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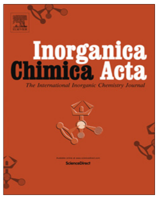

Synthesis, photophysical properties and electrochemiluminescence performances of a series of cationic iridium(III) complexes

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ABSTRACT

A series cationic cyclometalated iridium(III) complexes, ([Ir(pq)$_2$(bpy)][PF$_6$] (**1**), [Ir(dpq)$_2$(bpy)[PF$_6$] (**2**), [Ir(pqcm)$_2$(bpy)][PF$_6$] (**3**), [Ir(pqca)$_2$(bpy)][PF$_6$] (**4**), pqH = 2-phenyl-quinoline, dpqH = 2,4-diphenyl-quinoline, pqcmH = 2-phenyl-quinoline-4-carboxylic acid methyl ester, pqcaH = 2-phenyl-quinoline-4-carboxylic acid adamantanemethyl ester, bpy = 2,2′-bipyridine), have been synthesized. New iridium(III) complexes had been characterized by the NMR and mass spectroscopies, and the single crystal structure of complex **4** had been measured. In CH_3CN solution, the iridium(III) complexes **1** and **2** showed yellow emission at 560 nm, while the iridium(III) complexes **3** and **4** displayed pure red emission at 620 nm. The iridium(III) complexes had quantum efficiencies between 1.1% and 8.5%. The electrochemiluminescence (ECL) of cationic cyclometalated iridium complexes was investigated in CH_3CN/H_2O (40:60 (v/v)) with Au electrode as working electrode. The ECL peaks were obtained when tri-*n*-propylamine was used as co-reactant and positive initial potential scan direction was adopted. The ECL intensity of iridium(III) complex **1** is 3.8 times of $Ru(bpy)_3^{2+}$. The phosphorescence quantum efficiencies of complex **1** and $Ru(bpy)_3^{2+}$ are close, so the better ECL performance suggests that iridium complexes have great application potential in ECL detection. The ECL intensities of iridium(III) complexes had almost the same sequence with the quantum efficiencies of iridium(III) complexes.

1. Introduction

Electrochemiluminescence (ECL) has been paid considerable attention during the past several decades because it offers lower background noise, higher detection sensitivity, a wide dynamic linear range and requires simple and inexpensive instrumentation in comparison with other methods [1]. $[Ru(bpy)_3]^{2+}$ has become the most thoroughly studied ECL active molecule in both aqueous and nonaqueous solution since the first report in 1972 by Bard [2]. Recently, cyclometalated Ir(III) complexes used in OLED (organic light emitting devices) have been studied as new ECL reagents. Since these materials have high photoluminescence efficiencies compared to $[Ru(bpy)_3]^{2+}$ ($\Phi_{em} = 0.062$) [3]. Beyond that, cyclometalated iridium(III) complexes have unique photophysical properties, such as relatively long lifetime compared to purely organic luminophores, significant Stokes shift for easy separation of excitation and emission, excellent color tuning and stable redox characteristics [4]. The iridium complex (pq)$_2$Ir(acac), for example, was reported to have an ECL efficiency 77 times that of $[Ru(bpy)_3]^{2+}$ when TPA was used as the coreactant [5]. Although many neutral complexes of iridium(III) are known to be ECL active in a nonaqueous environment, often with a degree of efficiency much higher than $[Ru(bpy)_3]^{2+}$, their applications have been restricted because of their lower solubility in aqueous solutions and sensitivity to oxygen quenching [6]. On the other hand, charged iridium(III) cyclometalated polypyridine complexes have favorable solubility in aqueous solutions, comparable electrochemiluminescent intensity to that of $[Ru(bpy)_3]^{2+}$, and hold great promise as ECL-active probes in biological tagging for proteins, peptides, amino acids, antibodies, and oligonucleotides [7–12], provided that they may be easily functionalized by useful linkers for bioconjugation. By changing the structure of the ligand, one can modulate the HOMO and LUMO energies of charged Ir(III) complexes in order to tune the emission color and luminous efficiency [13–16]. In our previous work, it was found that the cyclometalated iridium(III) complex with 2,2′-bipyridine assistant ligands has much higher photoluminescence and ECL efficiencies than the complexes with the other N^N typical assistant ligands [17].

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In the present paper, different 4-substituents were introduced into the phenylquinoline ligands of cationic iridium(III) complexes in order to tune their photophysical properties. The synthesis and photophysical properties of the complexes were investigated. The electrochemical and ECL properties of these complexes were also studied in mixed solvents (40:60 (v/v) CH_3CN/H_2O).

2. Experimental

2.1. General

The solvents were purified by routine procedures and distilled under an atmosphere of dry nitrogen before use. All reagents, unless otherwise specified, were purchased from China Reagent and were used as received. NMR spectra were recorded on a Bruker AV400 spectrometer using $CDCl_3$ as solvent. Mass spectra were recorded on a Perkin Elmer Sciex API 365 mass spectrometer and a Shimadzu AXIMA-CFR plus MALDI-TOF mass spectrometer. UV–Vis absorption spectra were recorded on a Tu-1901 PC spectrophotometer. Photoluminescence (PL) spectra were measured with a LS55 fluorescence spectrophotometer.

2.2. Synthesis of iridium complexes

The synthetic route to iridium(III) complexes is given in Scheme 1. Iridium(III) complexes, **2** and **3** were synthesized by routine procedures according to related articles [17,18].

The four complexes were synthesized in two steps using standard method [17].

To a round-bottomed flask (25 mL), 2-ethoxyethanol (9 mL), 2-phenylquinoline (0.41 g, 2.0 mmol), $IrCl_3{\cdot}3H2O$ (0.20 g, 0.56 mmol) and water (3 mL) were added sequentially. The mixture was stirred under nitrogen at 120 °C for 24 h and cooled to room temperature. The precipitate was collected and washed with ethanol and acetone, and then dried in vacuum to give a red dimer ($[\{Ir(\mu\text{-}Cl)(pq)_2\}_2]$) (0.32 g, 0.25 mmol).

In a round-bottomed flask, 0.06 g (0.05 mmol) of $[\{Ir(\mu\text{-}Cl)(pq)_2\}_2]$ and 0.03 g (0.20 mmol) of 2,2′-bipyridine (bpy) were mixed together in 10 mL of CH_2Cl_2. The solution was then stirred at room temperature for 6 h. To the solution was added a methanol solution with an excess of potassium hexafluorophosphate for the ion exchange. The resulting product was filtered, washed with absolute ethanol and dried. The product was purified by chromatography on a silica gel column using dichloromethane/ethyl acetate (v/v = 2:1) as eluent. A yellow solid of **1** was obtained (79 mg, 81% yield).

^1H NMR ($CDCl_3$, 400 MHz) δ: 9.02 (d, J = 8.0 Hz, 1H), 8.93 (d, J = 4.0 Hz, 1H), 8.29 (t, J = 8.0 Hz, 2H), 8.20 (d, J = 8.0 Hz, 1H), 8.14 (d, J = 5.2 Hz, 1H), 8.01 (d, J = 8.0 Hz, 1H), 7.02–7.75 (dd, J = 5.6, 8.0 Hz, 2H), 7.38 (d, J = 6.8 Hz, 2H), 7.25 (d, J = 7.6 Hz, 1H), 6.32 (t, J = 6.8 Hz, 1H), 6.65 (d, J = 8.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 169.24, 165.07, 154.53, 150.89, 148.74, 147.34, 142.08, 140.00, 134.70, 131.47, 131.00, 129.12, 127.54, 127.11, 126.96, 126.38, 124.82, 124.66, 123.06, 117.34. MADIL-TOF MS (m/z): Calc. for $C_{46}H_{32}N_4Ir$: 832.990. Found: 832.649.

Orange complex **4** (in 76% yield) was prepared from the corresponding ligands by a similar procedure.

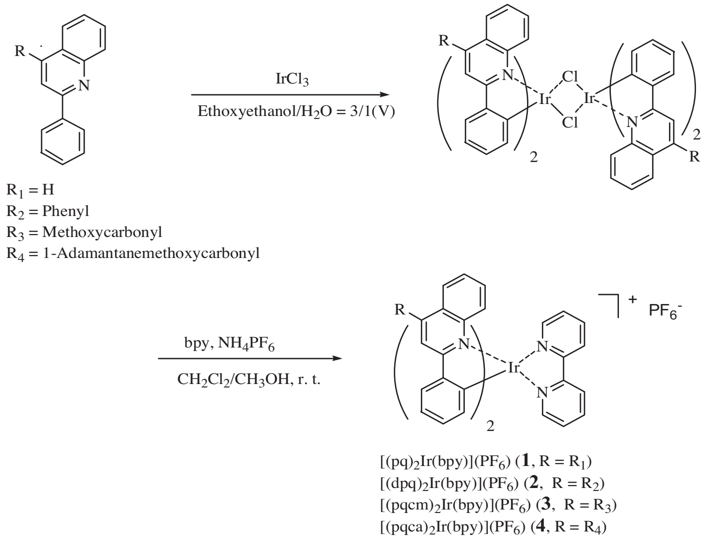
^1H NMR ($CDCl_3$, 400 MHz) δ : 8.61 (s,1H), 8.55 (d, J = 8.4, 1H), 8.25 (d, J = 8.4, 1H),8.11 (d, J = 5.2, 1H), 8.02 (t, J = 8, 1H), 7.97 (d, J = 8,1H), 7.42–7.46 (m, 2H), 7.39 (d, J = 8.8,1H) 7.22 (t, J = 7.6, 1H), 7.02 (t, J = 8, 1H), 6.86 (t, J = 7.6, 1H), 6.53 (d, J = 7.6, 1H), 4.04–4.20 (m, 2H), 2.07 (d, J = 18, 3H), 1.79 (s, 3H), 1.73 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 169.54, 165.15, 155.51, 150.85, 148.23, 147.34, 145.01, 140.20, 139.02, 134.73, 131.53,131.47, 128.24, 127.91, 127.31, 126.93, 125.07, 124.76, 124.42, 123.46, 118.54, 76.20, 39.56, 36.92, 33.66, 29.69, 28.03. MADIL-TOF MS (m/z): Calc. for $C_{64}H_{59}N_4O_4Ir$: 1140.395. Found: 1139.812.

2.3. Crystal structure determination

Single crystals of $[(pqca)Ir(bpy)](PF_6)$ were obtained by repeated recrystallization using a mixture of CH_2Cl_2 and hexane at room temperature, and then mounted on glass fibers. Diffraction data were collected on a Bruker SMART Apex CCD diffractometer with Mo Kα radiation at 296 K using a ω scan mode. Crystallographic and experimental data of $[(pqca)Ir(bpy)](PF_6)$ is listed in Table S1. Selected bond lengths (Å) and angles (deg) is listed in Table S2.

The cationic cyclometalated iridium(III) complexes were synthesized in two steps using standard method. The cyclometalated chloride-bridged dimers were prepared according to the reported procedures and were readily dissociated by bpy ligands in high yield (Scheme 1). These complexes were characterized by the NMR and MS spectroscopy.

In order to confirm the 3-dimensional structure of these complexes, the crystal structure of **4** was determined. The molecular



Scheme 1. Synthetic routes to iridium(III) complexes.

structure of complex **4** is depicted in Fig. 1. Complex **4** consists of a cationic distorted octahedrally coordinated iridium(III) center ligated by two pqca ligands and a bpy ligand with a negative $[PF_6]^-$ counter anion. The two pqca ligands chelate the iridium center with N–N in the *trans* and C–C in the *cis* configuration. The Ir–C bond lengths, ranging from 2.001(8) to 2.025(8) Å, are within the range reported for closely related complexes [19,20]. The Ir–N bond lengths of the pqca ligands spanning from 2.086(6) to 2.092(6) Å, are within the range reported for other mononuclear complexes containing analoguous Ir[C^N] moieties [21]. Moreover, the Ir–N bond lengths between the Ir center and the bpy ancillary ligand (from 2.172(6) to 2.193(6) Å) are longer than those between the Ir center and the pqca ligands because of stronger donating and back-bonding interactions between aryl groups of pqca and the iridium atom. The bpy chelation results in an N(2)–Ir(2)–N(2) bond angles of 75.3(3)°, appreciably lower than the idealized 90° value.

2.4. Cyclic voltammetry and electrochemiluminescence (ECL)

The cyclic voltammograms and ECL signals were both recorded on the MPI-E chemical luminescence instrument (Xi'an Remax Electronic Science Tech. Co. Ltd., China). Cyclic voltammetry (CV) measurements were carried out with a three-electrode cell consisting of a 3 mm-diameter glassy carbon working electrode, non-aqueous Ag/Ag^+ reference electrode and platinum plate auxiliary electrode ($50\ mm^2$). Non-aqueous CH_3CN was used as solvent and 0.1 M $TBAPF_6$ as supporting electrolyte. The concentration of ferrocene is 0.01 M and that of the iridium complexes are 1 mM. The scan rat is $20\ mV\ s^{-1}$.

ECL experiments were referenced with respect to an Ag/AgCl electrode (0.2 versus NHE). Platinum plate electrode was chosen as auxiliary electrode and Au electrode was chosen as working electrode. Before each experiment, the Au working electrode (3.0 mm diameter) was subjected to repeated scanning in the potential range of −0.5 to 1.4 V in a phosphate buffer (0.1 M), until reproducible voltammograms were obtained, and then rinsed with double distilled water [15]. A voltage of −600 V was supplied to the photomultiplier tube (PMT). In each experiment the working electrode was subjected to scan in the potential range of 1.1 V to 1.7 V at $0.1\ V\ s^{-1}$ using cyclic voltammetry and the light intensity recorded every 0.1 mV. The scanning direction is positive. Solutions used to obtain ECL were 5×10^{-9} M iridium(III) complexes and 0.025 M tri-*n*-propylamine (TPA) with 0.1 M (pH 7.4) potassium phosphate (aqueous and mixed 40:60 (v/v) CH_3CN/H_2O) as electrolyte.

2.5. The calculation method of HOMO, LUMO energy levels and quantum efficiencies (Φ_{em})

The HOMO and LUMO energy levels have played an important role in the reaction, CV is widely used to estimate them. The HOMO and LUMO levels are calculated according to the empirical formulae:

$$\mathrm{HOMO\ (eV)} = -I_p = -[(E_{p(anodic)} + E_{p(cathodic)}/2(vsFc+/Fc) + E^f(Fc+/Fc)]$$

$$E_g\ (eV) = hc/\lambda_{abs} = 1240/\lambda_{abs}$$

$$\mathrm{LOMO\ (eV)} = -E_A = E_g - I_p$$

where $E^f(Fc+/Fc)$ is the difference between the Ferrocene level and the vacuum level, the value is 4.8 eV according to the literature [22]. E_g is the difference between the Electron affinity (E_A) and the first ionization energy (I_p). In general, the point of intersection of the absorption curve and the curve of the photoluminescence spectrum is used to estimate the value of λ_{abs}.

The values of fluorescence quantum yield can be calculated according to the following formulae:

$$\Phi_{em} = \Phi_{std}(I_{unk}/A_{unk})(A_{std}/I_{std})(\eta_{unk}/\eta_{std})$$

Φ_{unk}, the fluorescence quantum yield of the sample.
Φ_{std}, the fluorescence quantum yield of the standard.
I_{unk}, the integrated emission intensity of the sample.
I_{std}, the integrated emission intensity of the standard.
A_{unk}, the absorbance of the sample at the excitation wavelength.
A_{std}, the absorbance of the standard at the excitation wavelength.
η_{unk}, the refractive indexes of the sample.
η_{std}, the refractive indexes of the standard.
(pure solvents were assumed)

3. Results and discussion

3.1. Photophysical properties

The UV–Vis absorption spectra of new iridium(III) complexes in CH_3CN at room temperature are shown in Fig. 2. The complexes **1** and **2** have the similar spectra, and the spectra of **3** and **4** are also very close. The strong absorption bands between 226 and 318 nm in the ultraviolet region are assigned to the spin-allowed π–π* transition of the ligands [23]. The moderately intense bands around 340 nm of **1** and **2** probably belong to spin-allowed singlet metal-to-ligand charge-transfer (MLCT). Correspondingly, the bands of **3** and **4** red shift to around 350 nm. The weak absorption bands above 400 nm can be assigned to a spin-forbidden triplet metal-to-ligand charge-transfer (MLCT).

The photoluminescence spectra of new complexes in CH_3CN solution at room temperature are shown in Fig. 3. Complex **1** emits intense luminescence with emission wavelength at 552 nm. The full width at half maximum (FWHM) of this transition is 58 nm. The emission wavelength of **2** is red shift to 566 nm because the

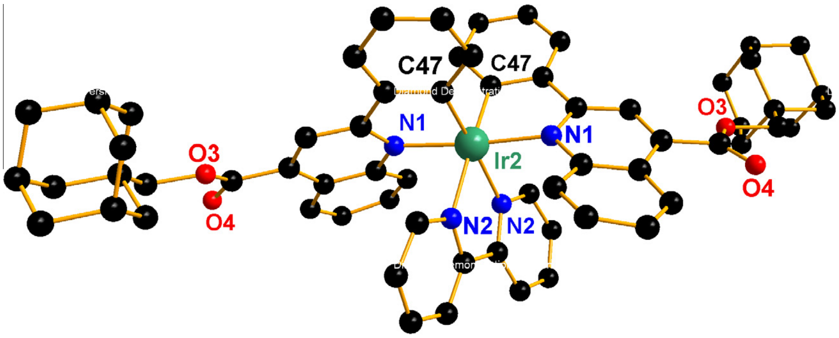


Fig. 1. Perspective view of $[(pqca)_2Ir(bpy)]^+$. H atoms omitted.

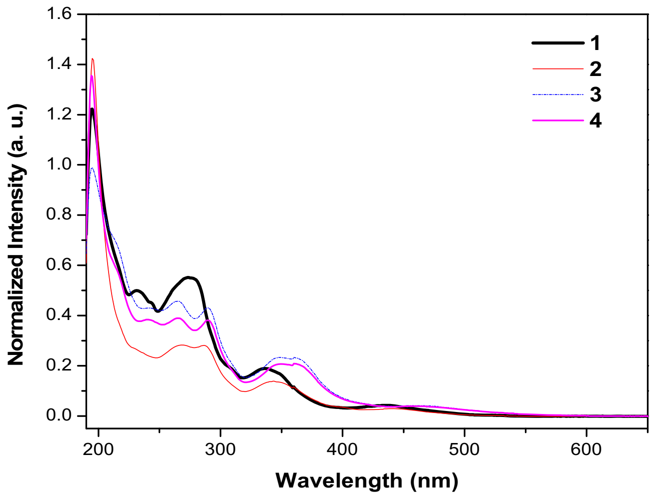


Fig. 2. UV–Vis absorption spectra of new iridium complexes in CH_3CN.

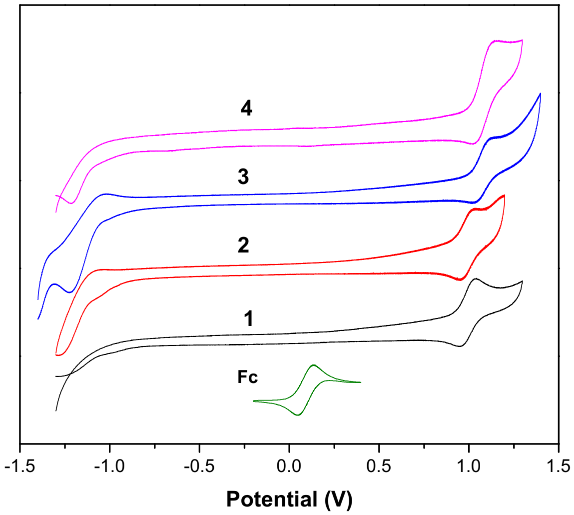


Fig. 4. Cyclic voltammogram of new complexes (reference is ferrocene (Fc)).

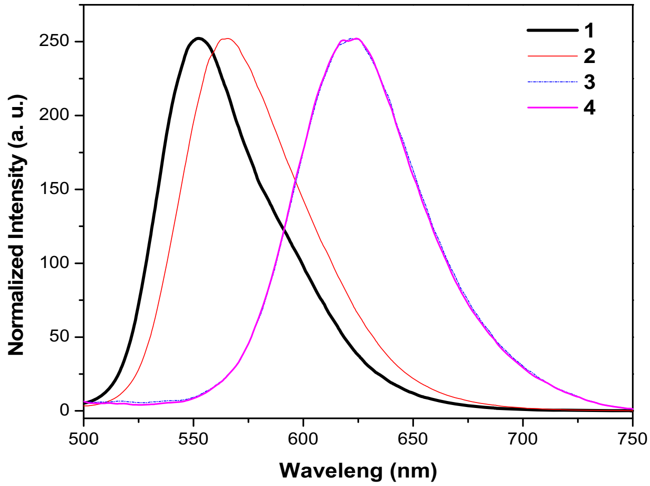


Fig. 3. The photoluminescence spectra of the iridium(III) complexes in CH_3CN solution.

degree of conjugation is enhanced by phenyl group, and the FWHM is 64 nm. Complexes **3** and **4**, which have the carboxylic ester group, have almost the same spectra. The emission wavelengths of **3** and **4** are respectively red shift to 621.5 and 624 nm compared to that of **1**. They have a more obvious red shift than **2** and this phenomenon could be explained by the properties that the carboxylic ester group can enhance the degree of conjugation and has the negative inductive effect. The red shifts are consistent with the UV–Vis absorption result. Their FWHM is 67 nm and has increased in the same trend with red shift. It should be noted that the peak wavelength of **3** and **4** corresponds to a pure red emission which is desirable for example in OLED applications.

The phosphorescence quantum efficiencies (Φ_{em}) of **1** and **2** in degassed CH_3CN solution are *ca.* 0.060 and 0.085, respectively, used a CH_3CN solution of $[Ru(bpy)_3]Cl_2$ ($\Phi_{em} = 0.062$) as the standard solution [3]. The efficiencies of **3** and **4** are all *ca.* 0.011, and are much lower than that of **1** and **2**. The main reason is that the reduced energy band gap has increased the nonradiative energy dissipation.

3.2. Determination of HOMO, LOMO energy level of Iridium complexes

The electrochemical properties of the iridium(III) complexes were examined by cyclic voltammetry, and the data are summarized in Table 1. All complexes show a quasi-reversible one electron oxidation wave with a half-wave potential at 0.989–1.095 V versus $Ag/AgNO_3$ during the anodic scan in CH_3CN solution, which is characteristic of the iridium(III) complexes. No clear reduction waves up to −1.5 V were detected. The HOMO (highest occupied molecular orbital) energy levels of the new complexes were calculated from cyclic voltammogram in comparison with ferrocene (4.8 eV) [22]. The thus obtained HOMO levels (−5.7 eV for **1**–**2** and −5.8 eV for **3**–**4**) in combination with the optical band gaps which were derived from the optical edges of absorption spectra, were used to calculate the LUMO (lowest unoccupied molecular orbital) energy levels [22]. Fig. 4 Both HOMO and LUMO data are collected in Table 1.

3.3. Electrochemiluminescence

Due to the reversible to quasi-reversible nature of the $Ir^{3+/4+}$ anodic redox couple, TPA was used as “oxidative-reductive” coreactant [24] to generate ECL. ECL performance was evaluated in mixed solvent (i.e., 40:60 (v/v) CH_3CN/H_2O) at an Au interface. As shown in Fig. 5, the ECL intensity peaks at potentials of 1.29–1.37 V. The peaks are very broad and positively shifted with respect to the oxidation potential in CH_3CN ($\Delta V \approx 0.3$ V). The ECL signal generated from the present system was mainly produced by the reaction of the oxidized complexes, with the TPA radical.

Table 1
The photophysical properties of new iridium complexes.

Sample	λ_{abs} (nm)	λ_{em} (nm)	E_{pa} (mv)	E_{pc} (mv)	$(E_{pc} + E_{pa})/2$(vs Fc+/Fc) (mv)	HOMO (eV)	LOMO (eV)	E_g (eV)	Φ_{em}	Φ_{ecl}
1	549	552	1053	939	906	−5.71	−3.45	2.26	0.060	3.8
2	553	566	1028	949	899	−5.70	−3.46	2.24	0.085	2.4
3	653	621.5	1170	1019	1005	−5.81	−3.91	1.90	0.011	0.89
4	662	624	1143	1025	994	−5.79	−3.92	1.87	0.011	0.95

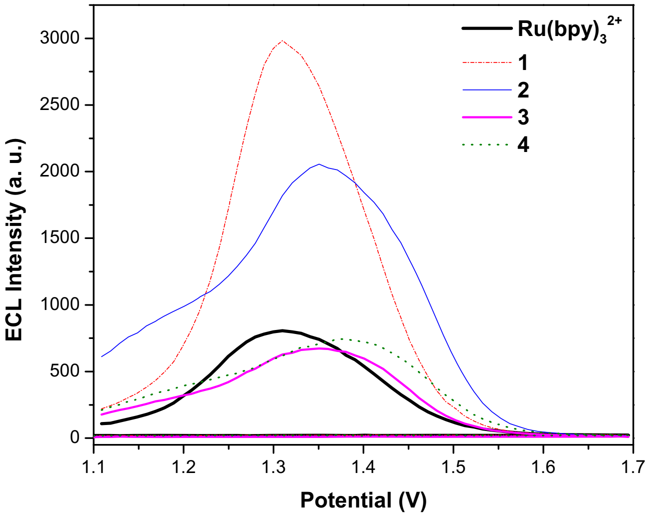


Fig. 5. ECL intensity of the new iridium complexes.

This indicated that the mechanism of the ECL waves followed the classic oxidative reduction coreactant mechanism, where oxidation of TPA generates a strongly reducing species TPA·[25]. The emission excited state was produced from the reaction of directly oxidized iridium(III) complex at the electrode surface with the TPA· radical. The ECL efficiency (Φ_{ecl}) can be directly related to the yield of the excited state generation (Φ_{es}) and to the emission efficiency (Φ_{em}) of the generated emitter: $\Phi_{ecl} = \Phi_{es} \times \Phi_{em}$ [26]. Consequently the higher are Φ_{es} and Φ_{em}, the more intense light emission can be observed in course of an ECL experiment. Using Ru(bpy)$_3^{2+}$ as a relative standard (taken as 1 in the same condition). Values of Φ_{ecl} 3.8, 2.4, 0.89, 0.95 were obtained for complexes **1**, **2**, **3**, **4**, respectively. Thus it is obvious that complexes **1** and **2** have much higher ECL intensity than [Ru(bpy)$_3^{2+}$] though their Φ_{em} is close. Iridium complexes are more effective luminous reagent than ruthenium complexes in this system. By comparing with Table 1 and Fig. 5, complex which has a higher quantum yield tend to have a higher ECL intensity. Thus fluorescence quantum yield has an obvious influence on ECL intensity.

4. Conclusions

In summary, four new cationic iridium complexes have been prepared, and the photophysical influence of the 4-substituents has been investigated. The electron-acceptor character of ester group led to a reduced HOMO–LUMO gap, consequently a red-shift of the UV–Vis absorption and luminescence spectra, but a reduced phosphorescence quantum efficiency and ECL efficiency. With closed phosphorescence quantum efficiency, iridium complexes have more effective ECL than ruthenium complexes in this system. These results are helpful for the analytical applications of cationic iridium complexes.

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Appendix A. Supplementary material

CCDC 956394 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.ica.2014.06.019.

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