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Micellar effects on photoinduced redox reactions of $Fe(bpy)_2(CN)_2$

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Abstract

Excitation of solutions of $Fe(bipy)_2(CN)_2$ by a 266-nm laser pulse produces a hydrated electron and the oxidized complex, $Fe(bipy)_2(CN)_2{}^+$, in the primary photochemical step, in homogeneous aqueous solution as well as in aqueous solutions containing cetyltrimethylammonium bromide (CTAB) or sodium dodecyl sulfate (SDS) micelles. In all cases nascent hydrated electrons react with ground state $Fe(bipy)_2(CN)_2$ to form $Fe(bipy)_2(CN)_2{}^-$, and comparison of the decay constants in the three media (H_2O: $k = 2.8 \times 10^{10}\ M^{-1}\ s^{-1}$; CTAB: $k = 2.9 \times 10^{10}\ M^{-1}\ s^{-1}$; SDS: $k = 5.5 \times 10^9\ M^{-1}\ s^{-1}$), shows that the reaction is essentially unaffected by CTAB micelles but is much slower in SDS solution. Similar micellar effects were found for the back reaction between $e_{aq}{}^-$ and $Fe(bpy)_2(CN)_2{}^+$. Rate constants for the scavenging of the photogenerated hydrated electrons by methyl viologen (MV^{2+}) cations and $NO_3{}^-$ anions were measured in the three systems, and the results indicate that for scavenging by MV^{2+} the rate constants are decreased in the micelle systems (k in H_2O, 8.4×10^{10}; CTAB, 3.5×10^{10} and SDS, $1.58 \times 10^{10}\ M^{-1}\ s^{-1}$), whereas for $NO_3{}^-$ the CTAB micelle decreases while the SDS micelle enhances the scavenging compared to water solution (k in H_2O, 8.3×10^9; CTAB, 7×10^8; and SDS, $2.05 \times 10^{10}\ M^{-1}\ s^{-1}$). For the comproportionation reaction between $Fe(bipy)_2(CN)_2{}^+$ and $Fe(bipy)_2(CN)_2{}^-$ both micelles reduce the rate (k in H_2O, 3.3×10^{10}; CTAB, 2.3×10^{10}; and SDS, $1.05 \times 10^{10}\ M^{-1}s^{-1}$), but while the reaction of $Fe(bipy)_2(CN)_2{}^+$ with MV^+ is increased in CTAB compared to water, it is slowed in SDS (k in H_2O, 2.4×10^{10}; CTAB, 8.9×10^{10}; and SDS, $1.8 \times 10^{10}\ M^{-1}s^{-1}$). All effects observed in these microheterogeneous systems can be uniformly interpreted in terms of Coulombic interactions between the actual reactants and the charged surface of the micelles. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: $Fe(bpy)_2$ $(CN)_2$; Redox reactions; Micellar effects

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1. Introduction

In the past 15 years there has been a number of studies of the photoredox properties of some mixed ligand cyano complexes $[M(L)_n(CN)_{6-2n}]^{2n-4}$($M = Fe$, Ru; $L = 2,2'$-bipyridine (bpy), 1,10′- phenanthroline (phen) and their derivatives) [1–4]. These complexes have been especially interesting because of the possibility of altering the energy levels of the metal-to-ligand charge transfer (MLCT) and the ligand field (LF) states by replacement of ligands, resulting in some cases in a reversal of ordering of these states [4–6]. A related remarkable feature of these complexes is that their visible spectra are very solvent dependent because of donor-acceptor interactions between the lone pairs of the cyano ligands and solvents behaving as acceptors [7–10]. For $Fe(bpy)_2(CN)_2$ the lowest excited state is an LF state, whereas for $Ru(bpy)_2(CN)_2$ the lowest excited state was observed to be an MLCT state [3]. Photochemical studies showed that biphotonic excitation of the latter complex at 355 nm (corresponding to the MLCT state) produces hydrated electrons [11], and photoionization of the corresponding iron(II) complex occurred by both continuous irradiation and flash photolysis, although at a much higher excitation energy [12–15]. Since the $M(bpy)_2(CN)_2$ complexes are neutral, they can be easily accommodated in the hydrophobic core of micelles in aqueous solutions, and, while a rather detailed study by Atherton dealt with the photoionization of the ruthenium complex in homogeneous aqueous and aqueous sodium dodecyl sulfate solutions [11], no attempt has been made by flash photolysis to elucidate the reactions that occur following photoinduced ionization of $Fe(bpy)_2(CN)_2$ in micellar systems. In this paper, we show, using laser flash photolysis experiments, how cationic and anionic micelles can affect such reactions in aqueous solution.

2. Experimental

$Fe(bpy)_2(CN)_2{\cdot}3H_2O$ was synthesized according to a published procedure [7]. The purity of the complex was

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checked by spectrophotometry and thermogravimetry. Sodium dodecyl sulfate (SDS), cetyltrimethylammonium bromide (CTAB), methyl viologen dichloride hydrate and potassium nitrate (Aldrich, reagent grade) were used as supplied. Stock solutions of the surfactants were always freshly prepared on the day of use. The final concentrations of SDS and CTAB in the solutions to be irradiated were 0.030 M and 0.022 M, and, since the critical micelle concentrations are 8.1 mM and 0.9 mM, and aggregation numbers are 62 and 61 [16], respectively, the actual concentrations of micelles were about the same in both systems. Solutions of the mixed reagents were prepared directly in 1 cm cuvettes, using micropipettes to deliver aliquots of stock solutions. The cuvettes were then bubble-degassed with pure argon for one hour and used immediately.

Transient species were generated and detected using the Nd-YAG laser system described previously [17]. For signals that were obvious monoexponentional decays, lifetimes were determined by linear regression analysis of the natural logarithm of absorbance, using QuattroPro 3.0 and MS Excel 5.0.

For measurement of time-resolved spectra, a large amount of solution to be irradiated was drawn through a flow-through cuvette so that fresh sample was flashed at each wavelength increment. Absorbances of the transient signals were extracted and stored at predetermined time delays. The path of the laser beam was at right angles with respect to the analyzing beam.

Quantum yields of hydrated electron production were obtained in a given sample by comparing the electron absorbance signal at 700 nm, extrapolated to $t = 0$, to that of a reference sodium hexacyanoferrate(II) solution flashed under identical conditions of laser power, wavelength, cuvette, geometric configuration of laser and analyzing beam, and optical density (OD) at the laser wavelength. A value of 0.52 was used as a reference quantum yield for production of hydrated electrons by hexacyanoferrate(II) at 266 nm [18].

3. Results and discussion

3.1. Formation and decay of hydrated electron

Excitation of the three types of solution of $Fe(bpy)_2(CN)_2$ by a 266 nm laser pulse leads in all cases to prompt formation of hydrated electron resulting in a strong absorption at 700 nm that decays by a simple first-order law, as shown in Fig. 1 for the homogeneous system. There is little effect on the electron decay rate in CTAB solutions, but in the presence of the anionic surfactant SDS, the decay of the hydrated electron is significantly depressed. Since, in all three cases, the decay rate is proportional to the concentration of $Fe(bpy)_2(CN)_2$, as shown in Fig. 2, one route of decay of the electron is through reaction with this ground-state complex. From the slopes of the plots in Fig. 2, the bimolecular rate constants are $2.8 \pm 0.2 \times 10^{10}$, $2.9 \pm 0.2 \times 10^{10}$, and $5.5 \pm 0.5 \times 10^9\ M^{-1}\ s^{-1}$ for homogeneous aqueous solution, CTAB, and SDS solutions, respectively. The rate constant obtained in homogeneous solution is in good agreement with the value determined earlier by pulse radiolysis [15] and indicates that the reaction between hydrated electron and the ground-state complex is diffusion-controlled in this system. In the presence of dodecyl sulfate, the ejected electrons are repulsed by the negatively charged surface of the micelles containing the neutral complexes in their hydrophobic core, thus hindering the scavenging reaction. Cationic micelles, on the other hand, do not significantly influence the apparent rate constant, a result that may be explained by two compensating effects: while there may be an accelerating effect from the attraction between the positively charged surface of the micelles and hydrated electrons, the potential barrier at the interface should cause a retardation.

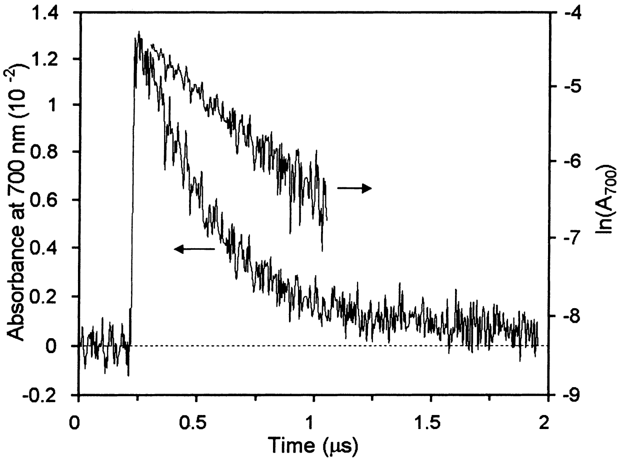

Fig. 1. Decay of hydrated electron at 700 nm in homogeneous aqueous (5×10^{-5} M $Fe(bpy)_2(CN)_2$).

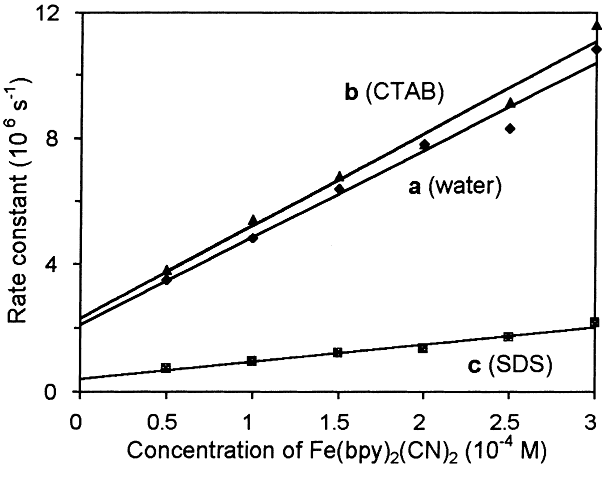

Fig. 2. Pseudo first-order decay rate constant of hydrated electron vs. $Fe(bpy)_2(CN)_2$ concentration in (a) homognenous aqueous solution, (b) 0.022 M CTAB, and (c) 0.030 M SDS.

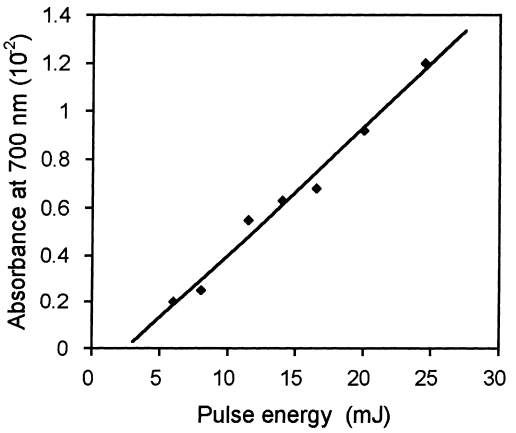

Fig. 3. Absorbance of hydrated electron at 700 nm as a function laser energy at 266 nm (homogeneous aqueous solution, 2×10^{-4} M $Fe(bpy)_2(CN)_2$), $l = 1$ cm.

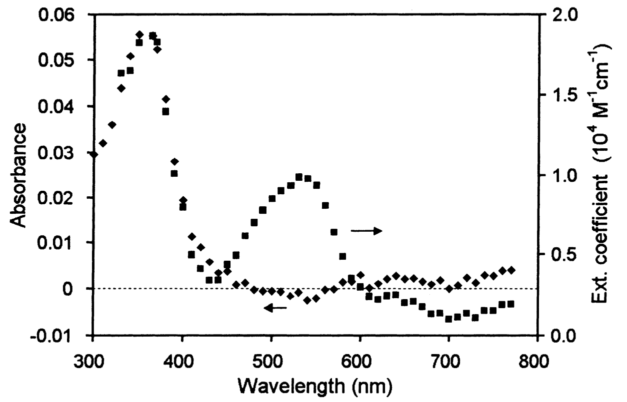

Fig. 4. Absorption spectrum 1 μs after a 266 nm laser pulse is delivered to a solution of 3×10^{-4} M $Fe(bpy)_2(CN)_2$ in homogeneous aqueous solution, and corrected spectrum of $Fe(bpy)_2(CN)_2^-$.

In homogeneous aqueous solution, the initial absorbance of the hydrated electron at 700 nm is directly proportional to the incident laser energy, as shown in Fig. 3, with similar relationships observed in micellar solutions. These results suggest that the formation of e_{hyd}^- from $Fe(bpy)_2(CN)_2$ in these systems is a consequence of monophotonic excitation, an idea consistent with the rather high excitation energy involved.

The quantum yields for formation of hydrated electron were determined in the three systems studied, yielding values of 0.037, 0.039, and 0.035 for homogeneous aqueous, CTAB, and SDS solutions. The invariability of the quantum yields obtained in the three systems suggests that the kinetic energy of the electron formed upon 266 nm excitation is high enough to allow the electrons to escape from micelles as efficiently as from the water cage in which they were generated. These values are nearly the same as the saturation quantum yields measured in continuous irradiation studies of similar systems in which nitrate ion was the electron scavenger [12,13], indicating that hydrated electrons detected by flash photolysis in the nanosecond time regime escape primary geminate recombination. This is consistent with the results of femtosecond studies which show that primary geminate recombination between electron and photoionized water takes place within 60 ps [19].

3.2. Formation and decay of $Fe(bpy)_2(CN)_2^-$

After absorption of the laser pulse in all three of the solution systems studied, a transient absorbance spectrum, similar to that shown in Fig. 4, is observed after the electron has completely decayed. Fig. 5 shows how the peak of the transient absorption band at 370 grows in and then decays. Since the rate of growing-in of this band is identical to the decay rate of the hydrated electron (Fig. 1) in all cases it is safe to assume that this is an absorption band of the reduced electron scavenger, i.e. $Fe(bpy)_2(CN)_2^-$. Similar reactions were observed in aqueous solutions of $Ru(bpy)_2(CN)_2$ and $Fe(bpy)(CN)_4^{2-}$) and confirmed by pulse radiolysis experiments [2,11,20].

Since the transient spectrum in Fig. 4 was taken in homogeneous aqueous solution containing 2×10^{-4} M $Fe(bpy)_2(CN)_2$ 1μs after the laser pulse, when all electron decay is complete, it should be the sum of the absorption of $Fe(bpy)_2(CN)_2^+$ and $Fe(bpy)_2(CN)_2^-$ ions, diminished by the bleaching due to the converted $Fe(bpy)_2(CN)_2$ complex, i.e.,

$$\varepsilon = \varepsilon_+ + \varepsilon_- - 2\varepsilon_0 \qquad (1)$$

where the subscripts indicate the charge of the corresponding complex. From the known values of ε for the neutral and oxidized complexes at 370 nm (4430 and 1560 M^{-1} cm^{-1}, respectively) as well as for the hydrated electron at 700 nm (19 000 M^{-1} cm^{-1}) [21], the extinction coefficient of $Fe(bpy)_2(CN)_2^-$ at 370 nm can be estimated from the ratio of the growing-in of the absorbance at 370 nm to the decay of the electron absorbance at 700 nm in the first 500 ns after

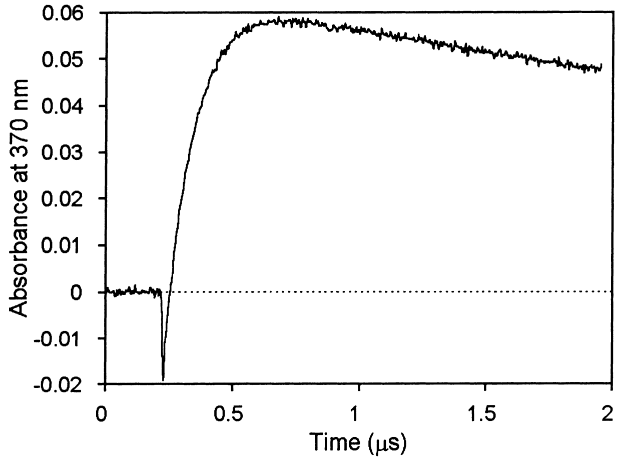

Fig. 5. Growth and decay of $Fe(bpy)_2(CN)_2^-$ at 370 nm for 3×10^{-4} $Fe(bpy)_2(CN)_2$ in homogeneous aqueous solution.

the laser pulse. This resulting value for ε_- at 370 nm ($18\,500 \pm 400$ M^{-1} cm^{-1}) gives a value of ε at the same wavelength, from which the values of ε, and hence ε_- can be determined at all wavelengths, yielding the entire spectrum of $Fe(bpy)_2(CN)_2{}^-$ (Fig. 4).

After reaching a maximum, the 700 nm absorbance signal in Fig. 5 decays by second-order kinetics, with $k/\varepsilon = 2.9 \pm 0.3 \times 10^6$ cm s^{-1}. Using a value of 11 200 M^{-1} cm^{-1} for ε, determined from Eq. (1), a value of $3.3 \pm 0.3 \times 10^{10}$ M^{-1} s^{-1} is obtained for the bimolecular rate constant for the comproportionation reaction between $Fe(bpy)_2(CN)_2{}^+$ and $Fe(bpy)_2(CN)_2{}^-$. This value well agrees with that determined earlier for the same complex (3.1×10^{10} M^{-1} s^{-1}) [15]. The corresponding values determined in CTAB and SDS solutions are $2.3 \pm 0.2 \times 10^{10}$ and $1.05 \pm 0.07 \times 10^{10}$ M^{-1} s^{-1}, respectively, indicating that the cationic and especially the anionic micelles hinder the reaction between the opposite charged complexes. This can be explained, again, by Coulombic interactions, since, in both cases, a reactant having a charge opposite to that of the actual micelle exists close to its interfacial region, whereas the other reactant, either incorporated into a micelle or free in the bulk solution, is electrostatically repulsed by the similarly charged micelle surfaces, thus retarding the comproportionation reaction.

In the solutions discussed so far it was assumed that the photogenerated hydrated electron was scavenged primarily by neutral ground state complex. In order to have broader picture of the micellar effects in these systems, the behavior of both cationic and anionic electron scavengers were also studied.

3.3. Addition of MV^{2+}

After the 266 nm laser pulse irradiation of homogeneous aqueous or micellar solutions of $Fe(bpy)_2(CN)_2$ containing methyl viologen dication the well-known spectrum of MV^+ radical cation [22–24], characterized by two main absorption bands at 395 and 605 nm, appears at a time delay of 1 μs. Fig. 6 shows that the growth of absorption of MV^+ followed at 395 nm occurs on the same time scale as the decay of the hydrated electron monitored at 700 nm, clearly demonstrating the scavenging of the electron by MV^{2+}. Fig. 7 shows plots of pseudo first-order rate constants for electron decay as a function of MV^{2+} concentration, taking into account the absorbance of the $MV^{\bullet +}$ formed. From linear regressions of these plots, the bimolecular rate constants for these systems have been determined, and for the homogeneous aqueous solution the value obtained is $8.4 \pm 0.5 \times 10^{10}$ M^{-1} s^{-1} which is in very good agreement with previous studies [25–27] of the reaction between $e_{hyd}{}^-$ and MV^{2+}. This value indicates this reaction in homogeneous aqueous solution is diffusion controlled.

The corresponding values determined in CTAB and SDS solutions (see Fig. 7, curves b and c) are $3.5 \pm 0.2 \times 10^{10}$ and $1.58 \pm 0.09 \times 10^{10}$ M^{-1}s^{-1}, respectively. Since the concentration of MV^{2+} did not exceed 2.5×10^{-4} M and that of the micelles (consisting of 61–62 surfactant ions) was about 3.5×10^{-4} M, the vast majority of the methyl viologen dications in the anionic micellar system were electrostatically bound to the micelle surface [28]. The apparent scavenging rate constant in this system is more than five times lower than that in homogeneous aqueous solution, which suggests that the surface-bound MV^{2+} ions scavenge less efficiently the hydrated electrons than do those in the solution bulk. This effect can be attributed to the repulsion by the negatively charged surface. In the CTAB solution, the apparent scavenging rate constant is also lower than that in homogeneous aqueous solution. In this case, although all the methyl viologen dications are free in the bulk, they cannot as efficiently compete for the ejected electron as the micelles having a large positive charge on their surface.

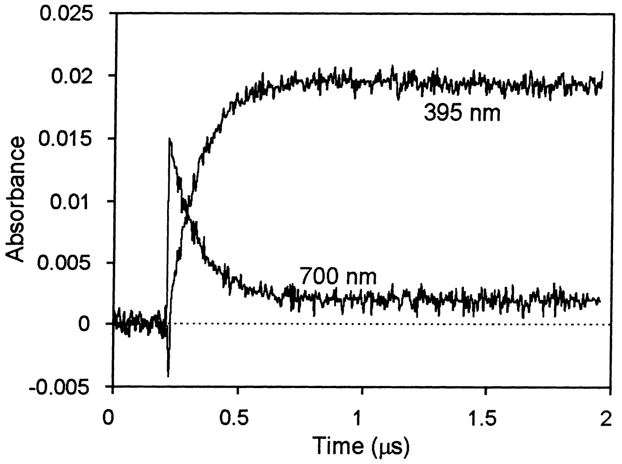

Fig. 6. Growth of $MV^{\bullet +}$ at 395 nm and decay of hydrated electron at 700 nm in homogeneous aqueous solution containing 1.5×10^{-4} M $Fe(bpy)_2(CN)_2$and 5×10^{-5} M MV^{2+}.

The $MV^{\bullet +}$ absorption decays via a second-order process in both homogeneous aqueous and micellar solution as shown in Fig. 8. This suggests a reaction with the $Fe(bpy)_2(CN)_2{}^+$ ions formed in the photoionization of the

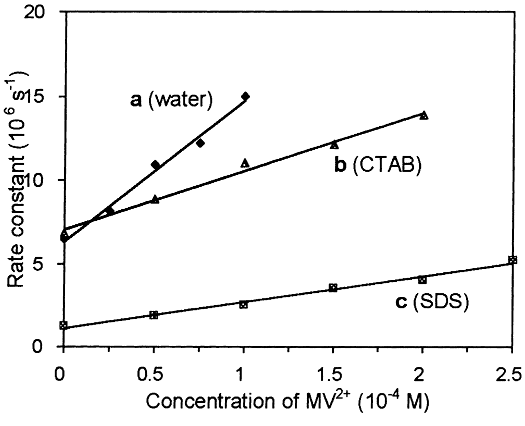

Fig. 7. Pseudo first-order decay rate constant of hydrated electron vs. MV^{2+} concentration in (a) homogeneous aqueous solution; (b) 0.022 M CTAB, and (c) 0.030 M SDS (1.5×10^{-4} M $Fe(bpy)_2(CN)_2$).

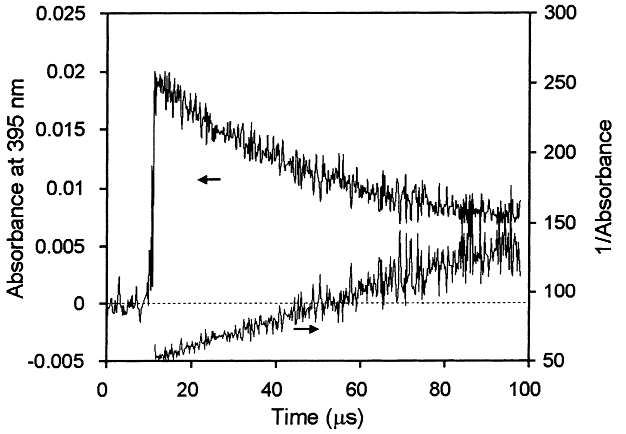

Fig. 8. Decay of $MV^{\cdot +}$ at 395 nm in homogeneous aqueous solution (1.5×10^{-4} M $Fe(bpy)_2(CN)_2 + 5 \times 10^{-5}$ M MV^{2+}).

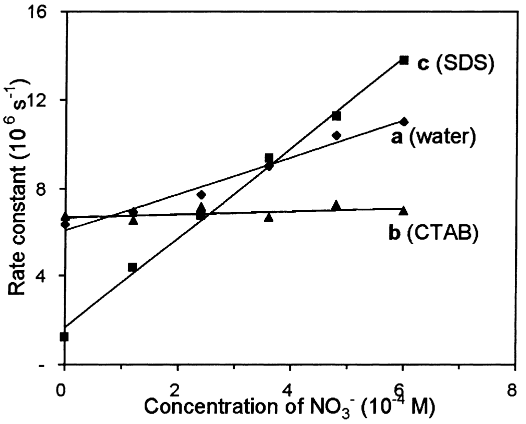

Fig. 9. Pseudo first-order decay rate constant of hydrated electron vs. NO_3^- concentration in (a) homogeneous aqueous solution, (b) 0.022 M CTAB, and (c) 0.030 M SDS (1.5×10^{-4} M $Fe(bpy)_2(CN)_2$).

neutral complex. Taking the value of the extinction coefficient of MV^+ to be 3.8×10^4 M^{-1} cm^{-1} at 395 nm [23] the resulting second-order rate constants of this reaction are $2.4 \pm 0.6 \times 10^{10}$, $8.9 \pm 2.3 \times 10^{10}$, and $1.8 \pm 0.4 \times 10^{10}$ $M^{-1}s^{-1}$ in the homogeneous aqueous, CTAB, and SDS solutions, respectively. Thus, this reaction is accelerated by the cationic and significantly slowed down by the anionic micelles, compared to the case of homogeneous aqueous solution. In this case reactants of similar (positive) charge interact in contrast to the reactions previously discussed. The anionic micelles attract both of them, and, because the micelle/reactant concentration ratio is well over ten, the reactants will be situated on different micelles. In the presence of CTAB, the repulsion by the cationic surfaces increases the effective concentrations of the reactants in the solution bulk. According to this explanation, a significant part of the $Fe(bpy)_2(CN)_2^+$ ions leave the parent micelles (the $MV^{\cdot +}$ radical ions in this system are predominantly formed in the solution bulk).

3.4. Addition of NO_3^-

Since, like methyl viologen dications, nitrate ions are also efficient electrons scavenger [29], addition of NO_3^- diminishes the lifetime of the hydrated electron formed in the systems studied. In homogeneous aqueous solution and in the presence of SDS (at constant $Fe(bpy)_2(CN)_2$ concentration) an increase in the concentration of NO_3^- ions results in a more efficient competition with the ground-state complex for the hydrated electron, decreasing its lifetime. The pseudo first-order rate constants for the electron decay monitored at 700 nm are shown in Fig. 9 as functions of the nitrate concentration, and from the slopes of these plots the apparent bimolecular rate constants for the electron scavenging reaction by nitrate were found, yielding $8.3 \pm 0.6 \times 10^9$ M^{-1} s^{-1} in homogeneous aqueous solution and $2.05 \pm 0.07 \times 10^{10}$ M^{-1} s^{-1} in the presence of dodecyl sulfate. The negatively charged micelles increase the rate of this reaction (by more than two fold) because they repulse both reactants, and thus enhance their actual concentration in the solution bulk.

Increase in the nitrate concentration in the range studied causes only a slight enhancement in the pseudo first-order rate constant in the presence of CTAB (Fig. 9, curve b). This result indicates that almost all the nitrate ions in this system are bound to the micellar surface, and, thus, retarded from the reaction with electrons ejected into solution. Moreover, nitrate ions (in this concentration range) do not efficiently influence the capture of hydrated electrons by the positively charged micelles.

4. Conclusions

Photoionization of $Fe(bpy)_2(CN)_2$ has been effectively utilized for the investigation of micellar effects on reactions of intermediates formed in the absence and in the presence of electron scavengers. Monitoring the formation and decay of the hydrated electron, $Fe(bpy)_2(CN)_2^-$, and $MV^{\cdot +}$ revealed that micelles of different charge significantly influence the reactions of these species. The effects observed can be uniformly interpreted in terms of Coulombic interactions between the actual reactants and the charged surface of the micelles. For this general interpretation, the rate constants determined in the systems studied have been summarized in Table 1. If the reactants are oppositely charged, both anionic and cationic micelles hinder their reactions because in each case one of them is attracted, while the other is repulsed by the micellar surface. If both reactants are similarly (either positively or negatively) charged, the micelles of similar charge increase, while those of opposite charge decrease their reaction rates. In the former case, repulsion enhances the actual concentration of the reactants in the bulk solution, while attraction to the oppositely charged micellar surface keeps them away from each other. If only one of the reactants is ionic, the micelles of similar charge cause a

Table 1
Bimolecular rate constants for reactions of similarly and oppositely charged reactants in homogeneous aqueous, CTAB, and SDS solutions

Reaction between reactants of	k ($10^{10} M^{-1} s^{-1}$)		
	SDS	Water	CTAB
Opposite charges			
$e_{aq}^{-} + C^{+a}$	0.75 ± 0.19	4.0 ± 0.8	4.4 ± 0.8
$e_{aq}^{-} + MV^{2+}$	1.58 ± 0.09	8.4 ± 0.5	3.5 ± 0.2
$C^{+} + C^{-}$	1.05 ± 0.07	3.3 ± 0.3	2.3 ± 0.2
Negative and zero charges			
$e_{aq}^{-} + C$	0.55 ± 0.05	2.8 ± 0.2	2.9 ± 0.2
Negative charges			
$e_{aq}^{-} + NO_3^{-}$	2.05 ± 0.07	0.83 ± 0.06	0.07 ± 0.05
Positive charges			
$MV^{+} + C^{+}$	1.8 ± 0.4	2.4 ± 0.6	8.9 ± 2.3

[a] $C = Fe(bpy)_2(CN)_2$

retardation of their reaction, due to repulsion, while attraction of the oppositely charged micelles containing the neutral reactants is compensated by the potential barrier at the interfacial region.

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