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Luminescent rigidochromism of *fac*-[Re(CO)$_3$(phen)(*cis*-bpe)]$^+$ and its binuclear complex as photosensors

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Abstract

The photophysical properties of the *fac*-[Re(CO)$_3$(phen)(L)]PF$_6$ and [(phen)(CO)$_3$Re(L)Re(CO)$_3$(phen)](PF$_6$)$_2$ complexes, where L = *cis*-1,2-bis(4-pyridyl)ethylene (*cis*-bpe) and phen = 1,10-phenanthroline, have been investigated. The *cis* isomer complexes, obtained photochemically from the corresponding non-emissive *trans*-bpe complexes, present an enhanced luminescence at room temperature. The broad emission profile is characteristic of the Re-based emissions from the lowest metal to ligand charge transfer state, ^{3}MLCT. In a rigid medium, such as PMMA or glassy EPA at 77 K, the emission maxima of these complexes present hypsochromic shifts as the medium rigidity increases. Changes in emission properties (energy and lifetime of excited states) are discussed based on the luminescent rigidochromic effect, as the energy of MLCT excited states increases in rigid media. This effect is associated with the solvent dipole orientation response to the changes in electronic configuration between ground and excited states.

Keywords: Rhenium carbonyl complex; Photoisomerization; Luminescent rigidochromism; Photosensor

1. Introduction

Luminescent transition metal complexes have been studied due to their very interesting photophysical properties and potential applications in many fields, such as molecular probes, luminescent sensors, photoswitches, optical memory and photonic devices [1–10].

Rhenium(I) carbonyl polypyridyl complexes, which usually exhibit high stability and fairly strong emission in the visible region, are considered one of the important classes of luminescent complexes. Furthermore, the photophysical properties of these compounds can be tuned by modifying coordinated ligands or solvent environment [11–14]. In particular, rhenium complexes with general formula *fac*-[LRe(CO)$_3$(α-diimine)] (L = Cl$^-$, Br$^-$, I$^-$) are potentially emissive in solution at room temperature as well as in organic glassy media at 77 K. The emission features are often associated to metal to ligand charge transfer (MLCT) state and are strongly affected by variation of the α-diimine, co-ligand L, solvent polarity and the rigidity of the environment. The latter effect is known as luminescent rigidochromism [15–19].

In our previous works [20–23] the photoisomerization of *trans*-1,2-bis(4-pyridyl)ethylene (*trans*-bpe) and its metal complex sensitized photochemistry have been investigated. In this work, luminescent properties of *fac*-[Re(CO)$_3$(phen)(*cis*-bpe)]$^+$ and [(phen)(CO)$_3$Re(*cis*-bpe)Re(CO)$_3$(phen)]$^{2+}$ in fluid solution and in rigid media are reported. In particular, the increase of the MLCT excited state energy, responsible for the reported rigidochromic effect, is discussed in terms of the medium reorganization energy.

2. Experimental

2.1. Materials

HPLC grade solvents (Aldrich) were employed in photophysical and photochemical measurements; all other solvents were reagent grade. *Trans*-bpe (Aldrich) was recrystallized several times from a methanolic solution by the addition of water. The pure *trans*-bpe was collected by filtration, washed with water and then with diethylether. [ClRe(CO)$_5$] (Strem), 1,10-phenanthroline (QM), poly(methyl methacrylate) (PMMA) (average M_W = 25,000) and trifluoromethanesulfonic acid (CF$_3$SO$_3$H), all from Aldrich, were used as-received.

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2.1.1. Synthesis of fac-[ClRe(CO)$_3$(phen)] and fac-[(CF$_3$SO$_3$)Re(CO)$_3$(phen)]

The *fac*-[ClRe(CO)$_3$(phen)] and *fac*-[(CF$_3$SO$_3$)Re(CO)$_3$(phen)] complexes were synthesized as previously described [20].

2.1.2. Synthesis of fac-[Re(CO)$_3$(phen)(trans-bpe)]PF$_6$·H$_2$O

The *fac*-[Re(CO)$_3$(phen)(*trans*-bpe)]PF$_6$ complex was synthesized following the procedure previously described [22]. In 60 ml of methanol, 0.30 g (0.50 mmol) of *fac*-[(CF$_3$SO$_3$)Re(CO)$_3$(phen)] and 0.64 g (3.5 mmol) of *trans*-bpe were dissolved and heated to reflux for 4 h. Saturated aqueous solution of NH$_4$PF$_6$ was added to a room temperature solution to precipitate yellow solid. The solid complex was washed with water and then with diethylether. Yield 60%. Anal. Calc. for ReC$_{27}$H$_{20}$O$_4$N$_4$F$_6$P: C, 40.72%; N, 7.04%; H, 2.51%. Found: C, 40.61%; N, 6.99%; H, 2.39%. ^{1}H NMR for *fac*-[Re(CO)$_3$(phen)(*trans*-bpe)]$^+$ [20] (CD$_3$CN, δ (ppm)): 9.62 (dd, 2H), 8.84 (dd, 2H), 8.54 (dd, 2H), 8.17 (s, 2H), 8.12 (q, 2H), 8.22 (dd, 2H), 7.39 (dd, 2H), 7.30 (dd, 2H), 7.30 (d, 1H), 7.18 (d, 1H).

2.1.3. Synthesis of [(phen)(CO)$_3$Re(trans-bpe)Re(CO)$_3$(phen)](PF$_6$)$_2$

The 0.33 g (0.55 mmol) of *fac*-[(CF$_3$SO$_3$)Re(CO)$_3$(phen)] was dissolved in 50 ml of THF, heated to reflux and added to 0.64 g (3.5 mmol) of *trans*-bpe dissolved in 5.0 ml of THF. The solution was stirred for 4 h under reflux. The yellow solid was separated by filtration, washed with ethanol and then with diethylether. Yield 69%. Anal. Calc. for Re$_2$C$_{48}$H$_{46}$N$_6$O$_6$P$_2$F$_{12}$: C, 37.41%; N, 5.45%; H, 1.70%. Found: C, 37.14%; N, 5.76%; H, 1.98%. ^{1}H NMR for [(phen)(CO)$_3$Re(*trans*-bpe)Re(CO)$_3$(phen)]$^{2+}$ (CD$_3$CN, δ (ppm)): 9.57 (dd, 4H), 8.81 (dd, 4H), 8.18 (dd, 4H), 8.13 (s, 2H), 8.08 (q, 4H), 7.19 (dd, 4H), 7.07 (s, 2H).

2.1.4. Cis isomer complexes

Photolyses of *fac*-[Re(CO)$_3$(phen)(*trans*-bpe)]$^+$ or [(phen)(CO)$_3$Re(*trans*-bpe)Re(CO)$_3$(phen)]$^{2+}$ were carried out directly in solution under 365 nm irradiation. This procedure leads to a mixture of *trans* and *cis* complexes (PSS $>$ 70% for the *cis*-isomer) confirmed by NMR spectroscopy. ^{1}H NMR for *fac*-[Re(CO)$_3$(phen)(*cis*-bpe)]$^+$ [20] (CD$_3$CN, δ (ppm)): 9.56 (dd, 2H), 8.83 (dd, 2H), 8.34 (dd, 2H), 8.18 (s, 2H), 8.09 (q, 2H), 8.06 (dd, 2H), 6.89 (dd, 2H), 6.80 (d, 1H), 6.53 (d, 1H) and for [(phen)(CO)$_3$Re(*cis*-bpe)Re(CO)$_3$(phen)]$^{2+}$ (CD$_3$CN, δ (ppm)): 9.55 (dd, 4H), 8.85 (dd, 4H), 8.00 (dd, 4H), 8.19 (s, 2H), 8.10 (q, 4H), 6.78 (dd, 4H), 6.50 (s, 2H).

2.1.5. PMMA films

The complexes in polymer films were prepared as described previously [22]. Acetonitrile solution of PMMA was mixed into a solution of the complex in the same solvent and then left to dry. The concentrations of each solution were adjusted to provide films in which the metal complex represents ca. 1.5% in mass. PMMA films prepared using the described procedure have been reported to have 30–40 μm of thickness [24].

2.2. Methods

Electronic absorption spectra were recorded on a Hewlett-Packard 8453 diode-array spectrophotometer.

NMR spectra were recorded at 300 K on a DRX-500 (500 MHz) Bruker Avance spectrometer using CD$_3$CN as solvent and TMS as internal standard.

Emission experiments were performed by using an ISS photon counting spectrofluorometer, model PC1, with a photomultiplier-based photon counting detector. The emission spectra of polymer films were obtained by using a front face arrangement for solid samples, in which the excitation light beam was focused where the photolyses had been carried out. Low temperature emission experiments were performed in EPA (diethylether/isopentane/ethanol, 5/5/2) solutions at 77 K.

Photolyses of the mono and binuclear complexes were carried out with an Oriel system described elsewhere [20].

Lifetime measurements were carried out using an Edinburgh Analytical Instruments spectrometer, model LP900S1, consisting of a continuum Surelite II-10 laser (Nd:YAG) (λ_{exc} = 355 nm). The detection apparatus, composed by a monochromator TM300 (Czerny-Turner) and a photomultiplier R955 (Hamamatsu), was positioned at 90° to the incident laser beam. The signals were recorded by an oscilloscope TDS520 (Tektronix).

3. Results and discussion

3.1. ^{1}H NMR

^{1}H NMR spectrum of [(phen)(CO)$_3$Re(*trans*-bpe)Re(CO)$_3$(phen)]$^{2+}$ in CD$_3$CN is presented in Fig. 1, as well as its labeling scheme. Assignments of proton resonance for this complex were made by analogy to the ^{1}H NMR spectra of *trans*-bpe and phen ligands, as well as to the *fac*-[Re(CO)$_3$(phen)(*trans*-bpe)]$^+$ complex, which have already been reported [20].

The proton signals of 1,10-phenanthroline coordinated to the binuclear species [(phen)(CO)$_3$Re(*trans*-bpe)Re(CO)$_3$(phen)]$^{2+}$, H_α, H_β, H_γ and H_δ, are shifted 0.45, 0.38, 0.43 and 0.22 ppm, respectively, when compared to the corresponding proton signals of the free ligand. The rhenium metal center deshields the chelating pyridine protons leading to the observed downfield shifts, as previously observed for *fac*-[Re(CO)$_3$(phen)(*trans*-bpe)]$^+$ and for α-diimine chloro complexes [25].

The coordination of bpe to the metal center lowers the symmetry and splits the degeneracy of each pairs of protons, as previously observed for *fac*-[Re(CO)$_3$(phen)(*trans*-bpe)]$^+$

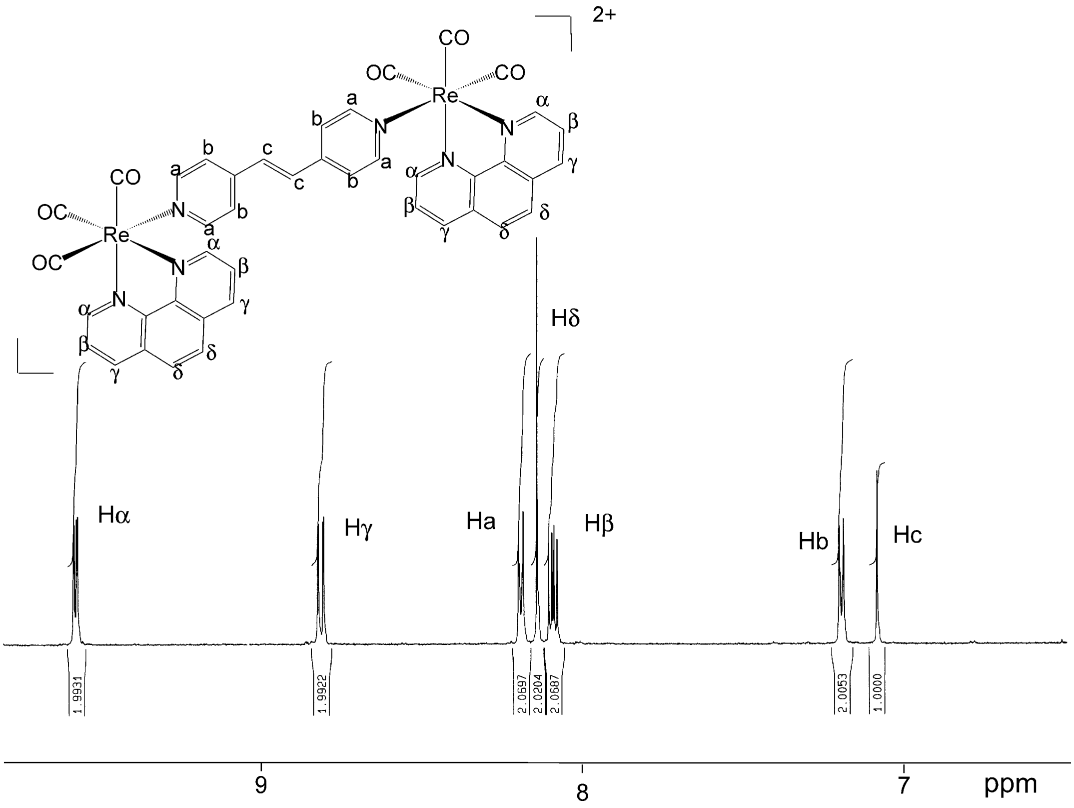

Fig. 1. ^{1}H NMR spectrum of $[(CO)_3(phen)Re(\textit{trans}\text{-bpe})Re(phen)(CO)_3]^{2+}$ in CD_3CN solution; 300 MHz, $T = 300$ K.

[20] and for other polypyridyl rhenium(I) complexes [25,27]. The formation of the binuclear complex restores the symmetry of the bridging ligand [25,27]. The NMR spectrum of $[(phen)(CO)_3Re(\textit{trans}\text{-bpe})Re(CO)_3(phen)]^{2+}$ reverts to a simpler splitting pattern, similar to that found for the free bpe. Three sets of resonance signals for the bridging ligand *trans*-bpe ($H_a = 8.18$ (d), $H_b = 7.19$ (d) and $H_c = 7.07$ ppm (s)) are assigned according to a given pair of equivalent nuclei. All peaks are shifted upfield upon coordination due to the anisotropic effect of the phenanthroline ligand [26].

3.2. Electronic absorption spectra

The absorption spectra of *fac*-$[Re(CO)_3(phen)(\textit{trans}\text{-bpe})]^{+}$ and $[(phen)(CO)_3Re(\textit{trans}\text{-bpe})Re(CO)_3(phen)]^{2+}$ in acetonitrile are presented in Fig. 2. The higher energy bands are associated with electronic transitions localized mainly in the 1,10-phenanthroline and ascribed to intraligand ($\pi_{phen} \rightarrow \pi^{*}_{phen}$) transitions [20].

The bands observed for both complexes around the 300–400 nm region are most likely assigned as MLCT ($d\pi_{Re} \rightarrow \pi^{*}_{L}$) transitions. However, the molar absorption coefficients of 10^4 l mol^{-1} cm^{-1} found in these complexes are much higher than the 10^3 l mol^{-1} cm^{-1} commonly observed for rhenium diimine MLCT transitions [11,12]. In fact, in this region, there is also a contribution of the red-shifted transition of coordinated *trans*-bpe ($\pi_{bpe} \rightarrow \pi^{*}_{bpe}$). Therefore, the lowest energy bands for both complexes have the contribution of MLCT and IL_{bpe} transitions [14,20,22]. It is also observed that the electronic spectrum of the bimetallic complex resembles that of the mononuclear one, but with higher absorptivities [27,28].

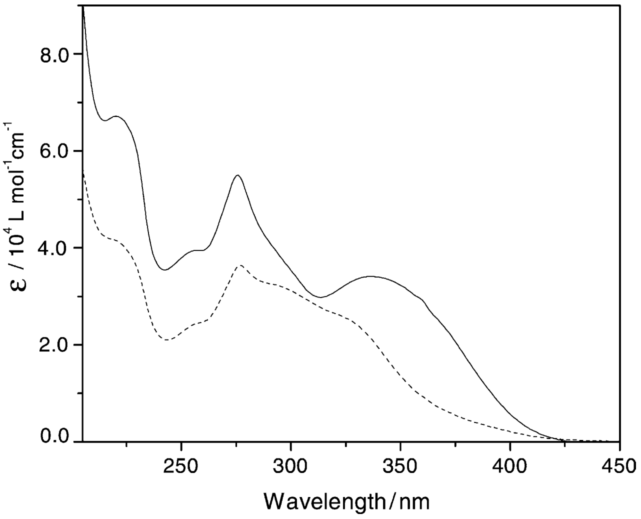

Fig. 2. Electronic spectra of *fac*-$[Re(CO)_3(phen)(\textit{trans}\text{-bpe})]^{+}$ (- - -) and $[(CO)_3(phen)Re(\textit{trans}\text{-bpe})Re(phen)(CO)_3]^{2+}$ (—) in acetonitrile solution at 298 K.

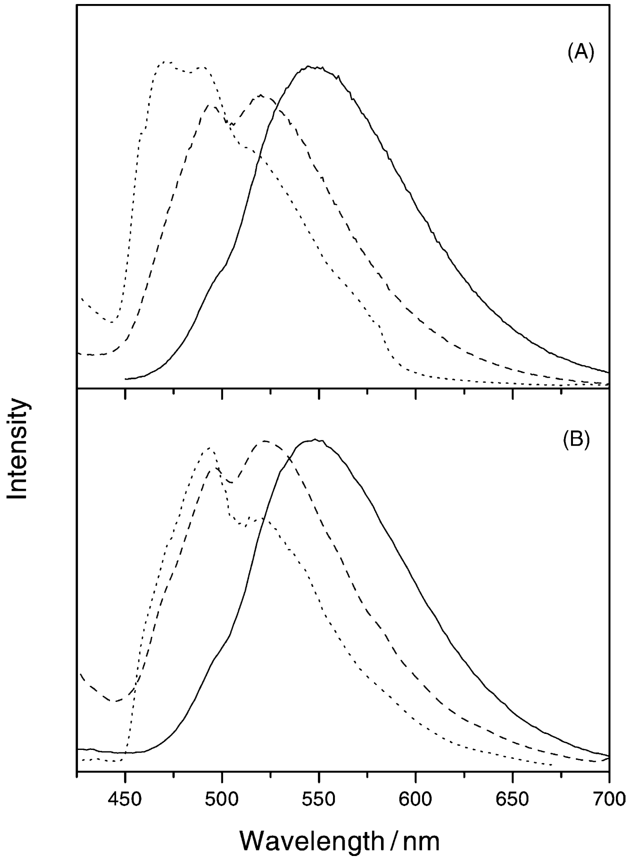

Fig. 3. Emission spectra in acetonitrile solution at 298 K (—), in PMMA at 298 K (– – –) and in EPA at 77 K (· · ·). (A) *fac*-$[Re(CO)_3(phen)(\textit{cis}\text{-bpe})]^+$; (B) $[(CO)_3(phen)Re(\textit{cis}\text{-bpe})Re(phen)(CO)_3]^{2+}$.

3.3. Photophysical properties

The emission spectra of *fac*-$[Re(CO)_3(phen)(\textit{cis}\text{-bpe})]^+$ and $[(phen)(CO)_3Re(\textit{cis}\text{-bpe})Re(CO)_3(phen)]^{2+}$ in acetonitrile and in PMMA at room temperature, as well as in EPA at 77 K, are shown in Fig. 3. The broad structureless band observed in acetonitrile solution for both compounds is typical of MLCT emission of Re(I) complexes [15–17]. The blue shift for mono and binuclear species in PMMA and EPA, in comparison to acetonitrile solution is, respectively, 43 and 38 nm in PMMA, and 70 and 41 nm at 77 K. The observed changes in spectra can be rationalized in terms of rigidochromic effect.

Table 1 presents the MLCT absorption and emission maxima for *fac*-$[Re(CO)_3(phen)(\textit{cis}\text{-bpe})]^+$ (complex 1) and $[(phen)(CO)_3Re(\textit{cis}\text{-bpe})Re(CO)_3(phen)]^{2+}$ (complex 2) in different media, as well as for other two *fac*-$[ClRe(CO)_3(L)]$ complexes [29] (complexes 3 and 4) in order to establish comparison.

The absorption maxima for complexes 1 and 2 in acetonitrile and PMMA (Table 1) present no environmental effect, while for the complexes 3 and 4 exhibit a slight shift in solvents with different polarity. This reveals the influence of solvent polarity on the short lived ^{1}MLCT state involved in absorption. It is also observed that absorption maxima do not present shifts with the matrix rigidity. On the other hand, substantial hypsochromic shift on the emission maxima (around 40 nm) is observed for all the complexes for measurements through the fluid to rigid environment. The observed shift is ascribed to the destabilized ^{3}MLCT state under rigid conditions. There are also evidences that the changes in the long lived ^{3}MLCT state is not only a temperature effect, since the data for all complexes show wide shifts in emission maxima at different environments at the same temperature (298 K).

The different behavior observed for absorption and emission spectra is related to the excited state involved in these transitions. The ^{1}MLCT state, reached right after absorption, is extremely short lived and decays via non-radiative relaxation to the ground state and/or to the emissive ^{3}MLCT state, which is relatively long lived. Changes in the rigidity of the environment are able to influence the ^{3}MLCT level, as it is the more susceptible state to variation in dipole–dipole interaction with the solvent molecules [3,30].

Table 1
Environment effects on MLCT absorption and emission maxima

Complexes	Environment	Absorption (nm)	Emission (nm)	Reference
(1) $[Re(CO)_3(phen)(\textit{cis}\text{-bpe})]^+$	CH_3CN, 298 K	332	550	This work
	PMMA, 298 K	333	507	
	EPA, 77 K		480	
(2) $[(phen)(CO)_3Re(\textit{cis}\text{-bpe})Re(CO)_3(phen)]^{2+}$	CH_3CN, 298 K	325	547	This work
	PMMA, 298 K	326	509	
	EPA, 77 K		506	
(3) $[ClRe(CO)_3(5\text{-Me-phen})]$	Benzene, 298 K	390	588	[29]
	CH_2Cl_2, 298 K	380	588	
	Polyester resin, 298 K		541	
	EPA, 77 K		531	
(4) $[ClRe(CO)_3(5\text{-Br-phen})]$	Benzene, 298 K	395	583	[29]
	CH_2Cl_2, 298 K	387	587	
	Polyester resin, 298 K		546	
	EPA, 77 K		535	

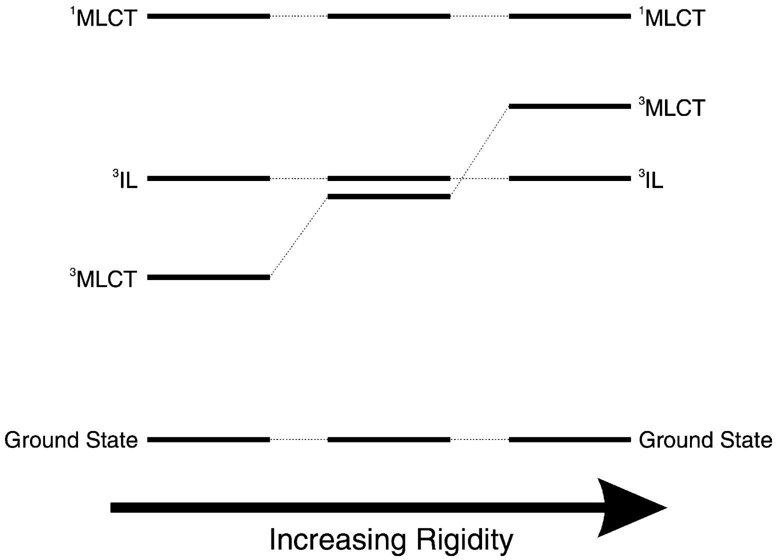

Fig. 4. Simplified energy diagrams.

This rigidochromic effect can be related to changes in the solvation environment. The surrounding solvent molecules are promptly able to orient around the complex to best accommodate its dipole moment. In a relaxed ^{3}MLCT excited state, the dipole moment is reversed from that of the ground state and is destabilized by environment. This relaxation process takes place readily in fluid solution, but is considerably restrained in a more rigid environment, resulting in a destabilization of the ^{3}MLCT excited state. Consequently, this increases the energy of ^{3}MLCT state and results in hypsochromic shift of the emission [2,3].

The scheme of Fig. 4 illustrates a simplified energy diagram that summarizes the relative differences in energies of the ^{1}MLCT, ^{3}MLCT and ^{3}IL excited states for the rhenium complexes as the rigidity increases. This diagram can explain the extension of rigidochromism on the emission (^{3}MLCT) and absorption (^{1}MLCT) bands observed for *fac*-[Re(CO)$_3$(phen)(*cis*-bpe)]$^+$ and [(phen)(CO)$_3$Re(*cis*-bpe)Re(CO)$_3$(phen)]$^{2+}$, as well as of the emission splitting pattern observed in Fig. 3.

At 77 K, one can see that the appearance of vibronic structure points to a transition from $d\pi_{Re} \rightarrow \pi^*_L$ to $\pi \rightarrow \pi^*$ emission from a ligand localized triplet. In a frozen medium, that part of the medium dielectric arising from dipole reorientations adds to the energy of the excited state increasing significantly the energy of the ^{3}MLCT state as depicted in the scheme of Fig. 4. On the other hand, the energy of the $\pi \rightarrow \pi^*$ states are relatively unaffected due to their centro-symmetric character [31]. This can change the excited state ordering and eventually the ligand localized state be the lowest one in frozen medium.

In PMMA there is a dual emission for both complexes with contribution from both blue shifted MLCT and $\pi \rightarrow \pi^*$ states, which is a typical behavior expected for the Re complexes with the MLCT energy lower than those of the $\pi \rightarrow \pi^*$ states [31]. The existence of dual emissions can often be discerned by wavelength dependent lifetime measurements and by temperature dependent spectral measurements, which are in progress.

The preliminary emission lifetimes for both complexes are less than 100 ns in EPA at room temperature, but in the microsecond domain at 77 K. This longer lifetime is another characteristic of ^{3}IL emission, since the organic triplets last a long time. However, further studies must be carried out in order to have a precise assignment.

This kind of effect has already been reported in several studies of a variety of rhenium(I) diimine complexes which have evidenced that the MLCT emission at room temperature transforms into pure IL, mixed IL/MLCT or dual IL/MLCT emissions at 77 K, depending on the relative energies of the states and the rate of internal conversion between them [11,12,18,32,33]. The effect arises from variation in the dipolar interactions between the excited state molecular species and the local solvent dipoles of the surrounding medium which affects the energy gap between them [34].

This study showed the tuning of emitting state of *fac*-[Re(CO)$_3$(phen)(*cis*-bpe)]$^+$ and [(phen)(CO)$_3$Re(*cis*-bpe)Re(CO)$_3$(phen)]$^{2+}$, from an MLCT to IL/MLCT or IL by varying the rigidity of the media. This alteration of emissive excited state results in wide changes in emission spectra and lifetimes, which can be conveniently used in luminescent sensor and/or photonic molecular devices.

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