LUMINESCENCE QUENCHING OF MIXED-LIGAND RUTHENIUM (II) COMPLEXES BY DIFFERENT QUENCHERS

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Abstract

The effect of ionic strength and acidity (pH) on the luminescence quenching of the excited states of a number of mixed-ligand Ru(II) complexes have been studied. The mixed-ligand Ru(II) complexes of diphenyl-thioethylene (dpte); 2,2′-bipyridine (bpy), 2-(2-pyridyl)-quinoline (pyq); 4,6-dichloro-2-(2-pyridyl)pyrimidine (dcppm); 4,6-dichloro-5-methyl-2-(2-pyridyl)pyrimidine (dcmppm); 4,6-dichloro-5-phenyl-2-(2 pyridyl)pyrimidine (dcpppm) with three quenchers: *N,N,N′,N′*-tetramethyl-*p*-phenlyene-diamine ($TMPD^{2+}$), methyl viologen (Mv^{2+}), and ethylenediaminetetraacetic acid (EDTA) have been used to study the effect of acidity. Whereas, for the effect of ionic strength, $[Ru(dpte)_2(dcpppm)]^{2+}$ /EDTA system has been used. The quenching rate constant (k_q) was found to increase with decreasing the ionic strength, while pH has the opposite effect. The quenching of mixed-ligand Ru(II) complexes by $TMPD^{2+}$ in aqueous solutions was shown to be dynamic and static in nature.

Introduction

The splitting of water by visible light is of great importance for the photochemical conversion and storage of solar energy. Several studies have been directed toward the generation of H_2 from aqueous solutions containing a photosensitizer to absorb the light and generate a long-lived excited state that can undergo electron transfer to a relay species.[1] The most thoroughly investigated model system is the one containing $Ru(bpy)_3^{2+}$ as the photosensitizer and methylviologen (1,1′-dimethyl-4,4′-bipyridinium dication; Mv^{2+}) as the electron relay.[2] As the search for new ruthenium (II)-diimine complexes that are capable of solar energy conversion and H_2 formation continues attempts are made to modify the behavior of such complexes by the use of substitution on the diimine ligand or through ligand variation. Recently, we have succeeded in synthesis of new mixed ligand Ru(II) complexes[3] in the hope of discovering new efficient photosensitisers for solar energy conversion. Mixed ligand Ru

(II)- complexes act as photosensitizers and catalysts for water splitting due to absorption of visible light that generates a long-lived excited state ^{3}MLCT (metal to ligand charge transfer).[4-6] This excited state has an ability of converting the excited energy to redox energy (electricity), also its ability to the photoreduction of water and generation of H_2 and the maximal photo and chemical stability.[5-10] Mixed ligand Ru (II) complexes are typically sensitizers used as solar energy converters, due to their unique ^{3}MLCT excited state.

Sufficient progress has been made with regard to theoretical and experimental studies of the electron transfer reactions,[11] several factors have been found to affect the quenching rate constant, some of these are: diffusion-control and cage release effect, the redox potentials of the donor and acceptor (the difference between the donor's oxidation potential and the acceptor's reduction potential determines the spontaneity of the process), the concentration of Ru(II)-complexes, ionic strength, and pH.

The effect of ionic strength and concentration on the quenching experiments has been investigated.[12,13] It has been reported that increasing the concentration of Ru (II)-complexes results in decreasing the quenching rate constant, and increasing ionic strength causes a decrease in both quantum yield of $MV^{+\cdot}$ and cage release efficiency of the system $[Ru\ (bpy)_3]^{+2}$/ EDTA/ MV^{+2}.[12-14] The effect of pH on the stability of quencher radical ($MV^{+\cdot}$, $TMPD^{+\cdot}$) also has been studied.[10,14]

It has been found that, methyl viologen cation react rapidly with *Ru (II) via electron transfer to produce Ru (III),[12-16] if an irreversibly oxidizable donor (such as EDTA) is present, then the reduced methyl viologen radical $MV^{+\cdot}$ will accumulate, and by the addition of catalysts such as PtO_2, hydrogen can be liberated efficiently from water as in equation (1):

$$\mathbf{MV^{+\cdot} + H^+ \xrightarrow{PtO_2} MV^{+2} + \tfrac{1}{2}\,H_2} \qquad \textbf{(1)}$$

A possible substrate for solar energy conversion is, $TMPD^{+2}$ that works as MV^{+2} but to a much lesser extent than MV^{+2}. This substrate never been investigated in literature and that is why we will focus on $TMPD^{+2}$ as photosensitizer for solar energy conversion.[11,17]

In this study we were motivated by the previous theories on factors that affect photochemical and electron transfer quenching, such as, the concentration of Ru (II) complexes, the electron relay species (MV^{+2},$TMPD^{+2}$), the effect of the ionic strength and the pH. Thus, in this paper, we report the results of the luminescence quenching of the excited states of new synthesized mixed- Ligand Ru (II) complexes by different quenchers (EDTA, $TMPD^{+2}$, MV^{+2}). Also, the results of the effect of ionic strength and acidity (pH) on the photochemical quenching are reported.

Results

Luminescence Quenching of the Ru (II)- Complexes by $TMPD^{+2}$

The Stren-Volmer constant (k_{sv}) of Ru (II)/ $TMPD^{+2}$ systems was evaluated in the early linear region at low quencher concentrations using the typical Stern-Volmer plots, and was studied as a function of time. Table (1) summarizes these results for $TMPD^{+2}$ as a function of time. Figure (1) shows the typical Stern-Volmer plot of the luminescence quenching of $[Ru(dpte)_2(dcpppm)]^{+2}$ by $TMPD^{+2}$.

Table 1. Stern –Volmer luminescence quenching data for Ru(II)-complexes/ $TMPD^{+2}$ system as a function of time. [Ru(II)]= $5x10^{-5}$ M. No control of pH and ionic strength

Complex	Liftime τ_o(ns)±5%	Time (hour)	K_{sv} (M^{-1})±10%	K_q (M^{-1} S^{-1}) ±10%
$[Ru (dpte)_2(bpy)]^{+2}$	328	0.0	20.55	$6.26x10^7$
		24	50.90	$1.55x10^8$
$[Ru (dpte)_2(pyq)]^{+2}$	251	0.0	83.10	$3.31x10^8$
		24	102.80	$4.10x10^8$
$[Ru (dpte)_2(dcpppm)]^{+2}$	686	0.0	175.11	$2.55x10^8$
		24	357.88	$5.22x10^8$
$[Ru (dpte)_2(dcppm)]^{+2}$	576	0.0	201.714	$3.50x10^8$
		24	383015	$6.65x10^8$
$[Ru (dpte)_2(dcmppm)]^{+2}$	610	0.0	261.10	$4.28x10^8$
		24	443.11	$7.26x10^8$

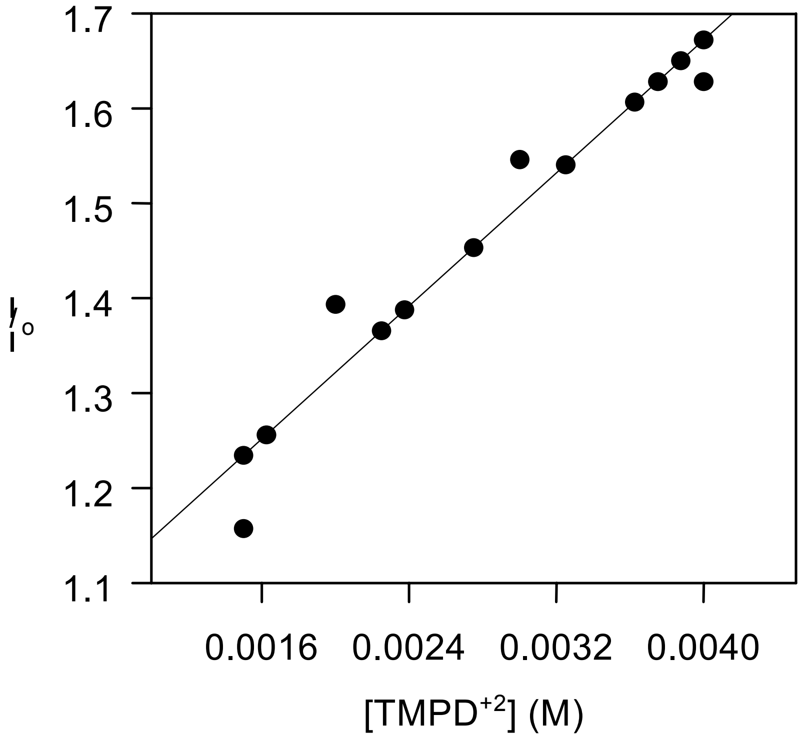

Figure 1. Stern –Volmer Plot of the luminescence quenching of $[Ru (dpte)_2 (dcpppm)]^{+2}$ ($5x10^{-5}$ M) by $TMPD^{+2}$.

Luminescence Quenching of the Ru (II)- Complexes by MV^{+2} and EDTA

The Stern-Volmer constants of Ru (II)/MV^{+2} and Ru(II)/EDTA systems at pH 4 and 10 were evaluated using Stern-Volmer plots. Figure 2 shows typical Stern-Volmer plot for Ru (II)/MV^{+2} system. Although many cases show a linear behavior, deviations from linearity sometimes occur. This can be attributed to aggregation between MV^{+2} and Cl^- at high MV^{+2} concentrations and to aggregation between EDTA and the sensitizer.[18] Tables (2-3) summarize these results for MV^{+2} and EDTA in acidic and basic media, respectively.

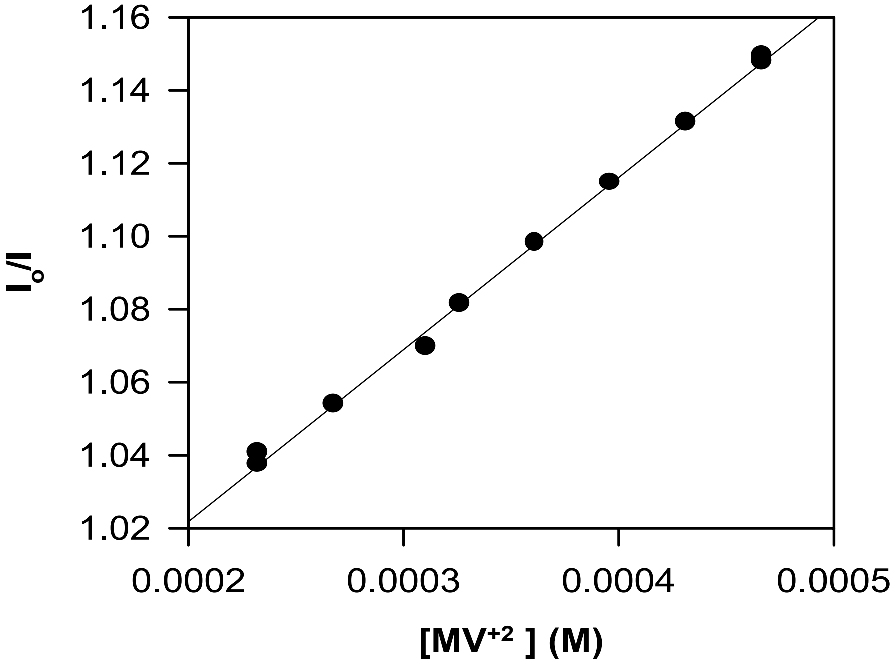

Figure 2. Stern –Volmer Plot of the luminscence quenching of [Ru (dpte)$_2$(dcpppm)]$^{+2}$ (5x10^{-5} M), by MV^{+2}, pH = 4.

Table 2. Stern –Volmer luminescence quenching data for Ru(II)-complexes/Mv^{+2} system as a function of pH. [Ru(II)]= 5x10^{-5} M.

Complex	Liftime τ_o(ns)±5%	pH	K_{sv} (M^{-1})±10%	K_q (M^{-1} S^{-1}) ±10%
[Ru (dpte)$_2$(bpy)]$^{+2}$	330	10	550	1.67x10^9
	325	4	543	1.67x10^9
[Ru (dpte)$_2$(pyq)]$^{+2}$	253	10	1461	5.77x10^9
	249	4	471	1.89x10^9
[Ru (dpte)$_2$(dcpppm)]$^{+2}$	686	10	1281	1.87x10^9
	520	4	428	8.23x10^8
[Ru (dpte)$_2$(dcppm)]$^{+2}$	578	10	1509	2.61x10^9
	496	4	426	8.58x10^8
[Ru (dpte)$_2$(dcmppm)]$^{+2}$	610	10	1824	2.99x10^9
	504	4	992	1.97x10^9

Table 3. Stern –Volmer luminescence quenching data for Ru(II)-complexes/EDTA system as a function of pH. [Ru(II)]= 5x10^{-5} M.

Complex	Liftime τ_o(ns)±5%	pH	K_{sv} (M^{-1})±10%	K_q (M^{-1} S^{-1}) ±10%
[Ru (dpte)$_2$(bpy)]$^{+2}$	330	10	4.38	1.33x10^7
	325	4	0.76	2.34x10^6
[Ru (dpte)$_2$(pyq)]$^{+2}$	253	10	10.46	4.14x10^7
	249	4	4.57	1.84x10^7
[Ru (dpte)$_2$(dcpppm)]$^{+2}$	686	10	88.96	1.30x10^8
	520	4	14.62	2.81x10^7
[Ru (dpte)$_2$(dcppm)]$^{+2}$	578	10	43.76	7.57x10^7
	496	4	32.45	6.54x10^7
[Ru (dpte)$_2$(dcmppm)]$^{+2}$	610	10	97.51	1.60x10^8
	504	4	38.40	7.62x10^7

Effect of ionic strength and pH on Luminescence Quenching

Table (4) summarizes the Stern-Volmer Luminescence quenching data for [Ru(dpte)$_2$(dcpppm)]$^{+2}$/EDTA at pH =11 and different values of ionic strength. Table (5) summarizes the Stern-Volmer Luminescence quenching data for [Ru(dpte)$_2$(dcpppm)]$^{+2}$/EDTA at ionic strength=2 M as a function of pH.

Table 4. Stern- Volmer luminescence quenching data for [Ru (dpte)$_2$(dcpppm)]$^{+2}$/ EDTA system at pH = 11.0 as a function of ionic strength. [Ru(II)]=5x10^{-5} M. $\lambda_{Ex.}$=450 nm, $\lambda_{Em.}$=575 nm. τ_o=686 ns.

Ionic Strength (M)	K_{sv} (M^{-1})±10%	K_q (M^{-1} S^{-1}) ±10%	Log k_q
1.82	98.26	1.43x10^8	8.16
2.00	94.37	1.38x10^8	8.14
2.10	87.25	1.27x10^8	8.10
2.26	78.42	1.14x10^8	8.06
2.40	77.52	1.13x10^8	8.05
2.53	77.02	1.12x10^8	8.05

Table 5. Stern- Volmer luminescence quenching data for [Ru (dpte)$_2$(dcpppm)]$^{+2}$/ EDTA system at ionic strength = 2 M as a function of pH. [Ru(II)]=5x10^{-5} M. $\lambda_{Ex.}$=450 nm, $\lambda_{Em.}$=575 nm.

pH	τ_o (ns)±5%	K_{sv} (M^{-1})±10%	K_q (M^{-1} S^{-1}) ±10%	Log k_q
4.2	520	15.4	2.96x10^7	7.41
5.5	520	23.6	4.54x10^8	7.66
6.3	686	45.7	6.66x10^7	7.82
7.5	686	52.5	7.65x10^7	7.88
8.6	686	65.3	9.52x10^7	7.98
9.5	686	77.4	11.28x10^7	8.05
10.4	686	89.2	13.00x10^7	8.11
11.2	686	96.6	14.08x10^7	8.15

Discussion

In this study, mixed- ligand (diimine and thiolene) Ru (II)-complexes[19-23] are chosen to achieve: (I) maximal photo and chemical stability, so using the dithiolene ligand (dpte), which is a π-acid ligand that split d-orbital strongly, therefore low-lying d-d states, which are most responsible for photodecomposition, will not exist; (II) high absorptivity, the presence of different ligands (diimine and dpte) reduce the symmetry of the complex, with the possibility of enhancing absorptivity, because the transferred electron is intrinsically localized on a single ligand which is the most easily reduced, and in the mixed ligand Ru (II) complexes the diimine ligands is more likely to be the easiest to reduce. All complexes in our study show high absorptivity.[3]

Luminescence Quenching of Ru (II)- Complexes by TMPD^{+2}

In Table (1) the Stern-Volmer slops (k_{sv}) are shown as a function of time for most complexes. Although most of the Stern-Volmer plots for this quencher show a linear behavior, deviations from linearity was occurred for some complexes at high quencher concentrations Figure 3. Thus, the Stern-Volmer constant was evaluated in the early linear region at low quencher concentrations. The [Ru (dpte) $_2$(dcmppm)]$^{+2}$/ TMPD^{+2} system shows the largest value of k_{SV}, due to the presence of CH_3- group at dcmppm ligand as an electron- donor which makes rich of electrons on the π-system that leads to electron- transfer easily from MLCT to the TMPD^{+2} (fast oxidative quenching), so it shows large (k_{SV}). And this value is larger than the value of K_{SV} for [Ru (dpte) $_2$(dcpppm)]$^{+2}$, due to presence of phenyl-group in [Ru (dpte) $_2$(dcpppm)]$^{+2}$, which stabilizes the electron by resonance effect (conjugated system), so it takes time for the electron-to be transferred to the TMPD^{+2} and leads to slower the oxidative quenching (low k_{SV}). For all complexes, the Stern-Volmer plots are curved at high concentration of TMPD^{+2} after 24 hour, and the value of k_{sv} become larger from that at zero time. This behavior can be explained in terms of oxidative electron transfer quenching and the present of the static quenching (complexation between the Ru (II)...TMPD^{+2} in the ground state) or to the formation of the exciplex and solvated ion pair. Table (1) shows that the value of k_{sv}, for all complexes, become larger after 24 hour, indicating an increase of the rate of the electron transfer quenching from Ru^{+2} to TMPD^{+2} to form Ru

(III)/ TMPD$^+$, postulating a competing electron transfer process occurring in a counter complex between Ru (II) and the quencher that gives rise to solvated ion pairs. Thus, Ru(II) and TMPD^{+2} in the ground state become closer to each other to form encounter complex and so the electron transfer becomes faster. The emission spectra of [Ru (dpte)$_2$(dcpppm)]$^{+2}$ in water shows the decreases in luminescence intensity as the concentration of TMPD^{+2} increases, indicating that the quencher (TMPD^{+2}) accelerates the decay of an electronically excited state by electron transfer quenching.

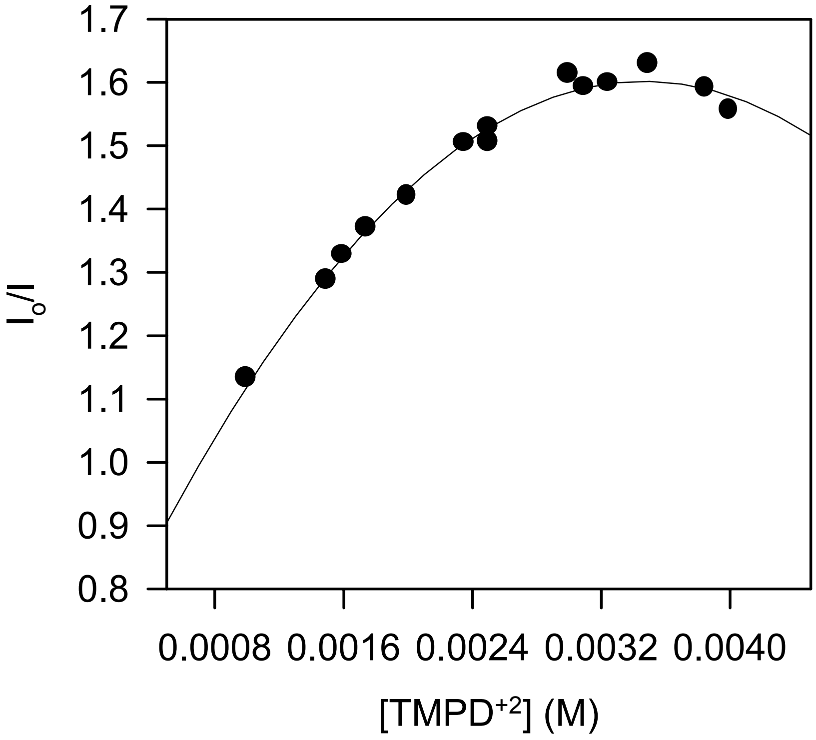

Figure 3: Stern –Volmer Plot of the luminescence quenching of [Ru (dpte) $_2$(dcppm)]$^{+2}$ (5x10^{-5}M) by TMPD^{+2}.

Luminescence Quenching of Ru (II)-Complexes by MV^{+2}

In Table (2) the Stern- Volmer constants (k$_{sv}$) are shown. The quenching of all of the complexes show a higher value of k$_{sv}$ in basic media than in acidic media. This behavior indicates that the basic media stabilizes the radical $MV^{+\cdot}$, and the acidic media protonates the nitrogen of the diimine ligands, so the quenching of the $Ru^*(II)$ is prevented and the electron transfer to the MV^{+2} to form $MV^{+\cdot}$ becomes slow.

$[Ru\,(dpte)_2(dcmppm)]^{+2}$ has the highest value of k_{SV} in acidic media compared to the other complexes. This behavior can be attributed to the presence of methyl group at the ligand which act as an electron donor, this will reduce the deficiency of electron on the ligand due to the protonation of the nitrogen in the diimine ligands in acidic media.

Luminescence Quenching of Ru (II)-Complexes by EDTA

In Table (3) the Stern-Volmer constants (k_{sv}) are shown. The quenching of all complexes shows a higher value of k_{sv} in basic media than in acidic one, indicating that basic media enhance the formation of Ru (I) and $EDTA^+_{ox}$ (R_2-N- $(CH)_2$-N^+-R_2), also in basic media EDTA present in (-3/-4) anion, so it enhance the reductive quenching between Ru (II) and EDTA, as follow:

$$\mathbf{Ru^*(II) + EDTA \longrightarrow Ru\,(I) + EDTA^+_{ox}} \qquad (2)$$

$$\mathbf{EDTA^+_{ox} + OH^- \longrightarrow EDTA'\;(R_2\text{-}N\text{-}\,CH2C^{\cdot}H\text{-}N\text{-}R_2)} \qquad (3)$$

$$\mathbf{EDTA' + Ru\,(II) \longrightarrow Ru\,(I) + Product} \qquad (4)$$

At pH 4, EDTA becomes protonated and quenched by Ru^* (II) slower than at pH 10, more over the protonated form of the excited state of Ru (II), exhibits a shorter life time and smaller quenching rate constant than does the un-protonated form. The Stern – Volmer plots of all complexes curved up or down at high concentration, due to presence of static and dynamic quenching at the same time.

Effect of ionic strength and pH on Luminescence Quenching of Ru-(II)-Complexes

The reductive quenching of ruthenium complexes by EDTA is known to produce Ru(I) and $EDTA^+$.[2] The quenching rate constant (k_q) derived from the slope (k_{sv}) of Stern-Volmer plot depends on pH and ionic strength, this shown in Tables (4-5). In the range of ionic strength used (Table 4), k_q decreases by a bout 21%. Since the study was done at a pH of 11.0, EDTA mostly exists as the triply or quadruply negatively charged species ($EDTA^{3-}$ and $EDTA^{4-}$). Under these conditions EDTA quenches very effectively Ru(II) complexes and produces Ru(I). The effect of ionic strength on k_q has been attributed to ion-pairing and aggregation which should drastically affect diffusion of the species and reduce quenching.

The salt effects for diffusion controlled reaction can be fitted by Bronsted –Deby equation.[24,25]

$$\mathbf{Log\ k = log\ k_o + z_1\, z_2\, A\, \mu^{1/2} /(1+B\, \mu^{1/2})} \qquad \mathbf{(5)}$$

Where, k: Quenching rate constant, k_o: Quenching rate constant at zero ionic strength. z: Valances of reactants, μ: Ionic strength, A and B: Constants of the theory.

The effect of pH on Luminescence quenching is shown in Table 5. these results show that basic media increases k_q but the effect is not linear. In acidic media, protonation of EDTA makes electron donation more difficult and hence electron-transfer quenching is reduced. In addition the possibility of protonation of the non-bonding nitrogen of dcpppm cannot be ignored.

The drastic reduction in lifetimes (τ_o) at a pH below 6 indicates protonation of the dcpppm ligand which modifies the excited state behavior and enhances its relaxation to the ground state. This process competes with quenching and reduces its efficiency.

Conclusions

The effect of ionic strength and acidity on the Luminescence quenching of the excited states of a new synthesized mixed-ligand Ru(II) complexes have been studied. The results of this study emphasize that the quenching rate constant increases with decreasing the ionic strength, by contrast the pH has the opposite effect. Furthermore, in this study it has been shown that the quenching of mixed-ligand Ru(II) complexes by $TMPD^{2+}$ in aqueous solutions to be dynamic and static in nature.

Experimental

Chemicals. All complexes were prepared and purified according to standard procedure[26-30] with slight modification.[3] The ligands were obtained from Fluka AG and LABORAT GMBH (Berlin West, Germany). Methylviologen dichloride (MV^{+2}) was obtained from Aldrich and purified by precipitating from methanol using ether. Na_2EDTA were obtained from Fluka AR and used without further purification. $TMPD^{+2}$ was obtained from Aldrich and was purified by vacuum sublimation, then the product was dissolved in methanol and precipitation by ether was done. Water was first distilled, deionized and redistilled from $KMnO_4$ then used as a solvent in the preparation of the solutions. All solvents used in the physical measurements were spectroscopic or HPLC grade.

*Quenching***.** Air-saturated fluorescence quenching data were obtained from the emission intensity at the maximum emission wavelength on a Perkins-Elmer MPF-44B spectroflurometer. All measurements were made in 1-cm cells at room temperature. NaOH and HCl were used to control the pH, and Na_2SO_4 was used for controlling the ionic strength. Lifetimes were measured using an Edinburgh Instruments model 199M photon counting system.

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Povzetek

Proučevan je bil vpliv ionske moči in kislosti na gašenje luminiscence novo sintetiziranih kompleksov mešanih ligandov z rutenijem(II). Za študij vpliva kislosti so bili izbrani *N,N,N′,N′*-tetrametil-*p*-fenilen-diamin ($TMPD^{2+}$), metil viologen (Mv^{2+}) in etilenediaminetetraocetna kislina (EDTA, za študij vpliva ionske jakosti pa system Ru(II)/EDTA. Konstanta hitrosti gašenja (k_q) narašča s padajočo ionsko močjo, medtem ko ima p*H* nasprotni učinek.