effects (MFEs) on the reaction kinetics or yields of photochemical reactions in the condensed phase have been studied [10–13]. The MFEs have been powerful for verifying the mechanism of photochemical reactions including triplet states. Previously, we obtained photogenerated triplet biradicals of donor–acceptor linked compounds, and found that the lifetimes of the biradicals were remarkably extended in the presence of magnetic fields up to 1 T [14–22].
As to the MFEs in donor–C₆₀ linked systems, C₆₀ shows interesting behaviors. First, C₆₀ has no magnetic nuclei based on hydrogen, and thus there are little hyperfine interactions due to the C₆₀ radical contributing to the MFEs. Second, C₆₀ is a highly symmetric molecule and therefore anisotropic Zeeman interaction due to the C₆₀ radical, is very small. Third, the g-value (g = 1.9982) [23] of C₆₀ is smaller than that of the radical cation of electron donor such as phenothiazine cation radical (g = 2.0052) [24]. Therefore, the difference in g-values between the donor and C₆₀ is expected to be large. On the basis of the three points, the MFEs in various donor–C₆₀ linked compounds are expected to provide useful information for studying the spin chemistry of C₆₀.

From these considerations, we have examined photoinduced electron-transfer reactions and MFEs on the photogenerated biradicals in phenothiazine(Ph)–C₆₀ [16–20] and zinc-tetraphenylporphyrin (ZnP)–C₆₀ [21,22] linked compounds and found novel MFEs on the photo-generated biradicals in benzonitrile.

Recently, we examined photoinduced electron-transfer reactions and MFEs on the dynamics of a radical pair that was generated from the intermolecular electron-transfer reaction between a C₆₀ cluster and methyl phenothiazine (MePH) [25]. Meanwhile, a fullerene derivative containing a positive charge (a monopyrrolidinium salt) (C₆₀N⁺) forms optically transparent clusters in the THF/H₂O mixed solvent. Also, transient absorption spectra of the cluster of C₆₀N⁺ and MePH system following laser excitation at 355 nm indicate that the photoinduced intermolecular electron-transfer from the triplet excited state of phenothiazine (PH) to C₆₀N⁺ cluster (C₆₀N⁺)ₙ occurs and the yield of the escaped phenothiazine cation radical (PH⁺) increases with increase of magnetic field. These MFEs are explained by spin-lattice relaxation mechanism in radical pair mechanism and most likely ascribed to the nanoclusters of C₆₀N⁺.

MFEs on the photoelectrochemical reactions of photosensitive electrodes are very important for practical applications of the MFEs to control the photoelectronic functions of molecular devices. Previously, we have examined MFEs on the photoelectrochemical reactions of photosensitive electrodes modified with zinc-tetraphenylporphyrin-viologen linked compounds [26,27] and semiconductor nanoparticles [28,29]. However, MFEs on the photoelectrochemical reactions of photosensitive electrodes modified with nanoclusters have not yet been reported.

In the present paper, we have examined MFEs on the photoelectrochemical reactions of photosensitive electrodes modified with nanoclusters containing of C₆₀ and phenothiazine, intended for utilization of C₆₀ as photosensitive electrodes.

2. Experimental

Synthetic procedures of a C₆₀ derivative containing a positive charge (C₆₀N⁺) were prepared according to the previous paper [25]. The structure of C₆₀N⁺ was confirmed by ¹H-NMR and MALDI-TOF MS spectra and elemental analysis [25]. ¹H-NMR spectra were measured on a JEOL model JMN-GSX 400. Mass spectra were measured by a MALDI-TOF-MS instrument (PE Biosystems Voyager DE). Steady-state absorption spectra were recorded on a Shimadzu UV-2500PC or UV-3150 spectrometer. Methylphenothiazine (MePH) (Wako, extra pure) and THF (Dojindo, spectrosol) were used as received (Scheme 1. Structural formula).

Clusters of C₆₀N⁺ were prepared by dissolving C₆₀N⁺ in the THF–H₂O mixed solvent. C₆₀N⁺ forms optically transparent clusters by the following procedure [25]. First, the suspension of C₆₀N⁺ and MePH were sonicated in THF for 15 min. Next, H₂O was injected into the suspension with a syringe. After sonication of the sample for 15 min, the suspension became optically transparent in the mixed solvent (THF/H₂O = 2:1) solution.

A gold planar electrode was fabricated by vacuum deposition of titanium followed by gold on a glass plate at 573 K. Self-assembled monolayers (SAMs) of HS(CH₂)₂SO₃Na⁺ (MPS) were prepared by immersing the gold electrode in an ethanol solution of MPS (0.1 mM). Modified electrodes with the clusters of C₆₀N⁺ alone and of the mixture of C₆₀N⁺ and MePH were fabricated by immersing the MPS-modified electrode in the THF–H₂O mixed solution containing the nanoclusters of C₆₀N⁺ alone or C₆₀N⁺ and MePH.

Morphology of the nanoclusters of C₆₀N⁺ alone or the mixture of C₆₀N⁺ and MePH on the modified electrodes or freshly cleaved mica was measured with atomic force microscopy (AFM: a Nanoscope IIIa (Digital Instrument)). After drying at ambient temperature, the AFM images of the nanoclusters were recorded in tapping mode.

Electrochemical and photoelectrochemical measurements were carried out by using a three electrode cell containing of the modified electrode as a working electrode, a platinum electrode as a counter electrodes, and a Ag/AgCl electrode as a reference electrode. Na₂SO₄ (0.1 M) was used as a. supporting electrolyte. Cyclic voltammetry (CV) was carried out on a BAS CV-50W instrument using the three-electrode cell. The photoelectrochemical measurements were carried out by irradiation with a 300 W Xe lamp through a monochromator or a
500 W high-pressure Hg arc lamp through a visible-pass filter (>400 nm). Photocurrents from the modified electrode were measured under a controlled potential at 0 V vs. Ag/AgCl with a potentiostat in the presence of triethanolamine (TEOA) (50 mM) as a sacrificial electron donor under nitrogen atmosphere. The MFEs on the photoelectrochemical measurements were carried out according to the previous paper [26–29]. The electrode cell was placed at pole-gap of an electromagnet. Electric field was applied to the magnetic field in previous papers [28,29].

3. Results and discussion

3.1. Preparation and characteristics of the modified electrodes with nanoclusters of C₆₀N⁺ alone or C₆₀N⁺ and MePH

Absorption spectrum of the mixture of C₆₀N⁺ (0.1 mM) and MePH (1 mM) in the THF:H₂O (2:1) mixed solvent is shown in Fig. 1. The absorption in the 400–800 nm regions is featureless. The similar spectrum was obtained in the case of C₆₀N⁺ (0.1 mM) alone in the same solvent [25]. The results suggest the formation of mixed clusters of C₆₀N⁺ and MePH in the THF:H₂O (2:1) mixed solvent.

In previous paper, AFM images were measured to investigate the formation of the clusters of C₆₀N⁺ alone or the mixture of C₆₀N⁺ and MePH. The AFM images on mica suggested that C₆₀N⁺ alone or the mixture of C₆₀N⁺ and MePH formed the clusters with diameter of ca. 100 nm in the THF–H₂O mixed solution. This value is similar to those estimated from dynamic light scattering (DLS) measurements. On the basis of the absorption, AFM, and DLS measurements, the formations of nanoclusters of C₆₀N⁺ alone or the mixture of C₆₀N⁺ and MePH were confirmed in the THF–H₂O (2:1) mixed solution [25].

Parts (a) and (b) of Fig. 2 shows AFM images of gold electrode and the modified electrode with the nanoclusters of C₆₀N⁺ alone on the gold electrode. The AFM images clearly indicated the modified electrodes with the nanoclusters (diameter of ca. 50 nm) of C₆₀N⁺ were fabricated by the above procedure in the experimental section. The AFM image of the modified electrode with the nanoclusters of C₆₀N⁺ and MePH on a gold substrate was similar to that in Fig. 2(b). These results indicated that the modified electrodes with the nanoclusters of C₆₀N⁺ alone or C₆₀N⁺ and MePH were fabricated by immersing the MPS-modified electrode in the THF–H₂O mixed solution containing the nanoclusters. The diameters in Fig. 2(b) were smaller than those in the previous paper [25]. The result may be ascribed to the experimental conditions including temperature.

Cyclic voltammogram (CV) of the modified electrode with the nanoclusters of C₆₀N⁺ and MePH is shown in Fig. 3. The oxidation peak (0.75 V vs. Ag/AgCl) ascribing to the one oxidation of phenothiazine moiety, due to PH⁺/PH, was clearly observed. On the other hand, the two broad reduction peaks (−0.40 and −0.80 V vs. Ag/AgCl) are attributable to the first (C₆₀⁰/C₆₀⁻) and the second (C₆₀⁻/C₆₀⁻²) reductions of the nanocluster of C₆₀N⁺. In the CVs of the modified electrodes with the nanoclusters of C₆₀N⁺ alone, the reduction peak (−0.40 vs. Ag/AgCl) ascribing to the first (C₆₀/C₆₀⁻) reduction of the nanocluster of C₆₀N⁺ was clearly observed. However, the second (C₆₀⁻/C₆₀⁻²) reduction of the nanocluster of C₆₀N⁺ could not be observed apparently. This difference may be ascribed to the morphological difference between the nanoclusters of C₆₀N⁺ alone and the mixture of C₆₀N⁺ and MePH.
Kamat et al. reported that the C60 cluster film electrochemically deposited on the ITO electrode were electrochemically active and exhibited two cyclic voltammetric reduction peaks (–0.63 and –0.98 V vs. Ag/AgCl) [30]. Similar results were obtained in the present modified electrodes as shown in Fig. 3. The slight difference in the above two cases may be ascribed to the difference in the size of C60 clusters. On the basis of these results, it is clear that C60N+ and/or MePH can act as redox species in the modified electrodes with the nanoclusters of C60N+ alone and/or the mixture of C60N+ and MePH.

### 3.2. Photoelectrochemical reactions of the modified electrodes with nanoclusters of C60N+ alone or the mixture of C60N+ and MePH

Photoirradiation of the modified electrode with nanoclusters of C60N+ alone or the mixture of C60N+ and MePH afforded anodic photocurrents. Fig. 4 shows the photocurrent action spectrum of the modified electrode with the nanoclusters of C60N+ and MePH. In the case of the modified electrode with nanoclusters of C60N+ alone, the similar action spectrum as that of Fig. 4 was observed. The photocurrent action spectrum was in fair agreement with the absorption spectrum (Fig. 1) of the THF–H2O (2:1) mixed solution containing the nanoclusters of the mixture of C60N+ and MePH or C60N+ alone. These results strongly indicate that the photocurrents are ascribed to photoexcitation of the nanoclusters of C60N+. The observations in the photoelectrochemical reactions in the modified electrode with the nanoclusters of the mixture of C60N+ and MePH are similar to those in the C60–donor cluster films as reported previously [30]. It is suggested that the photocurrent mechanism in the present modified electrode is similar to that of the previous paper [30]. However, it is not clear at the stage to verify whether the photocurrent is generated only from the photoreaction of C60N+ or from the photoinduced intermolecular electron-transfer reaction from MePH to the photoexcited state of C60N+ nanoclusters.

### 3.3. MFEs on photocurrents of the modified electrode with nanoclusters of the mixture of C60N+ and MePH

MFEs on the photocurrents of the modified electrode with nanoclusters of the mixture of C60N+ and MePH were examined to verify the photocurrent generation mechanism. In the presence of magnetic field, the photocurrents clearly increased.

The magnitude of MFEs on the photocurrent is expressed as follows:

\[
Q = \left( \frac{I(H) - I(0)}{I(0)} \right) \times 100, \tag{1}
\]

where \(I(H)\) and \(I(0)\) are the photocurrent in the presence and absence of magnetic fields, respectively. The \(Q\)-value (%) increased gradually with increasing magnetic field up to 0.5 T (ca. 3% at 0.5 T), as shown in Fig. 5.

Previously, we investigated the photoinduced electron-transfer reactions and MFEs in the cluster of C60N+–MePH by 355 nm laser excitation. The MFEs, that the yield of the escaped PH+ increased with increase...
of magnetic field, were clearly observed. The MFEs also indicated that the triplet radical pair was generated from photoinduced intermolecular electron-transfer from triplet excited state of PH to C_{60}N^{+} cluster [25].

Though the photoexcited species of C_{60}N^{+} cluster, (C_{60}N^{+})_n^+ in the present study is different from that of MePH in the previous study [25], the formation of triplet radical pair occurred in both cases. Therefore, the present MFEs observed in the photocurrents (Fig. 5) are expected to be explained by the same mechanism of the MFEs as reported in the previous paper [25]. In fact, the magnetic field dependence on the yield of the escaped PH^+ was in good agreement with that on the Q-value in Fig. 5. Accordingly, the MFEs on the photocurrents can be explained by spin-lattice relaxation mechanism in radical pair mechanism.

From the above discussions, the MFEs in the modified electrode with the nanoclusters of the mixture of C_{60}N^{+} and MePH can be explained by the radical pair mechanism between (C_{60}N^{+})_n^+ and MePH as shown in Scheme 1. First, (C_{60}N^{+})_n^+ is selectively photoexcited by the visible light, because MePH has no absorption bands in the visible region, generating the singlet excited state of (C_{60}N^{+})_n^-, {1((C_{60}N^{+})_n^-)^*}. Then, the intersystem-crossing process (k_{isc1}) occurs and the triplet excited state of (C_{60}N^{+})_n^-, {3((C_{60}N^{+})_n^-)^*} is generated. The intermolecular electron-transfer process (k_{ET(T)}) from MePH to {3((C_{60}N^{+})_n^-)^*} occurs, to give a triplet radical pair, {1((C_{60}N^{+})_n^- + MePH^+)^*}. This triplet radical pair is converted the initial ground state via a singlet radical pair, {1((C_{60}N^{+})_n^- + MePH^+)^*} (k_{isc2} and k_{CR}). The intersystem-crossing process (k_{isc2}) for the radical pair is rate-determining step for the decay of radical pair via reverse electron-transfer reaction, but the reverse electron-transfer process (k_{CR}) from the singlet radical pair to the ground state does not. In Scheme 2, the intersystem-crossing process (k_{isc2}) is influenced by magnetic field. In the absence of magnetic field, the three sublevels of triplet radical pair are degenerate. In the presence of magnetic field, Zeeman splitting of triplet sublevels occurs, and therefore the intersystem crossing process (k_{isc2}) is retarded by the spin-lattice relaxation from triplet sublevels. Therefore, the MFEs are explained in terms of spin-lattice relaxation mechanism [10–13]. As a result, the escape process (k_{esc}) from the triplet radical pair increased with increasing magnetic field, giving larger numbers of the escaped (C_{60}N^{+})_n^-, and/or the intermolecular hole hoping among the MePH moieties in the mixed nanoclusters. Therefore, the photocurrents in the modified electrode with the nanoclusters of the mixture of C_{60}N^{+} and MePH increased in the presence of magnetic field. In other words, the MFEs on the photocurrents also offer another experimental evidence of that the photoelectrochemical response of the modified electrode are ascribed to the photoinduced intermolecular electron-transfer reaction from MePH to {3((C_{60}N^{+})_n^-)^*} in the mixed clusters.

In the case of modified electrodes with the Langmuir Blodgett film of zinc-tetraphenylporphyrin-viologen linked compounds (ZnP(n)V(n = 4, 6, 8)), the photocurrent and the Q-value (%) increased with the increase of the magnetic field in lower magnetic fields (<0.3 T) and became constant in higher magnetic fields (>0.3 T) (ca. 15% at 0.5 T in the case of ZnP(8)V-modified electrode) [26, 27]. However, the magnetic field dependence for the modified electrode with the nanoclusters of the mixture of C_{60}N^{+} and MePH is different (Fig. 5). For example, the magnitude of the MFEs in ZnP(n)V-modified electrodes is larger than that in the case of the nanoclusters of the mixture of C_{60}N^{+} and MePH. In the transient absorption spectra of ZnP(n)V, the decay rate constants of the photogenerated radical pairs decreased steeply (<0.2 T) and became constant (>0.2 T) with increasing magnetic field [31]. In the transient absorption spectra of C_{60}N^{+}–MePH-nanocluster, the yield of the escaped radical of MePH increased with increasing magnetic field [25]. Therefore, the difference of the magnetic field dependence between ZnP(n)V-modified electrodes and C_{60}N^{+}–MePH-nanocluster modified electrode is responsible for the mechanism of the MFEs as similar to that of the MFEs in transient absorption spectra in solution as described above.

4. Conclusion

A fullerene derivative containing a positive charge (a monopyrrolidinium salt) (C_{60}N^{+}) or the mixture of C_{60}N^{+} and MePH formed optically transparent clusters (diameter ca. 100 nm) in the THF/H_2O mixed solvent, as were verified from absorption, AFM and DLS measurements. Modified electrodes with the nanoclusters of C_{60}N^{+} alone or the mixture of C_{60}N^{+} and MePH were fabricated on the SAM of MPS. Photoirradiation of the both modified
electrodes afforded stable anodic photocurrents, ascribing to the photoexcitation of nanoclusters of C_{60}N^{+}. The photocurrents in the modified electrodes with the nanoclusters of the mixture of C_{60}N^{+} and MePH increased with increasing magnetic field. The MFEs are ascribed to the contribution of triplet radical pair that is generated by photoinduced intermolecular electron-transfer from MePH to triplet excited state of the C_{60}N^{+} nanocluster. From the comparison of the MFEs between those in the photoelectrochemical and photoinduced electron-transfer reactions, it is suggested that the present MFEs can be explained by spin-lattice relaxation mechanism in radical pair mechanism.

The present study provides useful information for designing novel nanodevices whose photofunctions can be controlled by magnetic field. Further investigations on the modified electrodes with nanocluster of the mixture of C_{60}N^{+} and other donor (such as porphyrin) molecule, as well as the nanocluster of donor–C_{60} linked compounds, and the quantitative analysis of MFEs on the photocurrent generation are now in progress.

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