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#### **RESEARCH ARTICLE**



# Photoluminescent properties of novel design heteroleptic Zn(II) complexes

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#### Abstract

Three novel heteroleptic Zn(II) complexes containing 8-hydroxy quinoline and various pyrazolone-based derivatives were synthesized and their structures confirmed by <sup>1</sup>H-nuclear magnetic resonance, mass spectrometry, Fourier transform infra-red spectroscopy, UV-vis analysis and element analysis. All three complexes showed good photoluminescence properties in the solid state and in solution in the maximum emission range from 475 to 490 nm with a quantum yield of 0.45 to 0.51. Absorption spectra revealed that the complexes possessed a maximum absorption range of 272–281 nm with a band gap of 2.59–2.68 eV. The highest occupied molecular orbital and lowest unoccupied molecular orbital of all the complexes were determine by cyclic voltammetry. All complexes displayed high thermal stability. These characteristics were assessed to find suitability for an alternative cheap light emitter for organic light-emitting diodes.

#### KEYWORDS

heteroleptic complexes, photoluminescence, quantum yield

## 1 | INTRODUCTION

The field of organic light-emitting diodes (OLEDs) has shown tremendous growth in the past few decades due to the development of large numbers of applications for household and outdoor uses, and due to characteristics such as energy saving, friendliness to the environment and cost-effective merits, along with suitable appropriate color quality.<sup>[1–3]</sup> Photoluminescence materials have attracted much attention from research institutes and industrial sectors because of their potential application in OLEDs.<sup>[1,3]</sup> The photoluminescent bis(8hydroxyquinoline) zinc (Znq<sub>2</sub>) complex is the most important light emitter and electron transport material for developing OLEDs. The luminescence properties of quinoline derivatives and complexes with different substitutions on the quinoline ring have been reported as being low-cost, non-toxic and energy-efficient materials for OLED applications.<sup>[4–6]</sup> In OLEDs, Mq<sub>n</sub> coordination compounds act as

Abbreviations: CAS, Centre for Advanced Study; CV, cyclic voltammetry; CVD, chemical vapor deposition; DST, Department of Science and Technology; GUJCOST, Gujarat Council of Science and Technology; HOMO, highest occupied molecular orbital; LUMO, lowest unoccupied molecular orbital; MLCT, metal-ligand charge transfer; NMR, nuclear magnetic resonance; OLED, organic light emitting diodes; TSG, Technology System Group.

electron transport materials and it has been suggested that this ability arises from  $\pi$ - $\pi$  interactions between adjacent molecules.<sup>[7-12]</sup>

The recombination of an electron-hole generates a triplet excited state for maximum OLED light output.<sup>[13]</sup> This situation is achieved by dispersal of the electron accepter (A) and electron donor (D) environment or by linking by the electron acceptor and electron donor with suitable spacers.<sup>[14–18]</sup> The donor–acceptor system of the organic molecule is mostly used for tuning the emission color and band gap, generation of a  $\pi$ -conjugation charge, and faster charge transfer from donor to acceptor etc. Thus, refining our detailed knowledge of these photophysical and electrochemical events is required for developing a highly capable donor–accepter type material for OLED application.

In many cases, the electron-transporting mobility of zinc complexes with 8-hydroxyquinoline goes beyond that found for aluminum complexes, which are the most widely used electron-transporting materials for OLEDs. Thus zinc complexes may be potential candidates to enhance the electron-transporting properties in OLEDs.<sup>[19]</sup>

In the present study we designed and synthesized three novel donor-metal-acceptor (D-M-A) units of heteroleptic Zn(II) complexes containing fixed 8-hydroxyquinoline that acts as an electron donor unit. For the choice of electron acceptor unit we designed heterocyclic  $\beta$ -diketonate ligands: 5-hydroxy-3-methyl-1-phenyl-1H-pyrazole-4-carbaldehyde (L-1), 5-hydroxy-3-methyl-1-(p-tolyl)-1H-pyrazole-4-

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carbaldehyde (L-2) and 1-(2-chlorophenyl)-5-hydroxy-3-methyl-1Hpyrazole-4-carbaldehyde (L-3) for complex 1, complex 2 and complex 3 respectively. All complexes had good photoluminescence performance in the solid state and in different solvents with high quantum yields of 0.45–0.51.

## 2 | EXPERIMENTAL

#### 2.1 | Materials and methods

1-Phenyl-3-methyl-2-pyrazoline-5-one (E-Merck), 8-hydroxyquinoline and Zn(II) acetate (Sigma-Aldrich) were used as received. Solvents and other reagents were used after purification by a standard method described in the literature.<sup>[20,21]</sup> All experiments were performed under a nitrogen atmosphere using standard Schlenk techniques.

#### 2.2 | Characterization

Photoluminescence, quantum yield and lifetime were measured using a Perkin Elmer LS-55 spectrophotometer (Perkin Elmer, USA). UVvisible spectra were generated using a Shimadzu 160A spectrophotometer. Element analyses were performed at central drug research institute (CDRI), Lucknow, India. FT-IR spectra were recorded on an MB-3000 infra-red spectrophotometer. The <sup>1</sup>H-NMR spectra were recorded with an NMR spectrometer operating at frequencies of 400.13 MHz with a tetramethylsilane (TMS) standard on a Bruker 400 MHz spectrometer. Deuterated dimethyl sulfoxide (DMSO-d<sub>6</sub>) was used as the solvent for measurements and calibration was performed using the residual peak of the deuterated solvent (δ 2.52 for  $^{1}\text{H-NMR}$ ). The mass spectra of the complexes were recorded on a Shimadzu LCMS-2010 spectrometer, cyclic voltammetry (CV) was performed on a CHI660E electrochemical workstation, thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) were carried out using a Perkin Elmer Pyris 1 instrument at a heating rate of 10°C/ min under a nitrogen atmosphere.

#### 2.3 | Preparation of ligands and Zn(II) complexes

The ligands L-1, L-2 and L-3 were synthesized according to our early reports.<sup>[22-24]</sup>

# 2.3.1 Synthesis of [Zn(q)(L-1)] ((4-formyl-3-methyl-1-phenyl-1H-pyrazol-5-yl)oxy)(quinolin-8-yloxy)zinc (1)

A mixture of zinc(II) acetate (1 mmol, 0.18 g) and 8-hydroxyquinoline (1 mmol, 0.14 g) in a mixture of ethanol and water (20 ml, 3:1, v/v) was cooled down to 5°C and added dropwise to a solution of 5-hydroxy-3-methyl-1-phenyl-1*H*-pyrazole-4-carbaldehyde (L-1) (1 mmol, 0.20 g) in 5 ml ethanol at 5°C temperature. After addition, the reaction mixture was refluxed for 3 h under a nitrogen atmosphere. The complex 1 precipitate was filtered and washed with water (20 ml) and ethanol (20 ml) as eluent: yellow solid; yield 83%.

Spectral data: IR (KBr pellet cm<sup>-1</sup>); 504, 744, 1109, 1285, 1327, 1390, 1468, 1500, 1578, 1657. <sup>1</sup>H–NMR (400 MHz, d<sub>6</sub>-DMSO,  $\delta$  [ppm]): 1.99 (s, 3H), 6.80–7.00 (m, 2H), 7.25 (bs, 2H), 7.40–7.80 (m, 3H), 7.98 (bs, 3H), 8.32 (d, 1H), 9.09 (s, 1H). MS calcd for C<sub>20</sub>H<sub>15</sub>O<sub>3</sub>N<sub>3</sub>Zn

$$\label{eq:main_star} \begin{split} [M~+~H]^+~\textit{m/z}~410.74,~found~411;~anal.~Calcd~for~C_{20}H_{15}O_3N_3Zn;~C~58.48,~H~3.68,~N~10.23;~found;~C~58.24,~H~3.42,~N~10.36. \end{split}$$

#### 2.3.2 | 1.3.2 synthesis of [Zn(q)(L-2)] ((4-formyl-3-methyl-1-(p-tolyl)-1H-pyrazol-5-yl)oxy)(quinolin-8-yloxy)zinc (2)

A mixture of zinc(II) acetate (1 mmol, 0.18 g) and 8-hydroxyquinoline (1 mmol, 0.14 g) in a mixture of ethanol and water (20 ml, 3:1, v/v) was cooled down to 5°C and added dropwise to a solution of 5-hydroxy-3-methyl-1-(p-tolyl)-1H-pyrazole-4-carbaldehyde (L-2) (1 mmol, 0.21 g) in 5 ml ethanol at 5°C. After the addition, the reaction mixture was refluxed for 3 h under a nitrogen atmosphere. The complex 2 precipitate was filtered and washed with water (20 ml) and ethanol (20 ml) as eluent: yellow solid; yield 81%.

Spectral data: IR (KBr pellet cm<sup>-1</sup>); 507, 744, 790, 819, 1110, 1295, 1390, 1468, 1504, 1605, 1657. <sup>1</sup>H–NMR (400 MHz, d<sub>6</sub>-DMSO,  $\delta$  [ppm]): 2.19 (s, 3H), 2.21 (s, 3H), 7.19 (bs, 3H), 7.40–7.80 (m, 2H), 7.91 (bs, 3H), 8.20–8.50 (m, 2H), 9.11 (s, 1H). MS calcd for C<sub>21</sub>H<sub>17</sub>O<sub>3</sub>N<sub>3</sub>Zn [M + H]<sup>+</sup> *m/z* 424.77, found 425; anal. Calcd for C<sub>21</sub>H<sub>17</sub>O<sub>3</sub>N<sub>3</sub>Zn: C 59.38, H 4.03, N 9.89; found: C 59.27, H 4.01, N 9.96.

#### 2.3.3 | Synthesis of [Zn(q)(L-3)] ((1-(2-chlorophenyl)-4-formyl-3-methyl-1H-pyrazol-5-yl)oxy)(quinolin-8-yloxy)zinc (3)

A mixture of zinc (II) acetate (1 mmol, 0.18 g) and 8-hydroxyquinoline (1 mmol, 0.14 g) in a mixture of ethanol and water (20 ml, 3:1, v/v) was cooled down to 5°C temperature and add dropwise to a solution of 1-(2-chlorophenyl)-5-hydroxy-3-methyl-1H-pyrazole-4-carbaldehyde (L-3) (1 mmol, 0.23 g) in 5 ml ethanol at 5°C. After addition, the reaction mixture was refluxed for 3 h under a nitrogen atmosphere. The complex 3 precipitate was filtered and washed with water (20 ml) and ethanol (20 ml) as eluent: yellow solid; yield 88%.

Spectral data: IR (KBr pellet cm<sup>-1</sup>); 503, 744, 820, 1109, 1327, 1390, 1468, 1501, 1577, 1605. <sup>1</sup>H–NMR (400 MHz, d<sub>6</sub>-DMSO,  $\delta$  [ppm]): 2.12 (s, 3H), 7.16 (s, 1H), 7.30–7.70 (m, 6H), 7.93 (d, 2H), 8.42 (d, 1H), 9.08 (s, 2H). MS calcd for C<sub>20</sub>H<sub>14</sub>O<sub>3</sub>N<sub>3</sub>ZnCl [M + H]<sup>+</sup> *m*/*z* 445.18, found 445; anal. Calcd for C<sub>20</sub>H<sub>14</sub>O<sub>3</sub>N<sub>3</sub>ZnCl: C 53.96, H 3.17, N 9.44; found: C 53.93, H 3.14, N 9.51.

#### 3 | RESULTS AND DISCUSSION

All ligands and complexes were stable at atmospheric temperature and pressure and were soluble in common organic solvents. They gave satisfactory spectroscopic data and the results were in good agreement with the proposed structure in Scheme 1.

#### 3.1 | Structural description

The synthetic routes of ligands (L-1 to L-3) are shown in Scheme 1. The ligands were characterized by <sup>1</sup>H–NMR and ESI-MS. The complexes were characterized by element analysis, and FT-IR, TGA, DSC and mass spectrometry, <sup>1</sup>H–NMR and UV–visible analysis. The <sup>1</sup>H–NMR spectra of the ligands showed the characteristics of an enolic proton at  $\delta$  8.5–12.5, this enolic proton peak disappeared during complexation with the Zn(II) ion. FT-IR spectra of the complexes showed

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**SCHEME 1** Synthesis of ligands and Zn(II) complexes 1, 2 and 3

absorptions between 1550 and 1570 cm<sup>-1</sup> and 1468 cm<sup>-1</sup> which were assigned to the uC = N bond of the pyrazolone ring and the uC-O bond, respectively. Additional information from IR spectra came from the shift in stretching frequency of the uC = O bond, ~1680 cm<sup>-1</sup> of ligand to ~1650 cm<sup>-1</sup> after complex formation with Zn(II). The bands between 470 and 510 cm<sup>-1</sup> were assigned to uZn-O stretching.<sup>[25]</sup> The mass spectra of complexes 1, 2 and 3 showed the characteristic molecular ion signals at 411.08, 425.00 and 445.32 m/z respectively.

#### 3.2 | Photophysical properties

The ultraviolet-visible (UV-vis) spectra of complexes 1, 2 and 3 were recorded in acetonitrile at room temperature (Figure 1).



**FIGURE 1** Absorption spectra of complexes 1, 2 and 3 recorded in acetonitrile solution

Complex 1-Ligand L-1:	$\mathbf{R} = -\mathbf{H}$
Complex 2-Ligand L-2:	$\mathbf{R} = pera - CH_3$
Complex 3-Ligand L-3:	$\mathbf{R} = ortho - \mathbf{Cl}$

As shown in Figure 1 all these complexes displayed characteristics of a multiple absorption band below 350 nm, originating from ligandcentered (LC)  $\pi$ – $\pi$ \* transitions.<sup>[26]</sup> Conversely, the weaker absorption band above 400 nm should correspond to metal–ligand charge transfer (MLCT) transitions.<sup>[27]</sup> Energy bandgaps of complexes were estimated using equation 1:

Band gap energy 
$$(Eg) = hc/\lambda_{onset(UV-vis)}$$
 (1)

Photoluminescence (PL) spectra of complexes 1, 2 and 3 were recorded in the solid state and in solution in acetonitrile at room temperature (Figures 2 and 3).

The maximum emissions of complexes 1, 2 and 3 were at 487, 482 and 487 nm in acetonitrile and at 485, 480 and 478 in the solid



**FIGURE 2** Photoluminescence spectra of complexes 1, 2 and 3 recorded in acetonitrile solution



**FIGURE 3** Photoluminescence spectra of complexes 1, 2 and 3 recorded in the solid state

state respectively, where the Znq<sub>2</sub> complex showed maximum emission at 555 nm and 496 nm in acetonitrile and in solid state respectively. All three complexes showed similar PL spectra in solution and in the solid state respectively with a difference of 5 nm, which indicated that the PL properties of these complexes remained unchanged in the solvent and the solid state, these small shifts (~5 nm) indicated that there is little effect of phenyl ring substitution in pyrazolone ligands. Conversely, Znq<sub>2</sub> showed a large shift between solution and solid state in PL spectra due to its isomerization effect.<sup>[28]</sup> Therefore the Znq<sub>2</sub> complexes were less promising for wet and dry processability for OLEDs construction without color saturation as compared with complexes 1, 2 and 3.

The photoluminescence quantum yields ( $\Phi_{PL}$ ) were measured using equation 2:

Quantum Yield 
$$\Phi(x) = \Phi(r) * \frac{Abs(r)}{Abs(x)} * \frac{Area(x)}{Area(r)} * \frac{\eta 2(x)}{\eta 2(r)}$$
 (2)

where r = reference sample, x = unknown sample,  $\Phi$  = quantum yield, Abs = absorbance, Area = area of the entire emission, and  $\eta$  = refractive index of the solvent.

All complexes showed good quantum yield in the range 0.45–0.51 which is higher than that of  $Znq_2$  (0.03) in acetonitrile. In the solid state, the quantum yield of complexes 1, 2 and 3 were 0.48, 0.42, 0.47 respectively and was close to the  $Znq_2$  value (0.5).<sup>[29]</sup> The lifetime of complexes 1, 2 and 3 were in the range 0.03–0.045 msec. Here we correlated our lifetime data with quantum yield, but we found that there was no significant correlation because quantum yield depends on concentration, but fluorescence lifetime depends on the excited state of the electron and this is independent of the concentration of fluorophore.<sup>[30]</sup>

Importantly, the designed complexes showed good quantum yield with a blue shift compared with Znq<sub>2</sub>. Therefore complexes 1, 2 and 3 are a rational alternative because generally quantum yield at higher energy transition (blue shift) is lower when compared with lower transition (red shift).<sup>[30]</sup> Therefore, incorporation of the electron acceptor group of pyrazolone derivatives in a complex to form

a donor-metal-acceptor unit is responsible for tuning the color emission from green ( $Znq_2$ ) to blue for complexes 1, 2 and 3, at the same time as increasing quantum yield (0.42–0.48).<sup>[31]</sup>

#### 3.3 | Electrochemical properties

Cyclic voltammetry (CV) was performed on a CHI660E electrochemical workstation using a three-electrode assembly comprising a Pt disk as the working electrode, a non-aqueous Ag/Ag<sup>+</sup> reference electrode and an auxiliary platinum electrode. The experiments were performed at room temperature under a nitrogen atmosphere in dimethylformamide (DMF) using 0.1 M tetrabutyl ammonium perchlorate (TBAP) as the supporting electrolyte. The electrochemical properties of complexes 1, 2 and 3, were investigated by means of CV, shown in Figure 4.

The highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) energy levels were estimated using equations 3 and 4:

$$E_{HOMO} = -(E_{onset} \text{ vs Fc} + /Fc + 5.1) \text{ eV}$$
(3)

$$E_{LUMO} = E_{HOMO} + E_g \tag{4}$$

The estimated  $E_{onset}$  (V), energy bandgap ( $E_g$ ), HOMO and LUMO energy levels are summarized in Table 1.

The HOMO levels of complexes 1, 2 and 3 were –5.19, –5.30 and –5.09 respectively, whereas the LUMO levels were –2.51, –2.63 and –2.50 respectively. Therefore the complexes showed a difference in HOMO level of 0.21 and in LUMO level of 0.13 due to the effect of the phenyl ring substitution in the pyrazolone ligands. Pyrazolone ligands, therefore, are more useful for tuning the band gap for HOMO and LUMO levels of complexes for OLED construction. Conjugation of pyrazolone-based ligands offers such features as substitution effect, complexes 1 and 2 showed a similar band gap 2.68 that indicated that the effect of methyl substitution was less compared with that of complex 3, and showed that the lower band gap may be due to a chloride substitution.<sup>[30]</sup>



**FIGURE 4** Cyclic voltammograms of complexes 1, 2 and 3 recorded in DMF solution

TABLE 1 Photophysical and thermal parameters of complexes

Compounds	λ <sub>PL</sub> <sup>a</sup> [nm]	λ <sub>PL</sub> <sup>b</sup> [nm]	λ <sub>abs</sub> c [nm]	λ <sub>onset</sub> d [nm]	Eg <sup>e</sup> [eV]	E <sub>onset</sub> f [eV]	HOMO <sup>g</sup> [eV]	LOMO <sup>h</sup> [eV]	τ <sub>m</sub> ՝ [°C]	QY <sup>j</sup> (%)	QY <sup>k</sup> (%)	Life Time (ms)
[Zn(q)(L-1)]	487	485	272	461	2.68	0.09	-5.19	-2.51	338	0.51	0.48	0.033
[Zn(q)(L-2)]	482	480	277	463	2.67	0.20	-5.30	-2.63	321	0.45	0.42	0.039
[Zn(q)(L-3)]	487	478	281	478	2.59	-0.01	-5.09	-2.50	301	0.48	0.47	0.044

<sup>a</sup>Measured in acetonitrile. <sup>b</sup>Measured in solid state. <sup>c</sup>Measured in acetonitrile at concentration 5 × 10<sup>-6</sup> mol/L. <sup>d</sup>Estimated from absorption spectra. <sup>e</sup>Estimated from absorption onset from UV-visible. <sup>f</sup>Estimated from cyclic voltammetry. <sup>g</sup>Estimated from E<sub>1/2</sub> oxd and E<sub>onset</sub> obtained from cyclic voltammetry. <sup>h</sup>Estimated using HOMO and E<sub>g</sub>. <sup>i</sup>Estimated from DSC. <sup>j</sup>In acetonitrile. <sup>k</sup>In solid state.

#### 3.4 | Thermal properties

DSC and TGA measurements were performed for all the Zn(II) complexes under a nitrogen atmosphere. The DSC results suggest that complexes 1, 2 and 3 have peaks above 300°C. TGA results suggest that all complexes exhibit good thermal stability with high decomposition temperatures (>300°C). These high values are required and important for construction of OLEDs based on the evaporation method, as well as the chemical vapor deposition (CVD) method. In addition to this, the higher thermal stability suggests that the complexes are amorphous in nature without glass transition temperatures (T<sub>e</sub>).<sup>[32]</sup> Here complexes show endothermic peak in DSC at higher temperature which revels that all the complexes are in sloe existence of monomeric structure and they are not form any kind of dimer or oligomers at higher temperature.<sup>[34]</sup> Monomeric structure at higher temperature offer good film forming properties along with higher degree of order than dimer or trimer of oligomers and enhances device performance.<sup>[30]</sup> Further similar type of observation have also been reported previously by another research group.<sup>[35]</sup>

#### 4 | CONCLUSIONS

In summary, novel heteroleptic Zn(II) complexes were synthesized and characterized. Their photophysical and electrochemical properties suggested that synthesized complexes 1, 2, and 3 were all suitable as emissive materials for OLEDs. All complexes showed good luminescence properties in the solid and solution states. The designed complexes showed good quantum yield along with a blue shift when compared with Znq<sub>2</sub>. These materials are a feasible alternative because generally the quantum yield at higher energy transitions (shorter wavelengths) is lower. Furthermore, the CVs of the complexes indicated that they have bipolar properties and can be utilized in single-layer devices. These complexes showed higher glass transition temperatures, and a sole existence of monomeric structure. As well as these advantages, our designed Zn(II) complexes are a cheaper alternative as emitter materials for OLEDs compared with other emitter precursors such as IrCl<sub>3</sub> xH<sub>2</sub>O, Os and Pt.

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