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Synthesis and luminescent properties of novel polymeric metal complexes with bis(1,10-phenanthroline) group

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Abstract

The polymeric metal complexes of 1,4-bis([1,10]phenanthroline-[5,6-*d*]imidazol-2-yl)benzene (BPIB) with copper(II) and zinc(II) were synthesized and characterized by FT-IR, elemental analysis, conductivity measurements and gel permeation chromatography. The stoichiometry of the polymeric metal complexes was $[(C_{32}H_{18}N_8)_7(MCl_2)_8]$ (M = Cu(II), Zn(II)); BPIB forms a five-membered chelate ring. UV–vis and fluorescence spectra at room temperature revealed that the maximal emission of the complexes is red shifted as a result of coordination with the metal ions. Both complexes have good thermal stability and emit blue/green luminescence at 526 and 517 nm in the solid state and purple/blue luminescence at 423 and 434 nm in DMSO.

Keywords: Polymeric metal complexes; Synthesis; Luminescence properties; 1,4-Bis([1,10]phenanthroline-[5,6-*d*]imidazol-2-yl)benzene

1. Introduction

Since the first report on polymer light-emitting diodes (PLEDs) [1], a number of π-conjugated polymers have been intensively investigated in order to fabricate devices for industrial applications [2–4]. Among them, poly(1,4-phenylenevinylene) (PPV) has attracted a great deal of attention in recent years because of its high electroluminescent properties [5]. When people discovered that when the metal ions were introduced to polymers, the polymers not only exhibit the properties of polymers, but also possess properties of inorganic and organic small molecule metal coordination compounds, so tremendous interest has been drawn to the research on metal coordination polymers to investigate their potential applications.

In the past few years, many metal coordinated π-conjugated polymers have been synthesized. The synthesis and study of such polymeric metal complexes have been the subject of active research. Some π-conjugated polymers [6–8] have been investigated as PLEDs, which possess many advantages over inorganic and organic small molecular metal complexes, such as good thermal stability, process ability and easy film-forming ability.

1,10-Phenanthroline has a rigid framework and possesses a superb ability to coordinate many metal ions, which show potential for technological applications, due to their high charge transfer mobility, strong absorption in the ultraviolet spectral region, bright light-emission and good electro- and photo-active properties [9–13]. BPIB contains two 1,10-phenanthroline groups and has two nitrogen donors on two sides that can chelate to two metal centers [14]; BPIB has a big π-conjugated structure, which results in maximum emission leading to red shift [15–17]. Based on this, we use BPIB to chelate to metal ions in order to form the corresponding main chain polymeric metal complexes. The emission of these complexes will possibly be enhanced, because of the π-conjugated structure and the rigid structure of the benzene ring, which lead to decrease in the loss of energy by thermal vibrational decay and enhance their thermal stability.

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Furthermore, in order to realize full-color displays, improve durability and increase luminescence efficiencies, we try to design and synthesize some novel polymeric complexes: BPIB with Cu(II) (**2**) and Zn(II) (**3**). In this paper, we will discuss the synthesis and characterization of these polymeric metal complexes. The luminescent properties of these complexes were systematically investigated for the first time.

2. Experimental

2.1. Materials

All materials were obtained from Shanghai Chemical Reagent Co., Ltd. (Shanghai, China) and were used without any further purification. All solvents used in this work were analytical grade. The compounds were identified with ^{1}H NMR, FT-IR, UV–vis and elemental analysis (EA).

2.2. Instrument and measurements

The FT-IR spectra were obtained on a Perkin–Elmer Spectrum One Fourier transform infrared spectrometer (KBr pellet). NMR spectra were recorded on a Bruker NMR 400 spectrometer operating at 400 MHz at room temperature in DMSO-d_6 or $CDCl_3$ using TMS as the internal reference. Thermogravimetric analysis (TGA) was conducted under argon at a heating rate 20 K min^{-1} with a Shimadzu TGA-7, glass-transition temperature (T_g) was determined by differential scanning calorimetry (DSC) at a heat rate of 20 °C min^{-1} with a Perkin–Elmer DSC-7 thermal analyzer. Elemental analysis for C, H, and N was carried out using a Perkin–Elmer 2400 II instrument, metal ion and chlorine ion were measured by chemical method. Molar conductance measurements were carried out in DMSO solution (10^{-4} M) using a model LF Digi 55 conductivity. Ultraviolet–visible (UV–vis) spectra were measured with a Lambda 25 spectrophotometer. The fluorescence spectral analysis was conducted on a Perkin–Elmer LS55 luminescence spectrometer with a xenon lamp as the light source. The solubility of the polymers was determined using polymeric complexes in appropriate solvent at room temperature. The M_n and molecular weight distribution (M_w/M_n) were measured on a Waters 2414 gel permeation chromatography (GPC) instrument with a set of HT3, HT4 and HT5, μ-styrayel columns with DMF as an eluent (1.0 mL min^{-1}) at 50 °C, calibrated by polystyrene standard.

2.3. Synthesis

2.3.1. Synthesis of 1,10-phenanthroline-5,6-dione

This compound was synthesized according to the published literature [18]. A mixture of 1,10-phenanthroline (5.0 g, 28 mmol) and KBr (5.0 g, 42 mmol) was added to a three-necked flask equipped with a dropping funnel, and then an ice cold mixture of concentrated H_2SO_4 (50 mL) and HNO_3 (50 mL) was dropped. The mixture was heated to reflux for 6 h. The hot yellow solution was poured over 600 mL of ice and water and neutralized carefully with NaOH until neutral to slightly acidic pH was attained. Extraction with $CHCl_3$ was followed by drying with anhydrous Na_2SO_4 and removal of solvent, and the precipitate was purified further by crystallization from absolute ethanol to give 5.6 g (96%) of 1,10-phenanthroline-5,6-dione. ^{1}H NMR ($CDCl_3$, δ, ppm): 9.12 (2H, d, H-Py), 8.51 (2H, d, H-Py), 7.58 (2H, d, H-Py).

2.3.2. Synthesis of BPIB (*1*)

A mixture of 1,10-phenanthroline-5,6-dione (1.89 g, 9 mmol), ammonium acetate (13.86 g,180 mmol), terephthalaldehyde (0.603 g, 4.5 mmol) and glacial acetic acid (150 mL) was added to a three-necked flask and refluxed for 3 h, and then cooled to room temperature. The precipitate was collected, washed enough with H_2O for three times, and dried in vacuo at 60 °C, a greenish yellow powder was obtained. Yield 2.28 g, 46%. ^{1}H NMR (DMSO, δ, ppm): 13.97 (2H, s, H–N), 9.08 (4H, d, H-Py), 9.00 (4H, d, H-Py), 8.62 (4H, d, H-Py), 7.91 (4H, d, H-Ph). IR (KBr, cm^{-1}): 3402, 3064, 1608, 1557, 1503, 1451, 1399, 805, 739, 616. Anal. Calcd. for $C_{32}H_{18}N_8$: C, 74.70; H, 3.52; N, 21.78. Found: C, 74.31; H, 3.39; N, 20.78.

2.3.3. Synthesis of polymeric metal complexes

2.3.3.1. General procedure for the preparation of polymeric metal complexes. To a methanol solution (10 mL) of metal halides (6 mmol) was added a DMSO solution (20 mL) of the ligand BPIB (2.06 g, 4 mmol). The reaction mixture was stirred for 24 h at 60 °C. After removing the solvent, the precipitate was washed enough with absolute methanol until the filtrate was colorless, and then dried in vacuo at appropriate temperature.

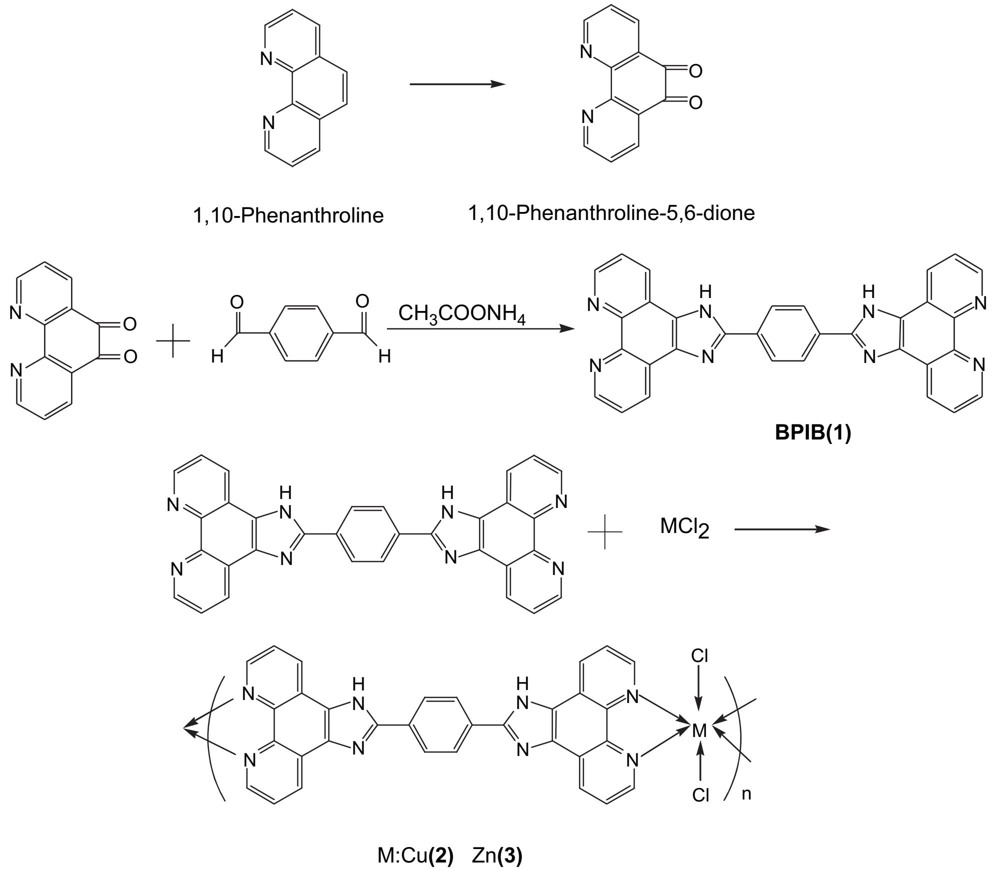
*Synthesis of BPIB–Cu(II)(**2**).* BPIB–Cu(II) was synthesized and purified by the same method. To a methanol solution (10 mL) of $CuCl_2 \cdot 2H_2O$ (1.023 g, 6 mmol) was added a DMSO solution (20 mL) of the ligand BPIB (2.06 g, 4 mmol), which gave a blue solid. Yield 2.26 g (87%). IR (KBr, cm^{-1}): 3470, 3076, 1651, 1602, 1475, 1452, 1403, 813, 732, 643, 557. Anal. Calcd. for $[(C_{32}H_{18}N_8)_7(CuCl_2)_8]$: C, 57.52; H, 2.71; N, 16.77; Cl, 12.13; Cu, 10.87. Found: C, 57.31; H, 2.69; N, 16.57; Cl, 12.26; Cu, 11.91. Λ_m (cm^2 Ω^{-1} mol^{-1}): 53.0.

*Synthesis of BPIB–Zn(II)(**3**).* BPIB–Zn(II) was synthesized and purified by the same method. To a methanol solution (10 mL) of $ZnCl_2$ (0.817 g, 6 mmol) was added a DMSO solution (20 mL) of the ligand BPIB (2.06 g, 4 mmol), which resulted in a light-yellow solid. Yield 2.16 g (83%). IR (KBr, cm^{-1}): 3515, 3096, 1653, 1608, 1475, 1451, 1402, 814, 730, 643, 560. Anal. Calcd. for $[(C_{32}H_{18}N_8)_7(ZnCl_2)_8]$: C, 57.34; H, 2.71; N, 16.71; Cl, 12.09; Zn, 11.15. Found: C, 57.11; H, 2.79; N, 16.63; Cl, 12.27; Zn, 11.53. Λ_m (cm^2 Ω^{-1} mol^{-1}): 31.0.

3. Results and discussion

3.1. Synthesis and characterization

The synthesis route of the ligand and polymeric metal complexes is presented in Scheme 1. 1,10-Phenanthroline-5,



Scheme 1. The synthesis route of the ligand BPIB and polymeric metal complexes **2** and **3**.

6-dione was synthesized via a simple convenient method according to the published literature [18], and the convert ratio of the reaction is almost complete. The ligand was synthesized on the basis of the method for imidazole ring preparation established by Steck and Day [19]. The ligand BPIB is hardly soluble in common organic solvents, but had good solubility in DMF and DMSO. The π-conjugated polymeric metal complexes were synthesized using $CuCl_2 \cdot 2H_2O$ and $ZnCl_2$ via a standard approach in which the metal ions and the ligand coordinate to carry out the polymerization. The corresponding polymeric complexes were afforded in good yield at appropriate temperature. All polymeric metal complexes had very good solubility in DMF and DMSO, but were hardly soluble in common solvents, such as THF, CH_2Cl_2 and $CHCl_3$. The poor solubility of metal complexes suggests their polymeric nature. The phenanthroline and benzene rings of the ligand form π-conjugation to improve the electrons, holes transmission properties and thermal stability. The polymerization condition is mild. All the attempts to prepare good quality crystals were failed. All these complexes were fully characterized by ^{1}H NMR, FT-IR, UV−vis, elemental analysis and conductance measurements.

The FT-IR spectra of polymeric metal complexes are also shown in Fig. 1. By comparison with the ligand, they are found to have similar band. The H−N band in complexes **2** and **3** shifts to 3470 and 3515 cm^{-1}, and the C═N band in complexes **2** and **3** shifts to 1651 and 1653 cm^{-1}, respectively. Other bands in the complexes have some different extent of shifting, which are similar to the description given in the literature [20,21]. The shift of all bands of the complexes is attributed to the fact that the ligand was coordinated with the metal ions. In all the complexes a medium and/or weak band is

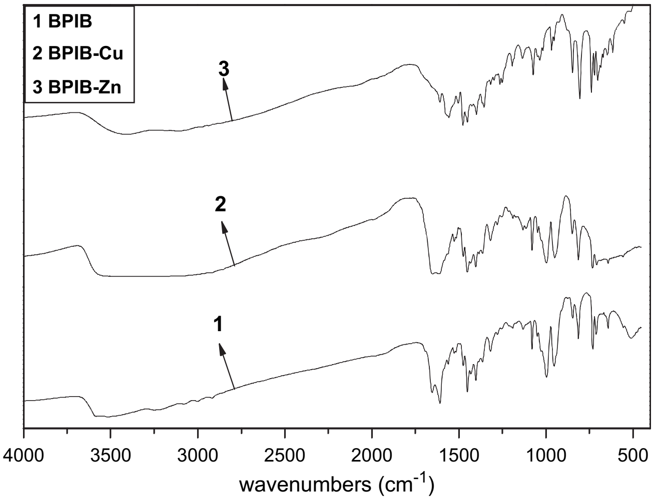


Fig. 1. FT-IR spectra (KBr) of the ligand BPIB and polymeric metal complexes **2** and **3**.

Table 1
Physical properties, GPC and TGA of complexes **2** and **3**

No.	Color	M.p. (°C)	$M_{n,GPC}$ [a] ($\times10^4$)	M_w/M_n	Yield (%)	T_d [b] (°C)	T_g [c] (°C)
2	Blue	>300	0.47	1.12	87	>342	232
3	Light yellow	>300	0.47	1.12	83	>341	256

[a] The measured M_n of the complexes **2** and **3** by GPC, PSt standard.

[b] The initial degradation temperature of all compounds by TGA at a heating rate of 20 K min^{-1}.

[c] Glass-transition temperature determined by DSC at a heat rate of 20 K min^{-1}.

observed at 557 and 560 cm^{-1} which can be attributed to the ν(M–N) modes [22]. In addition, the molar conductance values of the complexes **2** and **3** in DMSO solutions (10^{-4} M) are 53 and 31 cm^2 Ω^{-1} mol^{-1}, which is lower than the reported range for electrolytes in DMSO solutions [23]. These results are consistent with the light-electrolyte nature of the complexes. These data indicate that the ligand successfully chelate to metal ions.

The number average molecular weights, M_n, and polydispersities, M_w/M_n, are summarized in Table 1. Fig. 2 presents the GPC curves of complex **3**. GPC analysis was performed to determine the molecular weights and molecular weight distributions of the polymeric metal complexes, and the data indicate that all complexes have formed polymers.

According to the published literature [24–26], for example complex **2**, Cu(II) ions are bridged via BPIB ligand to form a main chain structure polymeric metal complex, and each Cu(II) ion coordinates four nitrogen atoms and the two chlorine atoms.

By comparison, the elemental analysis of both complexes with the content of C, H, N and M of the repeat unit of polymers, metal:ligand is in a 1:1 ratio for complexes **2**, **3**.

The degrees of polymerization (n) are 7 for complexes **2**, **3** according to the molecular weight of the repeat unit and the number average molecular weights, M_n, of polymers which were measured.

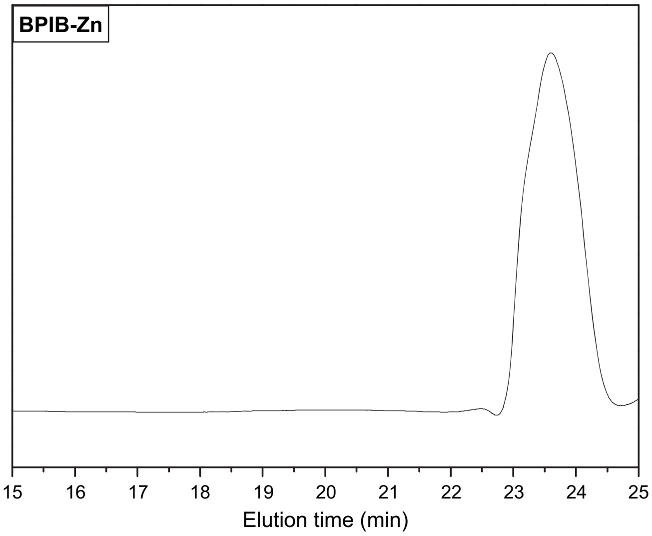


Fig. 2. GPC traces of complex **3**.

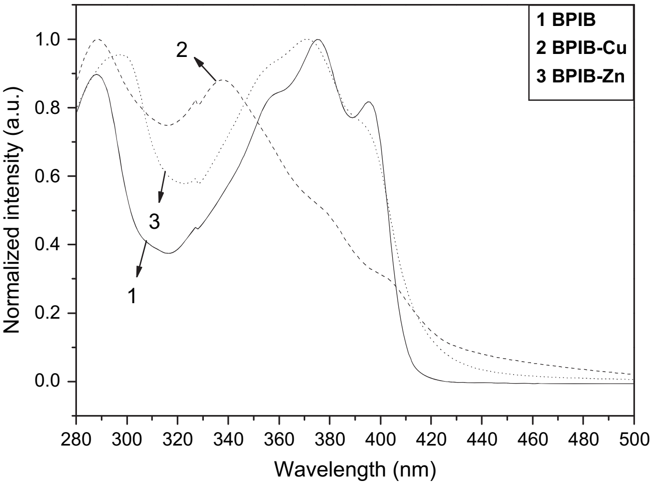


Fig. 3. UV–vis spectra of the ligand BPIB and polymeric metal complexes **2** and **3** in DMSO (1×10^{-4} M).

The results of measurement and calculation suggest that the formulas of complexes are $[(C_{32}H_{18}N_8)_7(MCl_2)_8]$ (M = Cu(II), Zn(II)). All these proofs indicate that the possible structures of the complexes are obtained as shown in Scheme 1.

3.2. Luminescence properties

3.2.1. UV–vis spectra

Fig. 3 shows the UV–vis absorption spectra of the complexes **1–3** in DMSO and Table 2 summarizes their peak positions in the UV–vis absorption spectra at room temperature. The absorption peak of complexes **2** and **3** in DMSO is located in 289 and 298 nm which is attributed to the π–π* intraligand transition of phenanthroline and benzene rings. There is one broad absorption peak observed at 338 and 375 nm in complexes **2** and **3**, which probably are assigned to the π–π* intraligand transition of the conjugated backbone (BPIB) and metal-to-ligand charge transfer (MLCT) transition. The broad band seems to be asymmetrically deformed, which indicates contributions of new molecular orbital coefficients (to form this band) in the absorption spectra of binuclear complexes. In the mononuclear complex, it is most likely that the π–π* transitions are perturbed by the presence of the metal center [27]. The absorption band of complexes **2** and **3** exhibits a blue-shift

Table 2
Optical properties of the ligand and complexes **1–3**

Compounds	Absorption (nm)	Fluorescence spectra			
		In solid state		In DMSO (10^{-4} M)	
		λ_{max} (em)	λ_{max} (ex)	λ_{max} (em)	λ_{max} (ex)
1	288, 375	483	403	422	397
2	289, 338	526	385	423	343
3	298, 370	517	395	434	382

comparing to that of the ligand, which might be due to make the density of electric charge asymmetry in ring, and decreases the extend of the symmetry of the ligand, after which was coordinated with metal ions. The UV–vis absorption spectra of both the complexes are tailing in the visible region, the reason for this phenomenon is the very weak electronic transition of the metal–metal interaction in both the complexes [28]. The absorption spectra of Zn complex is sharper than those of the Cu complex which might be due to the fact that Zn(II) (d^{10}) has more electrons than Cu(II) (d^9), as a result of which electronic transition takes place easily.

3.2.2. Fluorescence spectra

These luminescence properties of complexes **1**–**3** were investigated in solid state and in DMSO solution at room temperature. The fluorescence emission spectra of the complexes **1**–**3** are shown in Figs. 4 and 5. The luminescence data of ligand and polymeric metal complexes are summarized in Table 2. The fluorescence spectra of complexes **2** and **3** displayed maximum ($\lambda_{\mathrm{ex,\ max}}$) at 526 and 517 nm with excitation wavelengths at 385 and 395 nm in the solid state, which probably be assigned to metal-to-ligand change transfer (MLCT) transition. The fluorescence spectra of complexes **2** and **3** displayed maximum ($\lambda_{\mathrm{ex,\ max}}$) at 423 and 434 nm with excitation wavelengths at 343 and 382 nm in DMSO, respectively. These results indicate that the metal complexes emit purple/blue light with emission maximum ($\lambda_{\mathrm{f,\ max}}$) in 423 and 434 nm in DMSO solution, and blue/green in 526 and 517 nm in solid state, respectively.

Comparing with the ligand, polymeric metal complexes exhibit a red shift in solid state and in DMSO solution. The red shift is assigned based on the following two facts: the coordination of metal ions enhances the electron-accepting ability of ligand, decreases and stabilizes the electron transition energy of the intraligand charge transfer; on the other hand, the plane structure and rigidity of the coordination complexes are stronger than those of the ligand, which decrease energy loss and are apt to emit fluorescence. In addition, after being coordinated with metal ions, the π-conjugation extent of complexes **2** and **3** is more than that of the ligand, which apparently decreases the energy gap between the π and π* molecular orbital of the ligand [29]. The BPIB emits very weak fluoresce in solid state, which could be due to the superposition of the π–π* transition of phenanthroline. By comparison with the ligand it was found that the fluorescent emission of both the complexes is enhanced, implying that the energy transfer from metal ion to phenanthroline enhances emission.

In solid state, although coordinating with the same ligand BPIB, the maximum emission of complex **2** ($\lambda_{\max} = 526$ nm) has a larger red shift than **3** ($\lambda_{\max} = 517$ nm). It indicates that the metal ion plays a key role in fluorescent emission of complexes, because of the fact that Cu(II) (d^9) has few electrons than Zn(II) (d^{10}) which can accept electron from ligand easily. Similar to other complexes, the luminescence for the ligand is predominantly due to π–π* transition fluorescence [30,31].

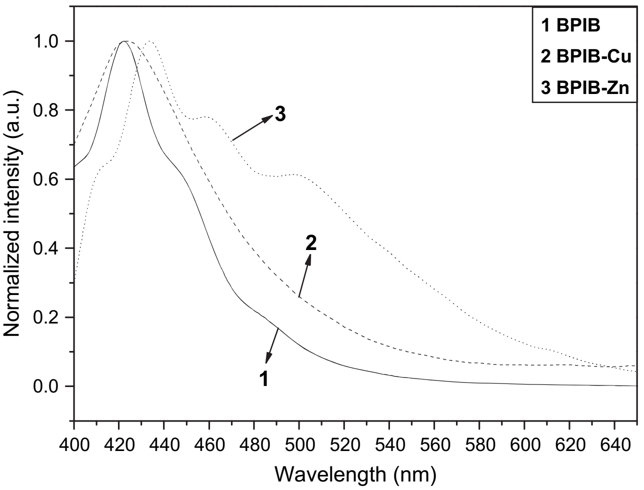


Fig. 4. Emission spectra of the ligand BPIB and polymeric metal complexes **2** and **3** in DMSO (1×10^{-4} M).

To understand the effect on the emission energy on going from the free ligand to the complex, a similar theoretical reason has been studied [16]. The highest occupied molecular orbital (HOMO) of complex is a π orbital, localized on the non-coordinating nitrogen atom and carbon atom in the phenanthroline and imidazole rings, owing to the low symmetry. Comparing with the free ligand, the energy of the HOMO level of the complex is much lower than that of the free ligand. The lowest unoccupied molecular orbital (LUMO) of the complex is a π* orbital consisting of mostly atomic orbitals from one of the pyridyl rings. There are essentially few contributions from the imidazole ring in the LUMOs of the complexes. The role of the metal ion is to increase coplanarity, conformational rigidity in the molecule structure and to decrease the

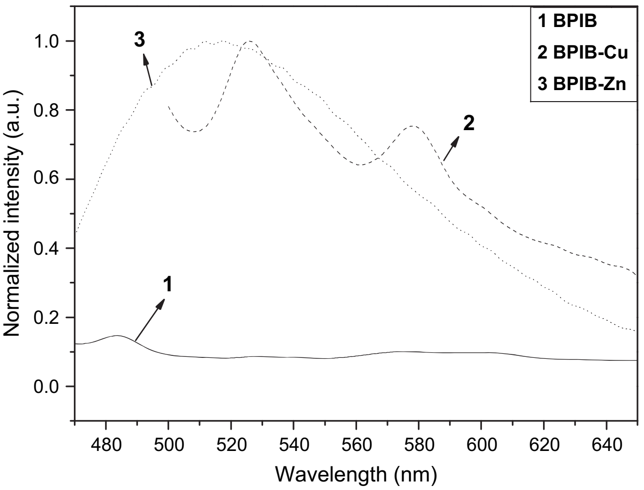


Fig. 5. Emission spectra of the ligand BPIB and polymeric metal complexes **2** and **3** in the solid state at appropriate temperature.

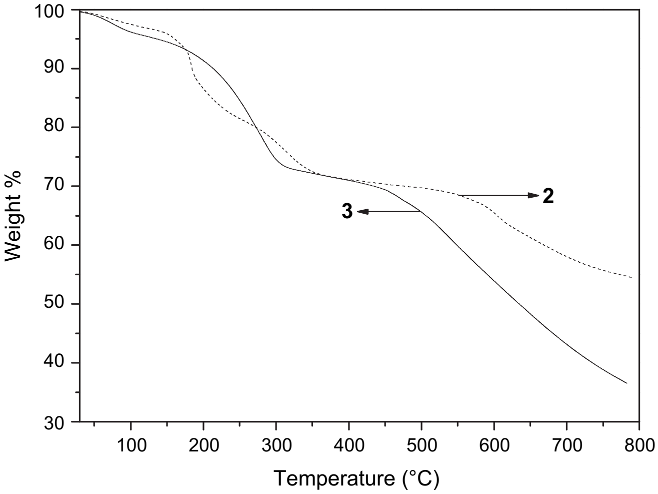


Fig. 6. TGA curves of complexes **2** and **3** at 20 K min^{-1} under Ar.

π–π* energy gap. The results lead to a red shift in the transition energy from ligand to metal center.

3.3. Thermal stability

The thermal analyses are shown in Table 1. For example, in complexes **2** and **3** (Fig. 6), the first decomposition stages occurred in the range 30–342 and 30–341 °C with a corresponding weight loss 4.0% for **2** and 4.5% for **3**, respectively, which were assigned to the loss of absorbed water molecules. The second stages of decomposition were observed at 341–594 °C (23.5% weight loss) and 342–497 °C (22.3% weight loss), which may be caused by the main chain decomposition of polymeric complexes. While the third stages occurred in the temperature ranges 594–800 and 497–800 °C with a corresponding weight loss 17.47 and 16.68%. The char yield % is about 55 wt% even at 800 °C. The DSC outcomes (T_g) (Table 1) of complexes **2** and **3** are found to be 232 and 256 °C (Table 1).

From TGA results, polymeric complexes are found to have formed stable five-member chelate ring [32–34], and have excellent thermal stabilities, which may be attributed to the fact that the M–N bond is highly polarized [35]. The T_g data indicate that all the complexes possess a very high transition temperature, which may serve as an advantage for PLED device fabrication because the use of the materials with high transition temperature as the active emissive layer or carrier transporting layer may provide the device with greater longevity [36].

4. Conclusion

Two novel main chain polymeric metal complexes **2** and **3** were synthesized and characterized. The fluorescence spectra of complexes **2** and **3** displayed blue/green luminescence at 526 and 517 nm in the solid state and purple/blue luminescence at 423 and 434 nm in DMSO solution. The two polymeric metal complexes have good emission properties and thermal stabilities, and thus can be used as PLEDs.

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