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## Luminescence quenching studies of $[\text{Ru}(\text{dMeObpy})_3]^{2+}$ complex using the quinone derivative-effect of micelles

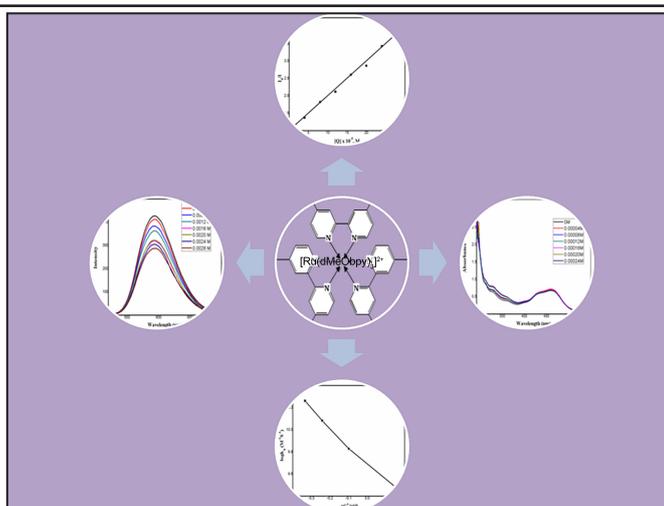
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### HIGHLIGHTS

- Ru(II) polypyridyl complex undergoes oxidative quenching with quinones.
- There is an enhancement of luminous intensity, and the complex has a longer lifetime in the presence of micelle.
- In the presence of CTAB, the hydrophobic interaction is predominant over electrostatic repulsion.
- Transient absorption spectra confirm the formation of  $\text{Ru}^{3+}$  species and quinone anion radicals.

### GRAPHICAL ABSTRACT



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### ABSTRACT

Quinones are considered a class of organic compounds having a quinonoid group and are the ultimate electron acceptors. Due to this property, they have favourable redox potential and the ability to form stable hydrogen bonds. Luminescence quenching is one of the most important techniques used to get information regarding the structure and dynamics of a luminophore. A variety of transition metal complexes have been synthesized and studied to comprehend the quinones' electron-accepting characteristics. Among these, Ru(II) polypyridyl complexes have widespread applications in electron transfer reactions due to their well-defined photophysical and photochemical stability. The reaction of excited state Tris(4,4'-dimethoxy-2,2'-bipyridine) ruthenium(II)tetrafluoroborate  $[\text{Ru}(\text{dMeObpy})_3](\text{BF}_4)_2$  complex with quinones was investigated through photoinduced electron transfer reaction in homogeneous and microheterogeneous medium. The luminescence quenching technique has been used to study this reaction. The complex has an absorbance maximum of 448 nm in aqueous medium. The quenching rate constants were deduced using the Stern-Volmer equation. The interaction between the complex and the quinones in a cationic micellar medium, cetyltrimethylammonium bromide (CTAB), was analyzed based on electrostatic interaction and hydrophobicity. The plot between  $R\ln k_q$  vs. reduction potential of the quinones, as well as the transient absorption spectra, confirmed the oxidative nature of quenching of the ruthenium complex in the presence of quinones. The quenching constant values are influenced by many factors, such as the nature of the ligand, medium, size, and structure of quenchers, and electron transfer distance between the donor and the acceptor. The formation of  $\text{Ru}^{3+}$  species is confirmed by its characteristic absorption at 600 nm.

## 1. Introduction

Quinones are a class of organic compounds having a quinonoid group. In the process of photosynthesis, they serve as electron acceptors and are considered an important functional moiety in various biological systems [1-3]. Their electron-accepting property is due to (i) possessing favourable redox potential, (ii) being able to be reduced to stable compounds like hydroquinones and semiquinones, and (iii) having the ability to form hydrogen bonds. The electron-accepting nature of quinones can be understood using newly designed photosensitive complexes. In addition, several research groups have reported quinone electron transfer reactions [4-7].

Microheterogeneous systems, a type of aqueous micelle-bound catalytic system, are an appealing alternative to toxic, expensive chemical solvents. They are a prelude to future surfactant assembly designs that may imitate redox processes in biological membranes. Micelles are extensively documented as a unique habitat for photochemical transfiguration because these micellar structures can increase electron transfer from electron donor to acceptor by isolating them. Micelles, made up of a hydrophobic fluid phase and an electrostatic interphase, provide an ideal habitat. Micelles, which are supramolecular accumulations resembling biosystems, change the photophysical and photochemical behavior of the probe molecules. These accumulations promote the incorporation or restriction of molecules in the micelle core or at the micelle interface through hydrophobic and electrostatic interactions.<sup>8</sup> The photoinduced electron transfer (PET) reaction between a donor and an acceptor in a solution is known to be strongly affected by various factors. It has been shown that medium effects such as the polarity of the liquid, the heterogeneity of the microenvironment, and the addition of salt will influence the dynamics of the electron transfer reactions. Transition metal complexes and detergents are still interesting because they are used to convert energy, speed up chemical reactions, and mark low-concentration species for study. Ionic Ru(II) polypyridyl complexes with hydrophobic ligands can be synthesized, allowing for binding to critical interfacial areas in microheterogeneous systems.

Ruthenium(II) polypyridyl complexes are also used as probes in micellar medium, and their properties vary with the nature of the surfactant. These organized media can promote electron transfer from the electron donor to the acceptor by concentrating them.<sup>9</sup> The photophysical characteristics of the Ru(II)-polypyridyl complexes  $[\text{Ru}(\text{NN})_3]^{2+}$ , frequently used as probes in micellar environments,<sup>9</sup> vary substantially depending on the surfactant type and concentration. Furthermore, the existence of micelles significantly impacts the kinetics of chemical processes. To influence

the kinetics of micellar solutions, micelles and reactants interact electrostatically and hydrophobically. Ruthenium(II) complexes have drawn considerable interest because they can undergo oxidation and reduction processes in the presence of visible light irradiation. These benefits can be used in a wide variety of applications, such as the photocatalytic breakdown of water or the development of solar systems [10].

Due to the light-sensitizing properties of ruthenium coordination compounds, these compounds have also been applied in the field of chemosensors [11]. These luminescent complexes have applications such as photocatalyst [12-14], sensors for biomolecular [15-17], phototherapeutic agents [18,19]. Moreover, these polypyridyl complexes are excellent photosensitizers from the viewpoint of luminescence and electrochemical properties, which have been extensively investigated for applications in inorganic and material chemistry [20]. Luminescence quenching is one of the most important techniques used to get a lot of information regarding the structure and dynamics of a luminophore. The intensity of the luminous molecules is reduced by a multiplicity of interactions among molecules, such as molecular rearrangement, excited state processes, ground state complex creation, electron and energy transfer, and so on. The quenching reactions of  $[\text{Ru}(\text{dMeObpy})_3]^{2+}$  complex with quinones in the presence of cationic micelle CTAB are discussed in this study. The formation of a quinone anion radical proved the reaction's electron transfer character. It was further supported by the emergence of  $\text{Ru}^{3+}$  transient species.

## 2. Experimental

### 2.1. Materials and methods

$\text{RuCl}_3 \cdot 3\text{H}_2\text{O}$ , ligand 4,4'-dimethoxy-2,2'-bipyridine (dMeObpy), and the quenchers were purchased from Sigma Aldrich. CTAB was procured from the Merck company. Studies on quenching use double-distilled water as the medium. According to the previously described procedure, the complex was prepared by reacting  $\text{RuCl}_3 \cdot 3\text{H}_2\text{O}$  with the corresponding ligand [21]. The resulting complex's chloride salt was treated with sodium tetrafluoro borate to produce the  $[\text{Ru}(\text{dMeObpy})_3][\text{BF}_4]_2$  complex. All the reactants were freshly prepared for each measurement. The complex concentration was kept constant at  $4 \times 10^{-5}$  M. The concentrations of the quenchers varied between  $4 \times 10^{-6}$  M to  $2.8 \times 10^{-7}$  M.

### 2.2. Instrumentation

A SYSTRONICS 2203 UV visible double-beam spectro-

photometer was used to measure the absorbance. A JASCO FP 8200 spectrofluorometer was used to record the emission measurements. Electrochemical measurements were taken using a cyclic voltammeter. The sample solutions were deaerated using  $N_2$  dry nitrogen purging for about 30 min to measure the emission and lifetime of the complex. The solution was kept in cold water to ensure that there was no volume change.

All spectral observations were performed at room temperature. Transient absorption spectra were acquired by utilizing a point-to-point technique to measure the absorbance changes (A) following the flash at intervals of 10 nm across the spectral range of 300 to 700 nm, averaging at least 30 decays at each wavelength. A Czerny-Turner monochromator controlled by a stepper motor and a Hamamatsu R-928 photomultiplier tube was used to investigate the rate of light decrease over time. Absorbance shifts were monitored after being exposed to 355 nm, which was employed to produce an excited state.

### 2.3. Synthesis of Tris(4,4'-dimethoxy-2,2'-bipyridine) ruthenium(II)tetrafluoroborate, $[Ru(dMeObpy)_3][BF_4]_2$

20 ml of ethylene glycol were used to dissolve  $RuCl_3 \cdot 3H_2O$  (1 mM) and 4,4'-dimethoxy-2,2'-bipyridine (3 mM) before refluxing for 4 h. After cooling to normal temperature, the solution was filtered to eliminate any insoluble contaminants. Next, the filtrate was added dropwise to a saturated sodium tetrafluoroborate solution until an orange precipitate formed. The last drying step involved a vacuum desiccator after the product had been filtered and rinsed with cold water and diethyl ether. Recrystallization was used to further purify the final product. The complex had maximum absorption ( $\lambda_{abs}^{max}$ ) and emission ( $\lambda_{em}^{max}$ ) wavelengths of 448 and 595 nm, respectively, in aqueous medium [22].

### 3. Results and discussion

Fig. 1 shows the structure of the quenchers and CTAB employed in this work. The absorption spectrum of the Tris(4,4'-dimethoxy-2,2'-bipyridine) ruthenium(II)tetrafluoroborate  $[Ru(dMeObpy)_3][BF_4]_2$  complex in CTAB medium shows a strong ligand-centred (LC) $_{\pi-\pi^*}$  transition in the region 260 to 280 nm. At 462 nm, there is a low energy absorption peak. This region is assigned to the  $d_{\pi-\pi^*}$  MLCT transition in the CTAB medium. The complex shows an absorption maximum of 448 nm in aqueous medium. Thus, the absorption maximum of MLCT undergoes a bathochromic shift by 14 nm, as shown in Table 1. The change in medium (CTAB) also affects the absorption maximum of the complex.

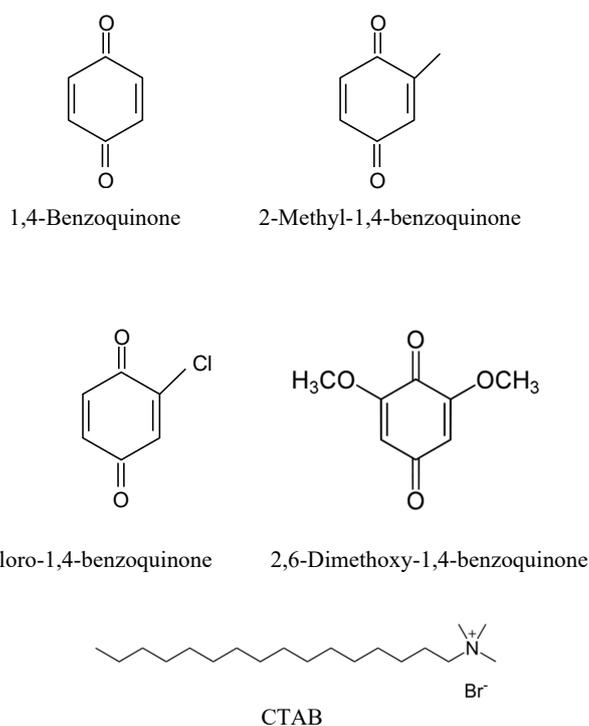


Fig. 1. Structure of Quinones and CTAB.

Thus, the presence of micelle lowers the energy level of the  $^3MLCT$  state and stabilizes it relative to the  $d-d$  state.

The structure of CTAB ( $[(C_{16}H_{33})N(CH_3)_3]Br$ ) is shown in Fig. 1. As per the literature survey, the cationic surfactant CTAB has a longer chain structure than any other surfactant and is composed of a 16-carbon long tail attached with three methyl groups in an ammonium head group. For the cationic micelles (CTAB), the hydrophobic interactions of the CTAB are predominant over electrostatic repulsion. So, CTAB micelle interaction with these complexes must arise from the hydrophobic effect of the photosensitizer ligands in the alkyl core of the complex. There is a slight change in the emission intensity of the Ru(II) complex with increasing concentration of CTAB. These observations substantiate the importance of hydrophobic interactions between the ligands of the probe molecule and the cationic micelle.

Some photophysical properties, such as the absorption and emission maxima and the excited state lifetime ( $\tau$ ) of the Ru(II) complex in aqueous and CTAB media, are tallied and quantified in Table 1.

Table 1. Absorption and emission spectral data and excited state lifetime of  $[Ru(dMeObpy)_3]^{2+}$  complex in aqueous and CTAB media.

Absorption maximum (nm)		Emission maximum (nm)		Excited state lifetime (ns)	
Aqueous	CTAB	Aqueous	CTAB	Aqueous	CTAB
448	462	595	596	232	253

As shown in the table, upon photoexcitation using light of wavelength 462 nm, the complex's fluorescence spectrum exhibited an emission band at 596 nm in the CTAB medium, and the complex has an emission maximum of 595 nm in aqueous medium, showing changes in the complex's intensity and emission maximum. The lifetime of the complex was also measured in both aqueous and CTAB medium, and as the table shows, there is an increase in the lifetime of the complex in the CTAB medium. These changes confirm that the hydrophobic interaction between the ligands and the cationic surfaces overcomes the electrostatic repulsive forces; therefore, they stabilize the <sup>3</sup>MLCT state of the metal complex [23]. The emission spectrum of the complex in CTAB and aqueous medium is displayed in Fig. 2.

The photochemical oxidation of  $[\text{Ru}(\text{dMeObpy})_3]^{2+}$  complex with increment addition of quinones was studied using the luminescence spectral technique. The absorption spectrum of  $[\text{Ru}(\text{NN})_3]^{2+}$  complexes in CTAB is shown in Fig. 3. The complex concentration was fixed at  $4 \times 10^{-5}$  M, and the quencher concentration varied from 0 to 0.00024 M. The absorption spectrum of  $[\text{Ru}(\text{dMeObpy})_3]^{2+}$  with an incremental concentration of 1,4-benzoquinone in the CTAB medium shows a formation of ground state complex, indicating that there may be static quenching.

Fig. 4 displays the emission spectra of the complex with changes in quencher concentration in the CTAB medium recorded at 298 K. The concentration of the quencher varies from 0 to 0.0028 M. As can be seen, there is a steady decrease in the emission intensity. This decrease in fluorescence intensity shows that quenching has taken place.

### 3.1. Stern Volmer Analysis

The Stern-Volmer equation was used to determine the

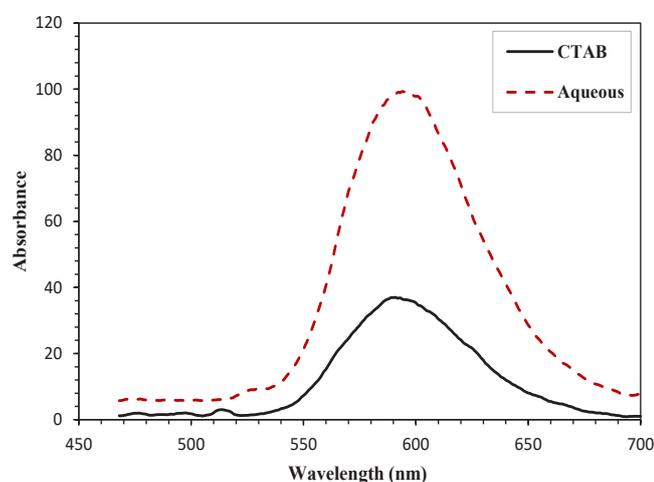


Fig. 2. Emission spectrum of  $[\text{Ru}(\text{dMeObpy})_3]^{2+}$  complexes in aqueous and CTAB medium.

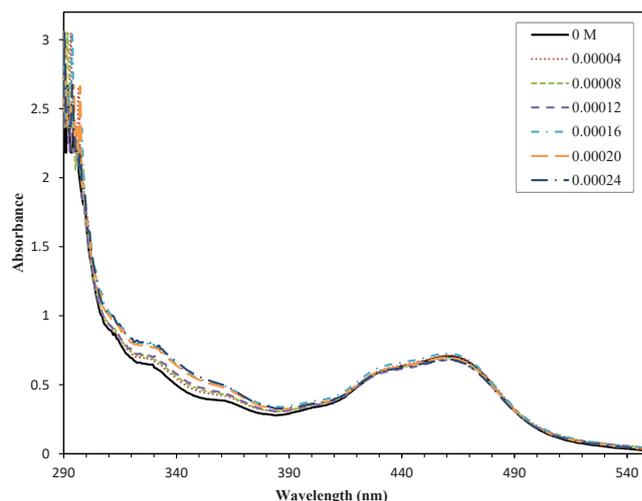


Fig. 3. The absorption spectrum of  $[\text{Ru}(\text{dMeObpy})_3]^{2+}$  with an incremental concentration of 2,6-dimethyl-1,4-benzoquinone in CTAB medium.

reaction's quenching rate constant ( $k_q$ ) using the emission intensity data (Eq. 1) [24].

$$I_0/I = 1 + k_q \tau [Q] \quad (1)$$

where  $I_0$  and  $I$  represent the intensity of emission in the absence and presence of quenchers, respectively, and  $\tau$  denotes the emission lifetime of the complex in the absence of the quencher. The lifetime of the complex was measured using a nanosecond flash photolysis spectrophotometer. The quenching rate constant was calculated graphically from the slope of the plot between  $I_0/I$  vs.  $[Q]$ . The Stern-Volmer plot for the electron transfer reaction of the  $^*[\text{Ru}(\text{dMeObpy})_3]^{2+}$  complex in the presence of the quencher 1,4-benzoquinone is shown in Fig. 5.

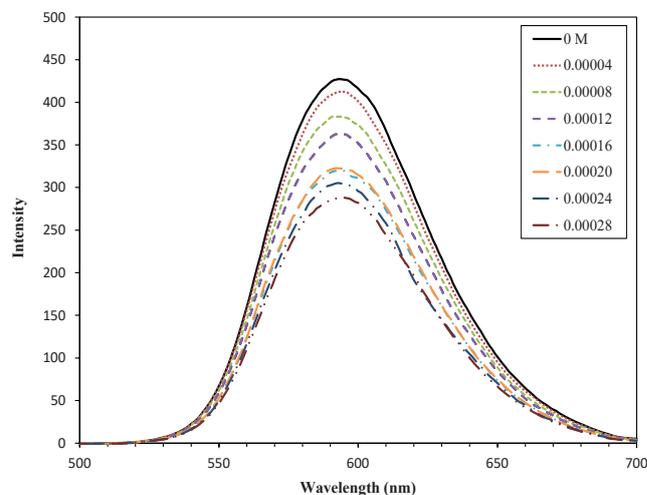
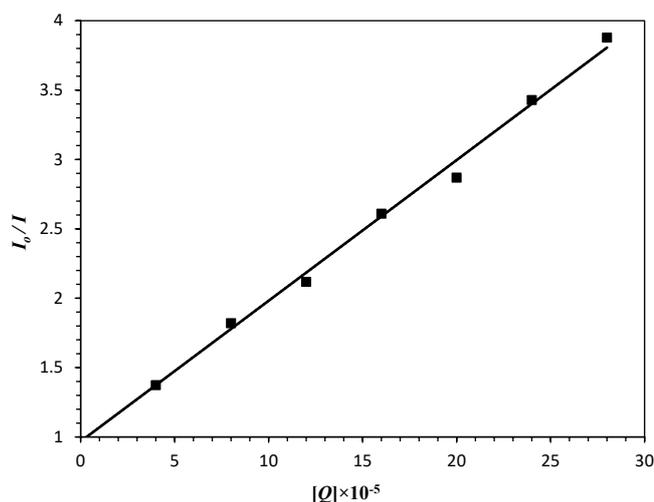


Fig. 4. Emission spectrum of  $[\text{Ru}(\text{dMeObpy})_3]^{2+}$  with an incremental addition of 2-chloro-1,4-benzoquinone in CTAB medium..



**Fig. 5.** Stern-Volmer plot for the luminescence quenching of  $[\text{Ru}(\text{dMeObpy})_3]^{2+}$  with 1,4-benzoquinone in aqueous medium.

### 3.2. Redox potential

Metal polypyridyl complexes show redox properties. Information on the ground state oxidation/reduction potential is essential for developing potential applications. This information is readily obtained from the simple electrochemical measurements of the redox potential and reversibility of oxidation and reduction waves. These studies allow the estimation of redox properties of the excited state. The oxidation potential of 1.1 V for the complex in the CTAB medium was recorded for the ground state in the presence of an  $\text{Ag}/\text{Ag}^+$  electrode. The excited state potential was obtained from the corresponding ground state redox potential and the spectroscopic excited state energy  $E_{0,0}$  (found to be 2.1 eV) [22]. The excited state oxidation potential of the complex was found to be -1.0V vs.  $\text{Ag}/\text{Ag}^+$ . The free energy change ( $\Delta G^0$ ) values are calculated from the values of the excited state oxidation potential of the  $[\text{Ru}(\text{dMeObpy})_3]^{2+}$  complex and the reduction potential of the quencher. The experimental bimolecular quenching constant reduction potential of the quenchers in aqueous and CTAB medium  $\Delta G^0$  values are given in Table 2.

### 3.3. Luminescence quenching studies

Even though the Stern-Volmer plot is linear, the absorption maximum of the complex exhibits slight changes. This shift indicates the formation of a ground state complex, which demonstrates the complex's quenching with 1,4-benzoquinone is static. Absorption spectrum measurements and the matching association constant values in aqueous and CTAB media confirm static quenching. The shifts in emission maxima show that the hydrophobic contact between the ligands and the cationic surfactant overrides the repulsive forces. These spectral alterations confirm the ruthenium(II) complex's binding to the micelle. The cationic micelle changes the excited state characteristics of the complex, and it remains in the aqueous phase due to coulombic repulsion from the cationic micellar medium.

When compared to the constant value for the aqueous medium, the value for the quenching rate constant goes down. The calculated free energy change ( $\Delta G^0$ ) in an aqueous medium shows that 2-chloro-1,4-benzoquinone has the highest reduction potential value and the lowest quenching rate constant of  $1.943 \times 10^{10}$  in aqueous medium. The quenching rate constant is greater in the aqueous media than in the CTAB medium. Based on these findings, it is argued that the hydrophobic contact between the probe's ligands and the cationic micelle has compensated for the electrostatic repulsion between the luminophore and the micelle.

The Benesi-Hildebrand plots from the absorption spectral data of  $[\text{Ru}(\text{dMeObpy})_3]^{2+}$  complexes with 2-methyl-1,4-benzoquinone are shown in Fig. 6. Photoluminescence studies have shown that the binding between  $[\text{Ru}(\text{NN})_3]^{2+}$  and CTAB micelles is probably Vander Waals or hydrophobic in nature. In addition to the nature of the substituent present in the complex and quencher, the ET distance between the reactants also affects the value of the bimolecular quenching constant  $k_q$ . The first-order rate constant ( $k_{red}$ ) values for the quinones are in the order of  $10^4$ - $10^5$  s<sup>-1</sup>. Binding constant ( $K_b$ ) values are in the order of  $10^4$ - $10^5$  M<sup>-1</sup>. Previtali has reported the effect of solvent on the quenching of  $[\text{Ru}(\text{NN})_3]^{2+}$

**Table 2.** The quenching rate constant ( $k_q$ ), the redox potential of the quinones vs.  $\text{Ag}/\text{Ag}^+$ , and free energy change for the oxidative quenching of  $[\text{Ru}(\text{dMeObpy})_3]^{2+}$  with the quenches..

Quencher	$k_q$ (M <sup>-1</sup> S <sup>-1</sup> )		$E_{red}^0$ vs $\text{Ag}/\text{Ag}^+$ (V)	$\Delta G^0$ (eV)
	Aqueous medium	CTAB		
2-Chloro-1,4-benzoquinone	$1.943 \times 10^{10}$	$1.830 \times 10^{10}$	-1.289	0.334
1,4-Benzoquinone	$3.558 \times 10^{10}$	$1.213 \times 10^{10}$	-1.207	0.242
2-Methyl-1,4-benzoquinone	$1.125 \times 10^{10}$	$6.750 \times 10^9$	-1.054	0.099
2,6-Dimethoxy-1,4-benzoquinone	$5.470 \times 10^{10}$	$3.020 \times 10^9$	-0.801	-0.154

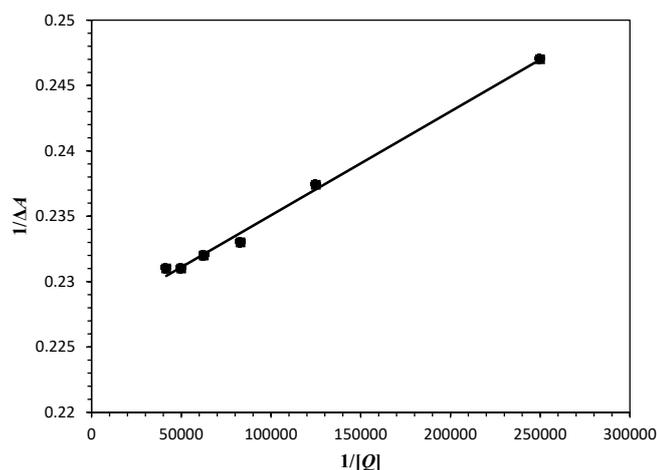


Fig. 6. The Benesi–Hildebrand plot from the absorption spectral data of  $^*[Ru(dMeObpy)_3]^{2+}$  with 2-methyl-1,4-benzoquinone in CTAB media.

complex by aromatic amines and methanol [25].

The data in Table 2 indicate that the substituents in the quencher affect the bimolecular quenching rate constant.  $\log k_q$  is plotted against  $\Delta G^0$  to investigate the effect of  $\Delta G^0$  on the reaction's bimolecular quenching constant. Fig. 7 depicts the plot, which has an intriguing shape with the  $k_q$  value first increasing as the exoergicity of the response ( $\Delta G^0$ ) increases. It approaches a maximum, but increasing  $\Delta G^0$  further decreases the value of  $k_q$ . The plot may lead us to believe the Marcus inverted zone was observed in the photoinduced ET reaction.

The linear plot of  $RT \ln k_q$  vs. reduction potential of the quinones (Fig. 8) provides us with additional support for the oxidative quenching of the  $[Ru(dMeObpy)_3]^{2+}$  complex.

### 3.4. Transient absorption spectral measurement

The transient absorption spectrum was used to confirm the oxidative nature of the quenching of ruthenium(II) polypyridyl complexes. The Ru(II) complex solution in the

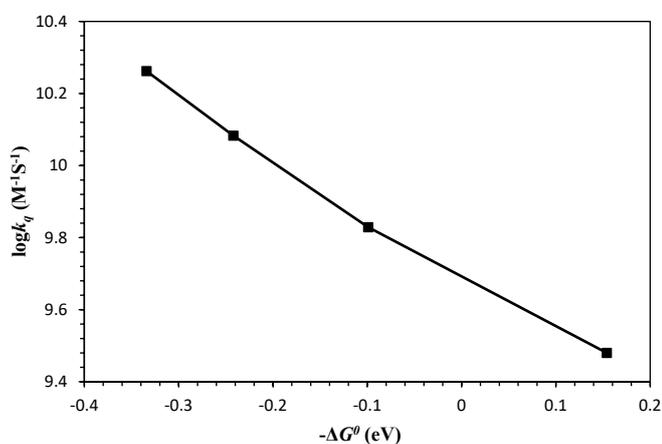


Fig. 7. The plot of  $\log k_q$  vs.  $\Delta G^0$  for different quinones.

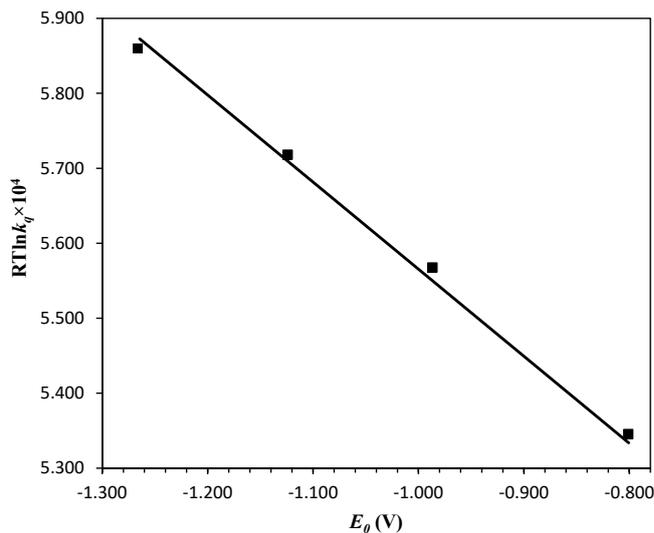


Fig. 8. The plot of  $RT \ln k_q$  vs. reduction potential of quinones in micellar medium.

CTAB medium was agitated with Argon gas and stimulated with laser flash photolysis at 355 nm. The static transient absorption spectrum of  $^*[Ru(dMeObpy)_3]^{2+}$  in CTAB medium at 100 ns in the presence and absence of the quencher 2,6-dimethyl-1,4-benzoquinone is shown in Fig. 9. As can be seen, it has a bleach near 450 nm and an enhanced absorption with a maximum near 370 nm. This is due to the existence of the modified bipyridyl anion radical. The spectrum at each time delay consists of bleach around 400 nm due to the loss of the ground state absorption,  $d_{\pi-\pi^*}$  (MLCT) transition. The bleach at 600-700 nm shows the relaxation of the excited state to the ground state. The production of  $Ru^{3+}$  ions and the formation of quinone anion radicals indicate the oxidative quenching of  $[Ru(dMeObpy)_3]^{2+}$  complex with quinones in the presence of cationic micellar medium, CTAB. The data

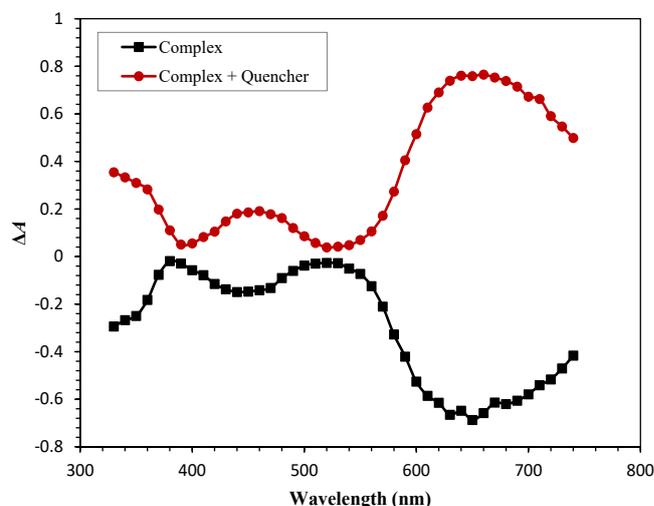


Fig. 9. Transient absorption spectra of CTAB solution of  $[Ru(dMeObpy)_3]^{2+}$  complex in the absence and presence of 2,6-dimethyl-1,4-benzoquinone at 100 ns.

in Table 2 show that quinones that possess greater reduction potential will have larger quenching rate constants, indicating the electron transfer quenching. The quinones' oxidative quenching is confirmed by their transient absorption spectra and the exponential value of  $\log k_q$  vs. reduction potential.

#### 4. Conclusion

The current study establishes the luminescence quenching of  $[\text{Ru}(\text{dMeObpy})_3]^{2+}$  photoredox reaction with quinones in aqueous and CTAB medium. The existence of hydrophobic interaction causes the medium to change from homogeneous to microheterogeneous, which has a major impact on the  $k_q$ . The bimolecular quenching constant values are affected by the reduction potential of the quinones, the solvent medium, the free energy change, and the electron transfer distance between the donor and acceptor. The study also confirms the structural effect on the electron transfer reaction of quinones with the excited state  $^*[\text{Ru}(\text{dMeObpy})_3]^{2+}$  complex. Both the transient absorption spectra and the linear plot of  $RT \ln k_q$  versus the reduction potential of the quinones provide evidence that the quenching process is oxidative in character. In the majority of cases, electrostatic repulsion between the micellar particles has been eliminated by hydrophobic contact. The majority of the quenchers that were being used exhibited this behavior quite obviously. To achieve a complete comprehension of this micellar catalysis phenomenon, additional study is being carried out.

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#### Disclosure statement

The authors certify that they have no commercial or associational interests that could pose a conflict of interest in relation to the submitted work.

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