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Novel aspects in the photochemical redox reactions of C_{60} using triethylamine donor

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Abstract

In the photochemical reduction of C_{60} with triethylamine donor monitored by near-IR spectroscopy, the primary mono-anion formed, $C_{60}^{-\bullet}$, converts to $C_{60}H^-$ species, which, after stopping the irradiation, recovers back to $C_{60}^{-\bullet}$. The $C_{60}H^-$ formation probably proceeds via a further photoexcitation of $C_{60}^{-\bullet}$, its intermediate di-anion formation coupled with a rapid protonization by the intermediately formed triethylamine cation. EPR experiments closely analogous to the near-IR investigations also confirmed the primary formation of $C_{60}^{-\bullet}$, its disappearance during prolonged irradiation as well as its reappearance after stopping irradiation. EPR spectra additionally reveal a further intermediate related to C_{60} mono- and di-anion, probably C_{60} mono-anion associate.

Keywords: C_{60} fullerene; Electron transfer; Anion radicals; Proton transfer; Triethylamine

1. Introduction

Fullerene C_{60} has frequently been reduced using a variety of methods such as cathodic [1–11], photochemical [12–21], or alkali metal reduction [22–24], and others as well [24–31]. Its mono-, di-, tri- and tetra-anions were well described by means of their characteristic near-IR (NIR) bands [6,9,15,17–19,22,23,26]. Thus, in the cathodic reduction four reversible one-electron steps were observed [1–6]. Less information is available on photochemical fullerene reduction processes [12–21]. The primary stages in the photoreduction of C_{60} with various electron donors using nanosecond pulses and rapid NIR detection on the nanosecond time scale were described by a dominant NIR band at 1070 nm, which is characteristic for C_{60} mono-anion [12–19]. We also systematically investigated the photoreduction of C_{60} and its derivatives under continuous UV/visible irradiation using Et_3N and TiO_2 donors, and verified the formation of $C_{60}^{-\bullet}$ with its NIR spectrum observed on the minute time scale [32,33]. In addition to NIR, we applied Electron Paramagnetic Resonance (EPR) spectroscopy, which indicated more species than were initially observed by NIR investigations [32–35]. A broadened EPR line with a peak-to-peak (pp) larger than 3 mT and a *g*-value around 2.0000 is generally observed, and was assigned to $C_{60}^{-\bullet}$ [1–6]. Narrow lines, frequently superimposed on the broadened one, remain a topic of discussion and controversy [31]. We investigated them in detail. In polar solvents, in primary stages of an in situ reduction, a narrow line **A** with $pp_A = 0.09$ mT and $g_A = 2.0000$ appeared during continuous irradiation, which was replaced then with line **B** having $pp_B = 0.042$ mT and $g_B = 2.0006$. This also vanished under prolonged irradiation. Radical **A** was assigned to C_{60} fullerene mono-anion, and **B**, tentatively, to di-anion or to some associated species of $C_{60}^{-\bullet}$ [32–35]. However, after stopping irradiation, the EPR lines **B** and **A** reappeared [35]. This unusual behaviour led us at this point to expand our EPR study into an analogous in situ NIR study, with a rapid NIR spectrometer.

2. Experimental details

Fullerene C_{60}, gold quality, originated from Hoechst (Germany). Solvents, i.e., toluene (for HPLC), acetonitrile (puriss., over molecular sieves) were purchased from Fluka. Triethylamine was obtained from Aldrich. Generally a solution of 0.1 mM C_{60}, 0.1 M Et_3N in 1:1 toluene:acetonitrile solvents were prepared under an argon or nitrogen atmos-

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phere. EPR experiments were carried out in a flat cell employing a Bruker 200 D Spectrometer on line with an Aspect 2000 computer. Under continuous irradiation with a medium pressure mercury lamp, the EPR spectra were recorded in time intervals of 40 s. NIR experiments were carried out with the same solution as that used in the EPR experiments, in a 1 × 1 cm four-window Suprasil cell, using a rapid scanning X/DAP NIR/XLW NIR spectrometer from Polytec (Germany). The spectra were taken at 5 s intervals, and the probe was continuously irradiated with a high-pressure mercury lamp perpendicularly to the beam of the NIR spectrometer. All measurements were carried out at 290 K.

3. Results and discussion

An illustration of EPR measurements carried out during an irradiation of C_{60} solutions and after irradiation stop is shown in Fig. 1. Immediately after starting the irradiation, EPR line **A** of $C_{60}{}^{-\bullet}$ is evident, then in the next scans, also line **B** is seen. Their intensities quickly pass a maximum and convert to zero after 15 min of irradiation. Upon stopping the irradiation, both lines **A** and **B** reappear with time. It should be mentioned that the narrow lines **A** and **B** are always observed in the polar solvents, in the initial stages of an in situ reduction using various techniques of generation [10,11,32–35]. **B** was identified as a consecutive product of **A**. However, their ratios and time evolution strongly depend on the substitution of C_{60} fullerene used, on the type of donor applied, as well as on its concentration, and further, on the intensity of irradiation, the method of generation, and other parameters as well [32–36]. Thus, given higher donor concentrations and more intensive irradiation, both the passing of the point of maximal concentration and the conversion of **A** to **B** are speeded up [32,33,35]. The EPR and NIR experiments presented here were (so far as possible) carried out under closely analogous conditions. Certainly, they cannot replace a combined in situ on line EPR/NIR experiment but they may be at least qualitatively comparable.

Near-IR spectra obtained in an experiment analogous to the EPR investigations are shown in Fig. 2. As already the beam of the spectrometer itself caused the generation of C_{60} mono-anion in the initial stage, the balance of the reference line is slightly distorted, and later some spectra indicate a negative absorbance (Fig. 4), when $C_{60}{}^{-\bullet}$ concentration converts to zero. The spectra were taken in 5 s intervals. During the first irradiation interval an enormous increase of $C_{60}{}^{-\bullet}$ concentration is evident ($\lambda = 1075$ nm) [6,23] which then decreases to zero after 80 s of continuous irradiation. Stopping the irradiation, the C_{60} mono-anion band recovers, increasing over time. In addition, in the first irradiation period of 5 s, a further intensive band at 996 nm, not characteristic for C_{60} mono-anion (nor for C_{60} di-anion [6,23]) is seen. However, the side bands of the standard C_{60} mono-anion spectrum are superimposed on it. More details are presented in the two-dimensional projection (Figs. 3 and 4).

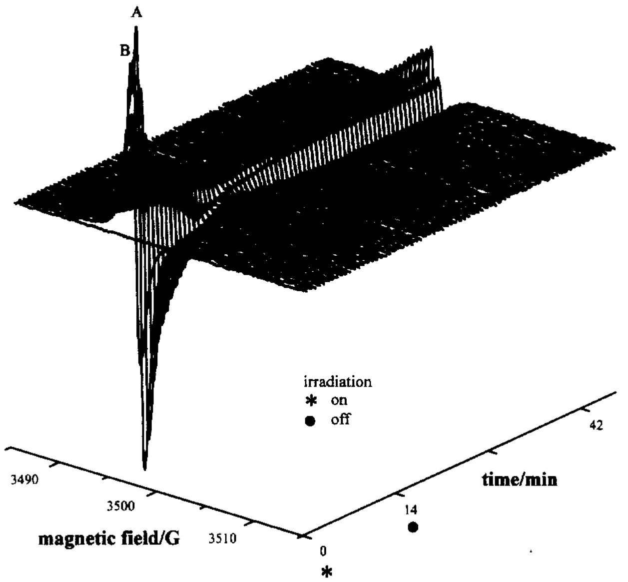


Fig. 1. The time evolution of EPR spectra observed during a continuous irradiation (*) and after stopping irradiation (●) of a 0.1 mM C_{60}, 0.1 M Et_3N in 1:1-toluene:acetonitrile solution.

Fig. 3 shows the evolution of NIR spectra during irradiation. Obviously from the inset in Fig. 3, during the first irradiation period of 5 s, in addition to the C_{60} mono-anion band at 1075 nm, the band at 996 nm also rises rapidly. Whereas the C_{60} mono-anion band then vanishes relatively quickly with increasing irradiation time, the band at 996 nm increases during the same interval. This is not so clearly seen from the spectra presented, as in the slightly decreasing integral absorbance at 996 nm, the rapid decrease of C_{60} mono-anion's side band is integrated with a dominating ratio. Taking this into account, we evaluated the individual contribution of this new band (subtracting $C_{60}{}^{-\bullet}$ absorbance from the integral absorbance) and an absorbance increase of the new band results as quoted in the inset of Fig. 3.

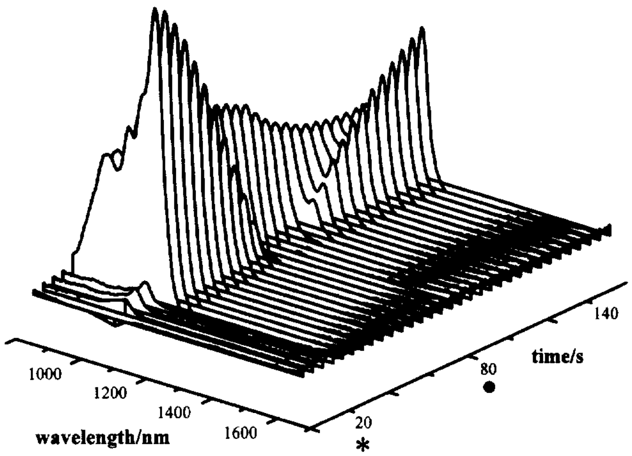


Fig. 2. The time evolution of NIR spectra during continuous irradiation (*) and after stopping irradiation (●) of a 0.1 mM C_{60}, 0.1 M Et_3N in 1:1-toluene:acetonitrile solution. The absorbance axis is quoted in Figs. 3 and 4.

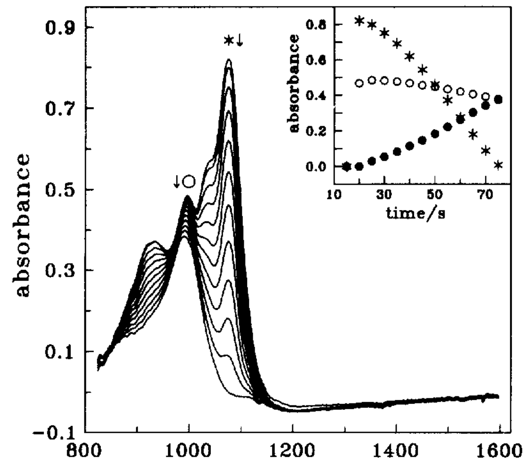


Fig. 3. NIR spectra from Fig. 2 observed during the irradiation period with the evaluation of absorbance changes (inset) for $C_{60}^{-\bullet}$ (*) and for $C_{60}H^-$ (●) after correction of the integral absorbance at 996 nm (○).

Fig. 4 shows the time behaviour after stopping irradiation. The C_{60} mono-anion band recovers again, and the evaluated individual absorbance of the band at 996 nm decreases. Detailed analysis from the other experiments reveals that this conversion passes an isosbestic point at 1024 nm. Consequently C_{60} mono-anion is regenerated from a product characterized by the band at 996 nm.

The near-IR and EPR experiments were carried out with 0.1 mM C_{60}, 0.1 M Et_3N in 1:1 toluene:acetonitrile under closely analogous conditions. The concentration changes of radical **A** observed in EPR investigations correlate qualitatively very well with the time behaviour found for the characteristic band of $C_{60}^{-\bullet}$ at 1075 nm (Figs. 1 and 2). This may be considered as strong supporting evidence for our previous assignment of line **A** to C_{60} mono-anion [32,33]. Respecting line **B**, the frequently assumed alternative explanation is that it originates from C_{60} di-anion. This view was also strongly supported by our previous findings that during electrochemical reduction, line **B** is only observed if the reduction potential is raised to the second reduction wave [10]. However, carrying out the NIR experiments under conditions analogous to those in which EPR line **B** was observed, the characteristic band of C_{60} di-anion at 950 nm [6,23] is missing in the NIR spectra presented. We assume that C_{60}^{2-} is probably still intermediately formed according to a mechanism discussed below. But it is rapidly oxidized with the unreduced fullerene, forming thereby an associate $[C_{60}\cdots C_{60}]^{2-}$ characterized by EPR line **B** [35]. Its NIR spectrum may be closely similar to that of $C_{60}^{-\bullet}$, so no new significant bands were observable. However, a combined in situ, one line NIR/EPR experiment definitely establishing the relation between the EPR line **B** and the NIR band of C_{60} di-anion at 950 nm is, due to some technical problems, so far not available.

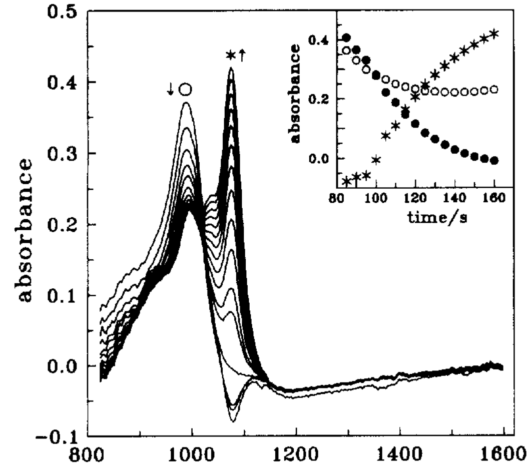


Fig. 4. NIR spectra from Fig. 2 after stopping irradiation with the evaluation of the absorbance changes for $C_{60}^{-\bullet}$ (*) and for $C_{60}H^-$ (●) after correction of the integral absorbance at 996 nm (○).

The NIR band at 996 nm observed in the photochemical reduction is closely similar to that described in Refs. [37,38]. These authors investigated the behaviour of electrochemically reduced C_{60} under the addition of acids [37,38]. Whereas $C_{60}^{-\bullet}$ showed itself to be a relatively weak base and was relatively stable upon the addition of weak acids, the C_{60} di-anion showed strong basic properties, and was easily protonated to $C_{60}H^-$ according to Eq. (1) [38,39]:

$$C_{60}^{2-}+H^+\rightarrow C_{60}H^- \qquad (1)$$

$$C_{60}H^-\rightarrow C_{60}^{-\bullet}+1/2H_2 \qquad (2)$$

The formation of $C_{60}H^-$ was coupled with the disappearance of the characteristic di-anion band at 950 nm and with the appearance of a new band at 990 nm [38]. Then, over time, the new band at 990 nm also vanished, and the characteristic band of $C_{60}^{-\bullet}$ at 1078 nm appeared [38]. This was explained by the decomposition of $C_{60}H^-$ to $C_{60}^{-\bullet}$ according to Eq. (2).

A phenomenon rather similar to that described above in the protonization of C_{60}^{2-} was also found in the photochemical reduction of C_{60} with triethylamine donor presented. As shown in Fig. 2, the band analogous to $C_{60}H^-$ was observed here at 996 nm and its absorbance increased with a longer period of irradiation. Then, after stopping irradiation, its absorbance decreased. Simultaneously with the decrease of that band, the absorbance of $C_{60}^{-\bullet}$ rose again. This conversion is characterized by an isosbestic point analogous to that reported for the de-protonization experiments ($\lambda_{iso}=1029$ nm [38]), and it strongly suggests that in the photochemical reduction we are also dealing with the formation of $C_{60}H^-$ and then with its subsequent decomposition according to Eq. (2). However there is a basic difference between the protonization reported in Refs. [37–39] and our photochemical experiments. Whereas the protonization in Ref. [38] was started from the electrochemically generated di-anion C_{60}^{2-} and proceeded according to Eq. (1), in our photoreduction we did not observe the reduction of C_{60} to its di-anion with the characteristic NIR band at 950 nm [6,23]. Therefore, a possible alternative explanation for $C_{60}H^-$ formation may be discussed.

The formation of $C_{60}^{-\bullet}$ in the presence of an electron donor may proceed in various routes. Three of them, evident so far in different experiments [12–19,40–48] are summarized in a simplified form in Scheme 1.

A straight-forward alternative would be the intermediate formation of a ground state charge transfer complex $[C_{60}^{\delta-}\cdots{}^{+\delta}NEt_3]$ and its separation to $C_{60}^{-\bullet}$. The formation of $C_{60}^{-\bullet}$ in such systems was confirmed at very high donor concentrations [40,42]. The second alternative represents the primary photoactivation of fullerene ($^*C_{60}$) which is then quenched with NEt_3 donor to an ion pair $[C_{60}^{-\bullet}\cdots{}^{+\bullet}NEt_3]$ and subsequently converts to $C_{60}^{-\bullet}$, especially in polar solvents [17–19]. The third route represents the primary formation of ground state charge transfer complex, and its photoactivation, followed then by the formation of ion pair complex $[C_{60}^{-\bullet}\cdots{}^{+\bullet}NEt_3]$ and separation to $C_{60}^{-\bullet}$. The quenching of the primary photoactivated fullerene excited singlet state ($^1C_{60}$), and by intersystem crossing formed a triplet state ($^3C_{60}$) may represent further widening of the route described in Scheme 1.

Comparing UV/visible spectra of separate C_{60} and NEt_3 solutions with their mixed solution ($C_{60}+NEt_3$) at the concentrations in the system used here, we found clear evidence for charge transfer formation, similar to that already described in Refs. [40,44–48]. This documents the possibility for the first and third suggested reaction route involving charge transfer intermediates. The second suggested route was confirmed in our previous experiments [32], where at the lower donor concentrations, $^*C_{60}$ in its triplet state was evident by means of EPR, converting to $C_{60}^{-\bullet}$ upon prolonged irradiation. This implies that all three suggested routes are probable but it is difficult to decide which of them may be dominating under given experimental conditions.

A further step, i.e., the observed formation of $C_{60}H^-$ is less plausible and more complex. According to our NIR data obtained, we can characterize it with an integral Eq. (3), where during the irradiation of $C_{60}^{-\bullet}$ in the presence of Et_3N donor, species $C_{60}H^-$ is formed.

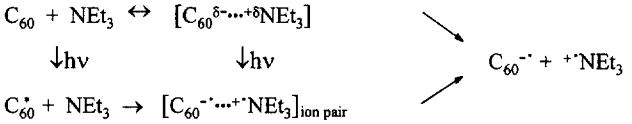
$$C_{60}^{-\bullet} \xrightarrow{h\nu,Et_3N} C_{60}H^- \qquad (3)$$

$$C_{60}^{-\bullet}+h\nu\rightarrow(C_{60}^{-\bullet})^* \qquad (3a)$$

$$(C_{60}^{-\bullet})^*+Et_3N\rightarrow[C_{60}^{2-}+{}^{+\bullet}NEt_3] \qquad (3b)$$

$$[C_{60}^{2-}+{}^{+\bullet}NEt_3]\rightarrow C_{60}H^-+Et_2NC^{\bullet}HCH_3 \qquad (3c)$$

There may be various alternative explanations for the formation of $C_{60}H^-$. The most plausible one, analogous to the above-described C_{60}^{2-} protonization, requires the reduction of $C_{60}^{-\bullet}$ to its di-anion and the presence of a proton donor (Eq. (1)). Neither requirement is evident in the investigated photochemical systems, since the reduction potential of C_{60} to C_{60}^{2-} is relatively high for proceeding in a simple electron transfer from triethylamine donor to C_{60} [15]. A further step requires proton donor, which is also for the most part not evident in such system, though Et_3N, after its oxidation, may fulfil this function. Our experiments are carried out under intensive irradiation using relatively high Et_3N concentrations. Consequently, a photoexcitation of the pre-formed $C_{60}^{-\bullet}$ to $(C_{60}^{-\bullet})^*$ (eventually in its complexed form) may be assumed (Eq. (3a)) although an exact excited state $(C_{60}^{-\bullet})^*$ is difficult to specify. The excited $(C_{60}^{-\bullet})^*$ may immediately abstract one electron from Et_3N and so intermediately form di-anion C_{60}^{2-} and $^{+\bullet}NEt_3$ (Eq. (3b)). Such an intermediate would represent a coupling of a strong base C_{60}^{2-} and a strong acid $^{+\bullet}NEt_3$. In this connection, a more hypothetical account of the path of their formation would involve immediate coupling with the protonization of C_{60}^{2-}, giving the observed product $C_{60}H^-$. The expected protonization step following electron transfer from triethylamine donor is well known from the literature [49–53]. Such a mechanism may be eventually verified in the future by means of time-resolved studies in advanced stages of C_{60} reduction with amino donors.



Scheme 1. Formation of $C_{60}^{-\bullet}$ via electron transfer from NEt_3.

Recently published paper reporting on the photochemical C_{60} reduction with nanoseconds resolution presents a NIR $C_{60}^{-\bullet}$ spectrum with a relatively high contribution to the absorbance at 950 nm. But this band is more characteristic for C_{60}^{2-} than $C_{60}^{-\bullet}$ at room temperature [6,8,23]. Possibly, this may indicate already a partial conversion of $C_{60}^{-\bullet}$ to C_{60}^{2-} in such photochemical reactions.

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