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Substituent effect on the fluorescence quenching of various tetraphenylporphyrins by ruthenium tris(2,2′-bipyridine) complex

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Abstract

The fluorescence quenching of various substituted tetraphenylporphyrins and their zinc complexes by ruthenium(II) tris(2,2′-bipyridine) has been investigated in dichloromethane. While fluorescence quenching was not effective for free base porphyrins, but in the case of zinc porphyrins fairly efficient. Plots for the Stern–Volmer quenching constant versus the calculated free energy change (ΔG_{red}) for photoinduced oxidative electron transfer (ET) processes from the excited singlet porphyrins to ruthenium(II) tris(2,2′-bipyridine) were shown to be linear. Among porphyrins/ruthenium(II) tris(2,2′-bipyridine) pairs examined in this study, ZnTMeOPP/ruthenium(II) tris(2,2′-bipyridine) pair exhibited most efficient fluorescence quenching as predicted to carry out most favorable photoinduced oxidative electron transfer processes by the calculated free energy changes (ΔG_{red}).

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Keywords: Photoinduced electron transfer; Fluorescence quenching; Porphyrins

1. Introduction

Considerable research effort has been devoted to the mimicry of the photoinduced electron transfer (ET) and charge separation in the natural photosynthetic reaction center [1–14]. One approach for modeling of the photosynthetic reaction center includes the synthesis of covalently linked donor–acceptor multicomponent system [1–10]. Another approach is the use of noncovalent assembly between donor and acceptor [11–14]. A large number of electron donors and acceptors are covalently or noncovalently linked in multicomponent photoinduced electron transfer systems. Choice of suitable donor–acceptor couple is most crucial in the design of multicomponent systems.

In natural photosynthetic systems, the primary electron transfer step occurs from a porphyrin-based complex [2]. Photophysical properties of porphyrins have been extensively investigated, and model compounds have been developed in order to gain a better understanding of natural photosynthetic mechanism [1–3,5,6,8–15]. Ruthenium(II) tris(2,2′-bipyridine) ($\mathrm{Ru(bpy)_3}^{2+}$) complexes have been of considerable interest because of their photophysical, photochemical and electrochemical properties [4,7,15]. Therefore, porphyrins and $\mathrm{Ru(bpy)_3}^{2+}$ complex are most popularly used as electron transfer sensitizers and currently employed as major components of photochemical molecular device converting light energy into chemical energy. Porphyrins or $\mathrm{Ru(bpy)_3}^{2+}$ complexes linked to a variety of electron acceptor (e.g. a quinone or a viologen) have been systematically studied [15]. Studies made with many chemically linked donor–acceptor model systems have served to reveal the effects of structural factors and the driving force on the rate of the charge-separation process. Increasing the forward electron transfer rate constant could be obtained by small energy gap between excited electron donor and charge separated state. Both porphyrins and $\mathrm{Ru(bpy)_3}^{2+}$ complex not only absorb visible light and functioning as a real redox unit, but also energy gap between excited electron donor and charge separated state is evaluated to be small. Moreover, either porphyrin or ruthenium complex subunit shows numerous examples of structural modulation through rational design of the ligand structure. This allows us to investigate the effects of structural factors and the driving force on the rate of the charge-separation process in the multicomponent system containing both porphyrin and ruthenium complex. In this context, some multicomponent systems containing both porphyrins and ruthenium complexes have been developed [16–20], but they usually favor energy transfer processes with respect to the desired electron transfer. In these systems, ruthenium complexes employed was not $\mathrm{Ru(bpy)_3}^{2+}$.

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However, two dyads containing zinc porphyrin and $Ru(bpy)_3^{2+}$ complex were reported recently. One used tetratolylporphyrin [19] bearing electron-donating substituent and the other used trifluoroacetamidophenylporphyrin [20] bearing electron-accepting substituent. In both cases, it was reported that efficient fluorescence quenching by photoinduced electron transfer was observed. These results motivate us to investigate systematically the structural effect on the photoinduced electron transfer and the resulting fluorescence quenching.

In the present study, porphyrins and $Ru(bpy)_3^{2+}$ complex were chosen as electron donor and electron acceptor, respectively. Tetraphenylporphyrins with various substituents and their zinc complexes are employed to investigate how variation of the excited state energy and redox potential of tetraphenylporphyrin controlled by various substituents affects the efficiencies of photoinduced electron- and/or energy transfer between tetraphenylporphyrin and $Ru(bpy)_3^{2+}$ complex. Fluorescence quenching of various substituted tetraphenylporphyrins by $Ru(bpy)_3^{2+}$ complex will reveal the information on the photoinduced electron- and/or energy transfer processes and best donor–acceptor pair of porphyrin and $Ru(bpy)_3^{2+}$ complex to accomplish efficient electron transfer.

2. Experimental details

$Ru(bpy)_3Cl_2$ was purchased from Aldrich. Chloride salt, $Ru(bpy)_3Cl_2$, was replaced with PF_6 salt, $Ru(bpy)_3(PF_6)_2$, by adding saturated aqueous NH_4PF_6. Various porphyrin derivatives were prepared using the known method [21–24] and purified by repeated recrystallization.

Absorption spectra were recorded on a Hitachi U-3210 spectrophotometer. Fluorescence spectra were obtained on an Aminco-Bowman Series 2 luminescence spectrometer. The concentrations of various porphyrin derivatives were controlled to be ca. 2×10^{-5} M while the concentration of $Ru(bpy)_3^{2+}$ was varied between 2×10^{-3} and 8×10^{-3} M. Fluorescence excitation wavelengths were 590 nm for ZnTCNPP and ZnTTP, 600 nm for ZnTPP, and 594 nm for ZnTMeOPP. Fluorescence quantum yields (Φ_f) were determined using TPP as a standard ($\Phi_f = 0.13$ in benzene) [25]. Electrochemical measurements were made using a Bioanalytical Systems (BAS) CV-50W potentiostat. Cyclic voltammetry was carried out in dichloromethane using the conventional three electrodes arrangement, consisting of a gold working electrode, a caromel reference electrode and a coiled platinum counter electrode. A 0.1 M tetrabutylammonium perchlorate (TBAP) was used as a supporting electrolyte.

3. Results and discussion

Figs. 1 and 2 show the absorption spectra of TTP and ZnTTP, respectively, with varying the concentration of $Ru(bpy)_3^{2+}$ in dichloromethane. UV–VIS absorption spectra show two major absorption bands, which can be assigned to the LC band of $Ru(bpy)_3^{2+}$ (288 nm) and the Soret band of porphyrin (420 nm). In addition to these two major bands, the MLCT band of $Ru(bpy)_3^{2+}$ (451 nm) and the Q bands of porphyrin (500–650 nm) are also observed. The comparison of the porphyrin Q bands in the mixture of TTP (or ZnTTP) and $Ru(bpy)_3^{2+}$ with the corresponding pure porphyrins shows slight changes of these bands suggesting some interaction, even if weak, between porphyrins and $Ru(bpy)_3^{2+}$ complex.

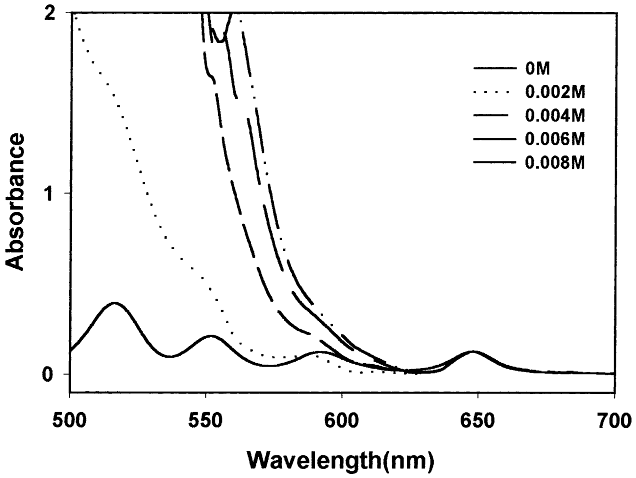


Fig. 1. The absorption spectra of TTP with varying the concentration of $Ru(bpy)_3^{2+}$ in dichloromethane. The concentration of TTP is 2×10^{-5} M.

The fluorescence wavelength maxima (650–660, 710–720 nm) of porphyrins are longer than those of their Zn complexes (600–610, 650–660 nm). Absorption and fluorescence data, and the excited state energy of various free base porphyrins are summarized in Table 1. Table 2 summarizes the absorption and luminescence data, and the excited state energy of various zinc porphyrins and $Ru(bpy)_3^{2+}$ complex.

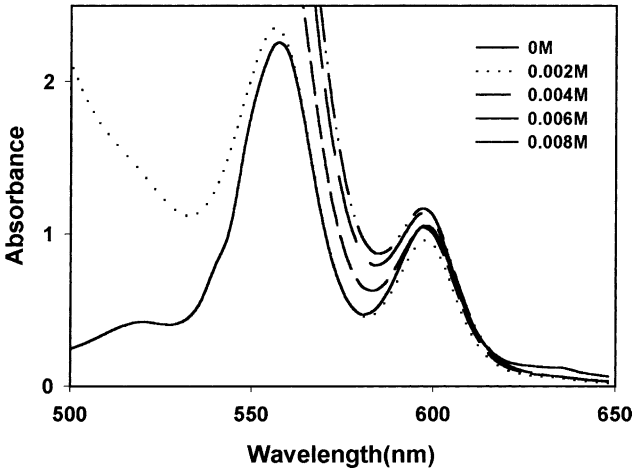


Fig. 2. The absorption spectra of ZnTTP with varying the concentration of $Ru(bpy)_3^{2+}$ in dichloromethane. The concentration of ZnTTP is 2×10^{-5} M.

Table 1
Absorption and emission data and excited state energy of various porphyrin derivatives[a] in dichloromethane

Compound	λ_a^{max} (nm)		λ_f^{max} (nm)	E_S (eV)	Φ_f	τ_f (ns)
	Soret band	Q bands				
TMeOPP	419	513, 545, 588, 651	656, 716	1.90	0.07	–
TTP	420	516, 552, 594, 650	655, 717	1.90	0.09	8.6[b]
TPP	419	516, 552, 594, 645	656, 717	1.90	0.09	–
TCNPP	421	515, 549, 589, 647	657, 717	1.90	0.08	–
TPFPP	422	516, 552, 594, 636	641, 709	1.94	0.05	8.5[c]

[a] The concentrations of porphyrin derivatives are 2×10^{-5} M.
[b] From [26].
[c] From [27].

Table 2
Absorption and emission data and excited state energy of various zinc porphyrin derivatives[a] in dichloromethane

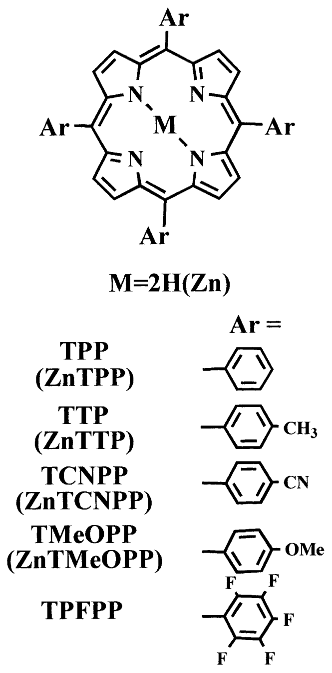
Compound	λ_a^{max} (nm)		λ_f^{max} (nm)	E_S (eV)	Φ_f	τ_f (ns)
	Soret band	Q bands				
ZnTMeOPP	420	550, 588	596, 645	2.10	0.032	–
ZnTTP	420	548, 586	600, 648	2.09	0.031	1.6[b]
ZnTPP	420	548, 584	597, 647	2.10	0.037	1.9[c]
ZnTCNPP	420	547, 586	600, 649	2.09	0.050	–
($Ru(bpy)_3^{2+}$)[d]	244, 254 (sh)	288, 451	626	1.98[e]	0.062	920

[a] The concentrations of zinc porphyrin derivatives are 2×10^{-5} M.
[b] From [27].
[c] In MeTHF from [28].
[d] Data in acetonitrile from [29]. Emission of $Ru(bpy)_3^{2+}$ is not related to fluorescence, but comes from triplet state.
[e] From [30].

Fluorescence quenching experiments for a variety of tetraphenylporphyrins containing various electron-donating (methyl, methoxy) and electron-accepting (pentafluorophenyl, cyano) substituents with increasing the concentration of $Ru(bpy)_3^{2+}$ have been conducted. Structures of porphyrin derivatives used in this study are represented in Scheme 1. The fluorescence spectra of TTP and TCNPP, and ZnTTP and ZnTCNPP at various concentrations of $Ru(bpy)_3^{2+}$ in dichloromethane are shown in Figs. 3 and 4, respectively. While fluorescence intensities of porphyrin derivatives remain nearly unchanged, the fluorescence intensities of zinc porphyrin derivatives gradually decrease as the concentration of $Ru(bpy)_3^{2+}$ is increased up to 8×10^{-3} M.

While fluorescence quenching of free base porphyrins by $Ru(bpy)_3^{2+}$ is not so effective, fluorescence of zinc porphyrins are quenched efficiently. Various ligands are known to coordinate on axial position of zinc porphyrin. Visible spectra are affected more largely in zinc porphyrin (Fig. 2) than free base porphyrin (Fig. 1). The absorption spectra revealed the static quenching, although the complexation-induced spectral changes are small. Ruthenium complex can coordinate to the zinc porphyrin and the local concentration of the quencher around the zinc porphyrin might be higher as expected. Then, efficient quenching can occur in the supramolecule.

Stern–Volmer quenching constants $k_q\tau$ for the fluorescence quenching of zinc porphyrins are listed in Table 4 and



Scheme 1. Structures of porphyrin and zinc porphyrin derivatives used in this study.

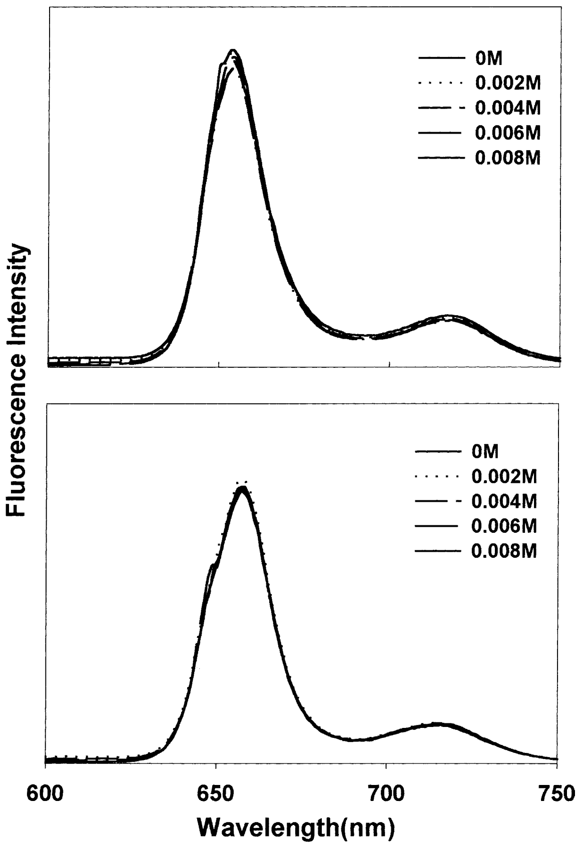


Fig. 3. The fluorescence spectra of TTP (top) and TCNPP (bottom) with varying the concentration of $Ru(bpy)_3{}^{2+}$ in dichloromethane. The concentrations of TTP and TCNPP are 2×10^{-5} M.

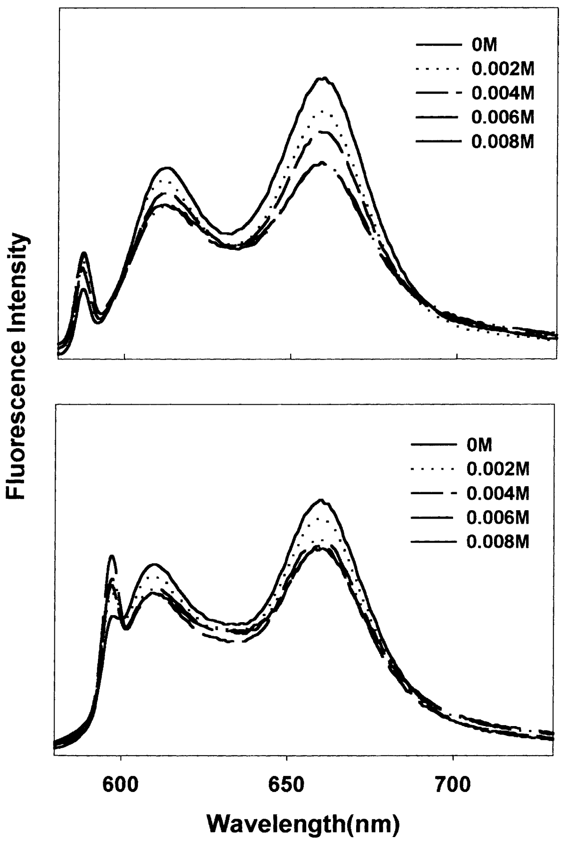


Fig. 4. The fluorescence spectra of ZnTTP (top) and ZnTCNPP (bottom) with varying the concentration of $Ru(bpy)_3{}^{2+}$ in dichloromethane. The concentrations of ZnTTP and ZnTCNPP are 2×10^{-5} M.

are shown to be the largest for ZnTMeOPP. Stern–Volmer quenching constant is increased as the substituent has more electron-donating ability. This indicates that the porphyrin derivatives containing more electron-donating substituents such as ZnTTP and ZnTMeOPP undergo probably faster photoinduced electron transfer amongst various porphyrin/$Ru(bpy)_3{}^{2+}$ pairs.

To confirm the electron transfer quenching, solvent effect on fluorescence quenching was investigated. In less polar solvent (toluene–acetonitrile mixed solvent (3/1, v/v)), fluorescence of ZnTTP proved to be much less efficiently quenched with adding $Ru(bpy)_3$ complex than in more polar solvent (CH_2Cl_2), as expected. This result supports that the fluorescence quenching may be due to the photoinduced electron transfer.

Fluorescence can be quenched through either energy transfer or electron transfer. To get the information on the energetics for energy transfer and electron transfer processes and to examine which one is energetically favorable quenching pathway, the electrochemical study was carried out in addition to spectroscopic measurements. Fig. 5 shows cyclic voltammograms of TMeOPP and ZnTMeOPP. The redox potentials of various porphyrins and zinc porphyrins, and the calculated free energy changes (ΔG_{ox}) for the photoinduced oxidative electron transfer processes ($P^*/Ru(bpy)_3^{2+} \rightarrow P^+/Ru(bpy)_3{}^{+}$) from various excited porphyrins and zinc porphyrins (P^*) to $Ru(bpy)_3{}^{2+}$, and the calculated free energy changes (ΔG_{red}) for the photoinduced reductive electron transfer processes ($P^*/Ru(bpy)_3^{2+} \rightarrow P^-/Ru(bpy)_3{}^{3+}$) from $Ru(bpy)_3{}^{2+}$ to various excited porphyrins and zinc porphyrins are also collected in Tables 3 and 4, respectively. From these data, the energy diagrams for the photoinduced electron transfer between various excited porphyrins or zinc porphyrins and $Ru(bpy)_3{}^{2+}$ could be represented.

Schematic representation in Scheme 2 (or Scheme 3) shows the main pathways of ET and charge recombination (CR) processes, as well as excitation and emission in the present TMeOPP (or ZnTMeOPP) and $Ru(bpy)_3{}^{2+}$ systems. There are probable /CR channels not only between the singlet porphyrins and corresponding $P^+/Ru(bpy)_3{}^{+}$ states in TMeOPP ($\Delta G_{\text{ox}} = 0.37$ eV) and ZnTMeOPP ($\Delta G_{\text{ox}} = -0.05$ eV), but also between the singlet porphyrins and corresponding $P^-/Ru(bpy)_3{}^{3+}$ states in TMeOPP ($\Delta G_{\text{red}} = 0.53$ eV) and ZnTMeOPP ($\Delta G_{\text{red}} = 0.43$ eV). Another probable ET/CR pathway exists from the triplet state of $Ru(bpy)_3{}^{2+}$ to form $P^+/Ru(bpy)_3{}^{+}$ ($\Delta G = 0.08$ eV for TMeOPP, -0.12 eV for ZnTMeOPP) or to form $P^-/Ru(bpy)_3{}^{3+}$ ($\Delta G = 0.45$ eV for TMeOPP, 0.55 eV for

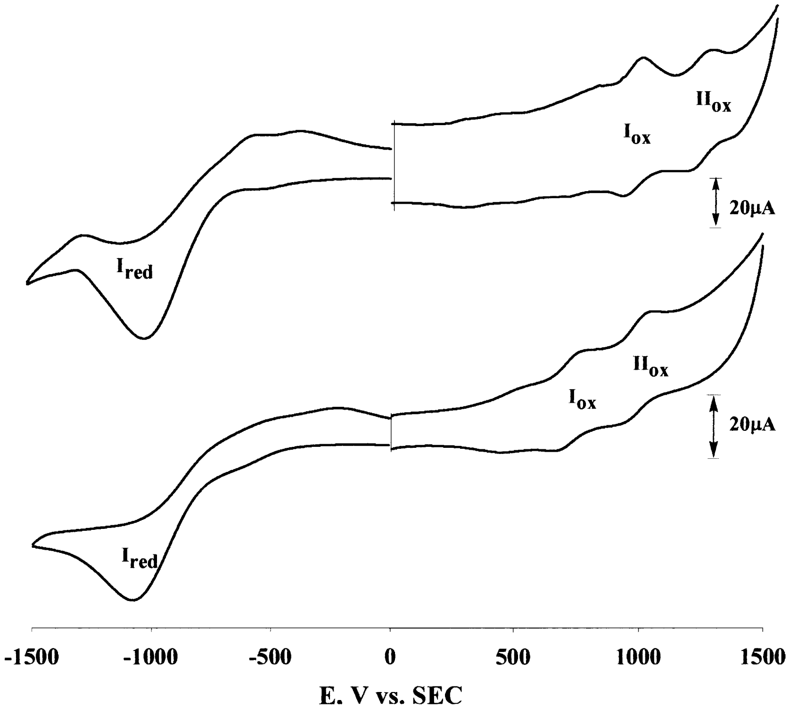


Fig. 5. Cyclic voltammograms of TMeOPP (top) and ZnTMeOPP (bottom).

ZnTMeOPP). In both cases of reductive electron transfer to form $P^-/Ru(bpy)_3{}^{3+}$, the energy costs are very unfavorable. Actually, the only exergonic pathway is the electron transfer from singlet zinc porphyrins to form corresponding $P^+/Ru(bpy)_3{}^+$ states, although the electron transfer from the triplet state of $Ru(bpy)_3{}^{2+}$ to form corresponding $P^+/Ru(bpy)_3{}^+$ states, which is only slightly endergonic, is energetically possible.

Fluorescence quenching studies revealed considerable quenching for zinc porphyrins while little quenching for free base porphyrins. As shown in Tables 3 and 4, the calculated free energy changes for the photoinduced electron transfer processes from the photoexcited singlet state of porphyrins to $Ru(bpy)_3{}^{2+}$ also suggest that the photoinduced oxidative electron transfer is energetically favorable in all zinc porphyrins examined except ZnTCNPP, even though unfavorable in all free base porphyrins. The ΔG_{ox} and ΔG_{red} values however suggest that this quenching is not due to reductive electron transfer from the excited singlet porphyrin to $Ru(bpy)_3{}^{2+}$, but because of probable oxidative electron transfer reducing the singlet porphyrin. The energies of the singlet zinc porphyrins are estimated to be close to or higher than their corresponding $P^+/Ru(bpy)_3{}^+$ states allowing electron transfer/charge recombination occur between these energy levels.

In the case of ZnTMeOPP, efficient quenching of ZnTMeOPP and the appearance of $Ru(bpy)_3{}^{2+}$ emissions are found upon porphyrin excitation. Energy transfer from singlet free base porphyrins (1.9 eV) to form triplet $Ru(bpy)_3{}^{2+}$ (1.98 eV) is energetically unfavorable, but energy transfer from singlet zinc porphyrins (2.10 eV) to form triplet $Ru(bpy)_3{}^{2+}$ (1.98 eV) is possible. However, this is an indirect process. It is necessary that change singlet to triplet state in a compound occurs: $^1P^* \rightarrow {}^3P^* + Ru \rightarrow P + {}^3Ru^*$

Table 3
Charge separated state energies (E_{css}), and redox potentials of various porphyrin derivatives by $Ru(bpy)_3{}^{2+}$ in dichloromethane

Compound	$E_{1/2}$ (V)		E_{css} (eV)		ΔG_{ox}	ΔG_{red}
	Oxidation	Reduction	P^+/Ru^+	P^-/Ru^{3+}		
TMeOPP	0.94	−1.14	2.27	2.43	0.37	0.53
TTP	0.93[a]	−1.20[a]	2.26	2.49	0.36	0.59
TPP	1.08[b]	−1.21[b]	2.41	2.50	0.51	0.60
TCNPP	1.29[b]	−1.03[b]	2.62	2.32	0.72	0.42
TPFPP	1.48[c]	−0.85[c]	2.81	2.14	0.87	0.20

[a] From [31].
[b] From [32].
[c] From [33].

Table 4
Charge separated state energies and redox potentials and Stern–Volmer quenching data of various zinc porphyrin derivatives by $Ru(bpy)_3^{2+}$ in dichloromethane

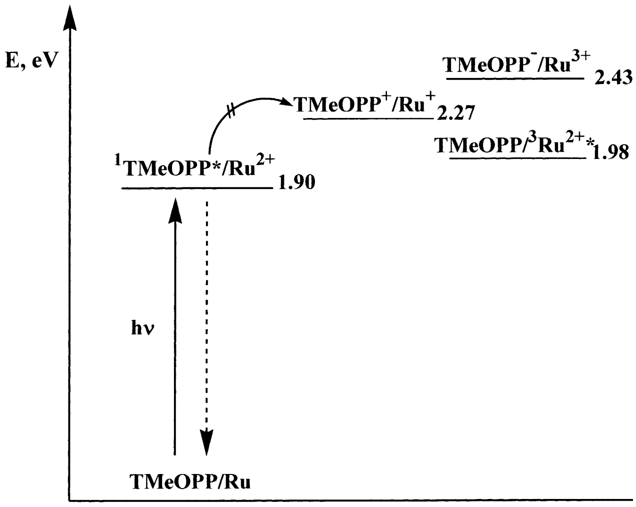
Compound	$E_{1/2}$ (V)		E_{css} (eV)		ΔG_{ox}	ΔG_{red}	$k_q\tau$[a]
	Oxidation	Reduction	P^+/Ru^+	P^-/Ru^{3+}			
ZnTMeOPP	0.72	−1.24	2.05	2.53	−0.05	0.43	37
ZnTTP	0.74	−1.22	2.07	2.51	−0.02	0.42	35
ZnTPP	0.82	−1.20	2.15	2.49	0.05	0.39	32
ZnTCNPP	0.89	−1.16	2.22	2.45	0.13	0.36	29
$Ru(bpy)_3^{2+}$	1.29[b]	−1.33[b]	–	–	–	–	–

[a] For Stern–Volmer quenching experiments, the concentrations of zinc porphyrin derivatives of 2×10^{-5} M were employed and the concentration of $Ru(bpy)_3^{2+}$ was varied between 2×10^{-3} and 8×10^{-3} M.

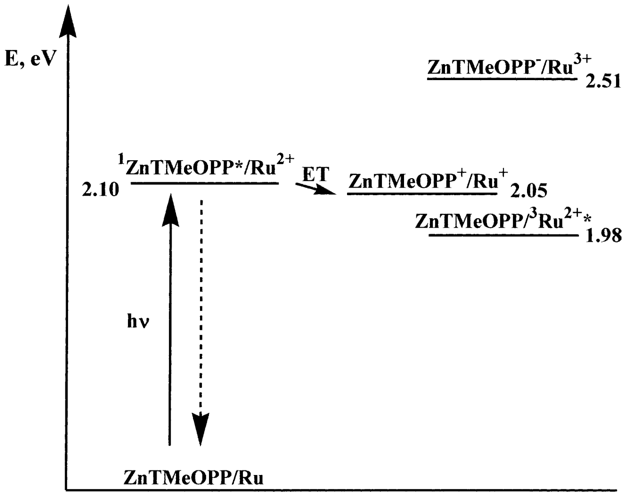
[b] Data in acetonitrile from [29].

or $^1P^* + Ru \rightarrow P + {}^1Ru^* \rightarrow {}^3Ru^*$. The excitation spectra revealed that the emissions were obtained upon photoexcitation of $Ru(bpy)_3^{2+}$, thus ruling out energy transfer as the origin of $Ru(bpy)_3^{2+}$ emission in these systems. It suggests an electron transfer channel for singlet quenching of ZnTMeOPP, supported by the negative ΔG_{ox} values for these electron transfer processes of −0.05 eV for ZnTMeOPP.

Stern–Volmer quenching constant shows good correlation with ΔG_{ox}, but not ΔG_{red}. In comparison with the mixture of ZnTCNPP and $Ru(bpy)_3^{2+}$, the mixture of ZnTTP and $Ru(bpy)_3^{2+}$ shows very efficient fluorescence quenching due to more favorable energy gap ΔG_{ox} (see Fig. 4). Zinc porphyrin containing more electron-donating substituent exhibits lower oxidation potential to yield more negative ΔG_{ox}. Stern–Volmer constant for fluorescence quenching of ZnTMeOPP, which has lowest ΔG_{red}, is greatest. This assures that the electron transfer is possibly one of the major factors contributing to the quenching mechanism. ZnTMeOPP was proven to be the best electron donor towards $Ru(bpy)_3^{2+}$ in photoinduced electron transfer donor–acceptor system.



Scheme 2. Energy-level diagram showing the deactivation processes of the excited singlet TMeOPP and $Ru(bpy)_3^{2+}$.



Scheme 3. Energy-level diagram showing the deactivation processes of the excited singlet ZnTMeOPP and $Ru(bpy)_3^{2+}$.

4. Conclusions

Fluorescence quenching studies of various free base porphyrins and zinc porphyrins by $Ru(bpy)_3^{2+}$ have been conducted and revealed fairly effective quenching for zinc porphyrins in contrast to free base porphyrins. Zinc porphyrins containing electron-donating substituent exhibited more efficient fluorescence quenching. The good correlation was observed between Stern–Volmer constant and ΔG_{ox}, even though the free energy change of photoinduced oxidative electron transfer from the singlet excited zinc porphyrin to $Ru(bpy)_3^{2+}$ is only slightly exergonic. It suggests that most probable pathway for fluorescence quenching is photoinduced oxidative electron transfer from the excited singlet porphyrin to $Ru(bpy)_3^{2+}$. ZnTMeOPP/$Ru(bpy)_3^{2+}$ was identified as a best donor–acceptor pair among various substituted porphyrin/$Ru(bpy)_3^{2+}$ pairs examined.

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