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Selective Detection of Transition Metal Ions using Ruthenium(II)-Phenanthroline-Biquinoline-4,4'-Dicarboxylic Acid Complex: A Colorimetric and Spectrophotometric Approaches

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A novel ruthenium(II) polypyridyl complex $[Ru(dmphen)_2(H_2bqda)](PF_6)_2$ (dmphen = 2,9-dimethyl-1,10-phenanthroline, H_2bqda = 2,2′-biquinoline-4,4′-dicarboxylic acid and PF_6 = counter ion) was synthesized and evaluated for its cation-sensing properties. The complex was characterized using UV-Visible and FTIR spectroscopy as well as ^1H and ^{13}C NMR spectroscopy. High-resolution mass spectrometry and elemental analysis further supported the proposed molecular composition and coordination structure. Cation-sensing studies were conducted in aqueous HEPES buffer (pH 7.2) containing 1% acetonitrile using a range of metal ions including Cu^{2+}, Fe^{3+}, Co^{2+}, Ba^{2+}, Cd^{2+}, Li^+, Mg^{2+}, Ni^{2+}, Zn^{2+} and Zr^{2+}. The complex showed distinct colorimetric and spectrophotometric responses toward Cu^{2+}, Fe^{3+}, Co^{2+} while exhibiting negligible changes in the presence of the other cations. UV-Visible titration studies revealed bathochromic shifts in the metal-to-ligand charge-transfer band and the appearance of new absorption bands suggesting interactions between the complex and the target cations. Binding constants (K_b) calculated using the Benesi-Hildebrand method supported a 1:1 binding model with the highest apparent affinity observed for Cu^{2+} ($K_b = 3.18 \times 10^4\ M^{-1}$). These findings highlight the potential of the synthesized Ru(II) complex as a colorimetric and spectrophotometric probe for Cu^{2+}, Fe^{3+}, Co^{2+} under the experimental conditions investigated.

Keywords: Ruthenium(II) complex, Colorimetric sensing, Spectrophotometric sensing, Binding constant.

INTRODUCTION

Designing chemosensors capable of selectively and sensitively detecting metal cations remains an important challenge in analytical science [1], biology [2], medicine and environmental monitoring [3]. This interest arises from the essential roles of metal ions in biological functions and their widespread involvement in industrial and environmental processes. Among the various metal-ion recognition systems [4], transition metal complexes containing nitrogen-donor ligands have attracted considerable attention due to their versatile coordination properties and tunable photophysical and redox characteristics [5,6]. In particular, multifunctional chelating ligands offer potential advantages in metal-ion recognition by providing suitable coordination sites and enabling measurable changes in spectroscopic properties [7,8]. When incorporated into metal complexes, these ligands can contribute to the development of chemosensors capable of detecting target metal ions through changes in their optical or physico-chemical responses [9,10].

Ruthenium(II) polypyridyl complexes have been widely investigated for chemical sensing applications due to their favourable and tunable photophysical properties. These complexes often exhibit large Stokes shifts, visible-light excitation, relatively long excited-state lifetimes and good photostability compared with many conventional organic luminophores [11]. Such characteristics make them promising candidates for the development of optical chemosensors. The detection of metal cations is particularly important because of their roles in biological systems, environmental processes and industrial applications [12,13]. Accordingly, several Ru(II) polypyridyl complexes have been developed for the recognition and quantification of Cu^{2+} ions [14]. Despite these advances, limitations remain in the practical application of many reported sensors [15,16]. Some probes require predominantly organic solvent media such as acetonitrile, ethanol or methanol, while others

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exhibit slow response times or limited suitability for aqueous and biologically relevant environments. These limitations may restrict their applicability in environmental and biological analysis [17,18].

Despite substantial progress in metal-ion sensing, the development of chemosensors with adequate sensitivity and selectivity for the reliable detection of Cu^{2+}, Fe^{3+}, Co^{2+} remains an important research objective [19,20]. Potential challenges include insufficient sensitivity at low analyte concentrations and interference from competing metal ions, which can affect the reliability of analytical measurements. Furthermore, the influence of quinoline-derived ligands and their functional groups on metal-ion recognition in Ru(II) polypyridyl systems warrants further investigation. A clearer understanding of the interactions between these complexes and target cations is also necessary to establish the relationship between ligand structure, coordination behaviour and spectroscopic response. Addressing these aspects may contribute to the development of Ru(II)-based chemosensors for applications in environmental monitoring, industrial effluent analysis and related analytical fields.

In present study, a ruthenium(II) polypyridyl complex, $[Ru(dmphen)_2(H_2bqda)]^{2+}$, was synthesized and investigated for its cation-sensing properties. The complex incorporates dmphen (2,9-dimethyl-1,10-phenanthroline) and H_2bqda (2,2′-biquinoline-4,4′-dicarboxylic acid) as coordinated ligands, with hexafluorophosphate (PF_6^-) serving as the counterion. The carboxylic acid functionalities of H_2bqda provide potential sites for additional interactions with metal cations, while the Ru(II) coordination framework offers a spectroscopically responsive platform through its metal-to-ligand charge-transfer (MLCT) transitions. An important feature of this investigation is the evaluation of the complex under predominantly aqueous conditions using HEPES buffer containing only 1% acetonitrile. Its responses toward Cu^{2+}, Fe^{3+} and Co^{2+} were examined using colorimetric and UV-Visible spectrophotometric measurements, together with binding constant and detection limit analyses. This study therefore explores the potential of a carboxyl-functionalized Ru(II) polypyridyl complex as an optical receptor for selected metal cations and examines its sensing behaviour under the experimental conditions employed.

EXPERIMENTAL

All chemicals were used as received without further purification. Ruthenium(III) chloride ($RuCl_3·3H_2O$), 2,9-dimethyl-1,10-phenanthroline (dmphen), 2,2′-biquinoline-4,4′-dicarboxylic acid (H_2bqda) and the perchlorate salt of cations *viz.* Cu^{2+}, Fe^{3+}, Co^{2+}, Ba^{2+}, Cd^{2+}, Li^{2+}, Mg^{2+}, Ni^{2+}, Zn^{2+} and Zr^{2+}, methanol, acetonitrile and NH_4PF_6 were supplied by Sigma-Aldrich.

Synthesis of $[Ru(dmphen)_2(H_2bqda)]^{2+}$ complex: The $[Ru(dmphen)_2(H_2bqda)]^{2+}$ complex was synthesized by refluxing $RuCl_3·3H_2O$, dmphen, H_2bqda and ethylene glycol for 8 h in a nitrogen-filled atmosphere. The resultant solution was cooled and transferred into aqueous NH_4PF_6 solution to isolate the reddish-brown complex as the salt of hexafluorophosphate (Fig. 1). The $[Ru(dmphen)_2(H_2bqda)]^{2+}$ complex thus

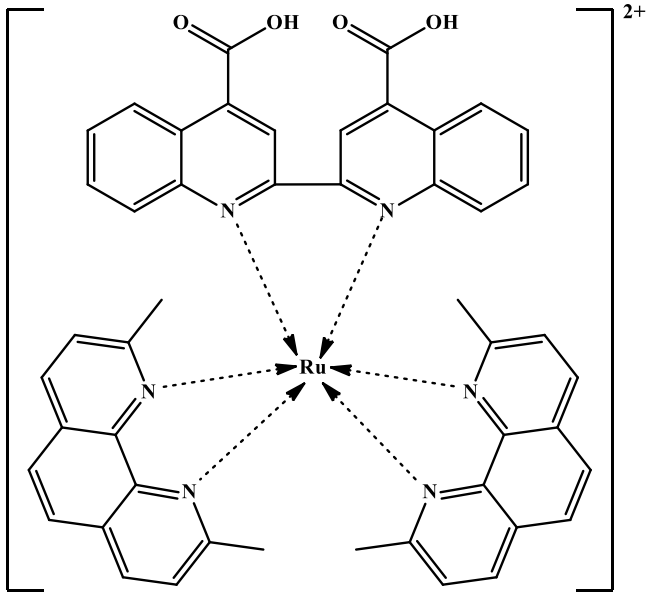


Fig. 1. Structure of $[Ru(dmphen)_2(H_2bqda)]^{2+}$ complex

obtained was filtered, rinsed with water and vacuum dried. It was then recrystallized using a 9:1 mixture of methanol and acetonitrile.

Characterization: The UV-Visible spectrum of the Ru(II) complex was recorded using a Shimadzu UV-1800 double-beam spectrophotometer. FTIR spectral analysis was carried out with a Shimadzu FTIR double-beam spectrometer. The ^1H and ^{13}C NMR spectra were recorded using a Bruker AVANCE III spectrometer operating at 400 MHz for ^1H NMR and 125 MHz for ^{13}C NMR in DMSO. HRMS mass spectrum was recorded using Bruker Autoflex max LRF mass spectrometer. The percentage composition of C, H, O and N was measured by elemental analysis using Truspec Micro analyzer.

Cation sensing studies: Spectrophotometric titrations were employed to investigate the interactions between the $[Ru(dmphen)_2(H_2bqda)]^{2+}$ complex and various cations in an aqueous HEPES buffer (pH 7.2) with 1% CH_3CN. A stock solution of the complex (5×10^{-4} M) was prepared, from which 2.5 mL was taken for the titration experiment. Aliquots of cation solutions in microliter volumes were added stepwise using a micropipette. Following each addition, the solution was stirred and the UV-Visible spectrum was recorded immediately. The procedure was repeated until no further significant changes in the spectra were detected. The binding constant (K_b) of $[Ru(dmphen)_2(H_2bqda)]^{2+}$ complex with cations were calculated using Benesi-Hildebrand method.

RESULTS AND DISCUSSION

The UV-visible spectrum of the $[Ru(dmphen)_2(H_2bqda)]^{2+}$ complex shows bands at 210, 286 and 428 nm (Fig. 2a). The sharp peaks at 210 and 286 nm indicates the π-π* transition of the ligands. The peak at 428 nm represents the metal to ligand charge transfer (MLCT).

The FTIR spectrum of $[Ru(dmphen)_2(H_2bqda)]^{2+}$ complex exhibits characteristic absorption bands at 3473, 2960, 2856, 2675, 1973, 1635, 844, 653 and 557 cm^{-1} (Fig. 2b). The broad

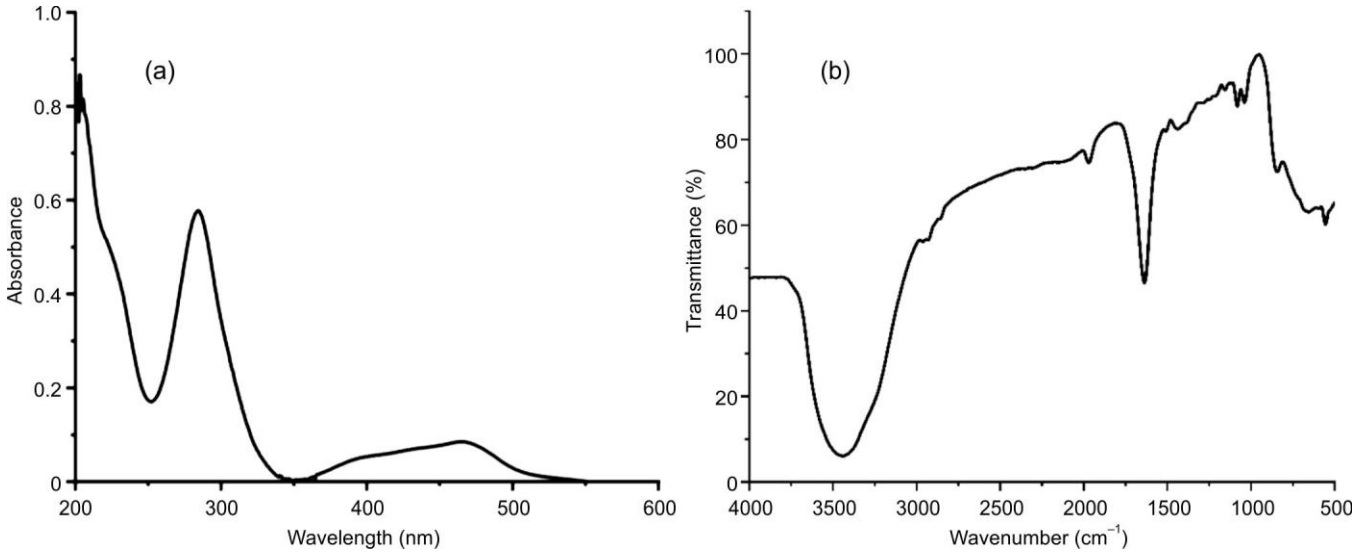


Fig. 2. (a) UV-Visible and (b) FTIR spectra of $[Ru(dmphen)_2(H_2bqda)]^{2+}$ complex

band at 3473 cm^{-1} is attributed to the O–H stretching vibration of the carboxylic acid groups. The weak bands at 2960 and 2856 cm^{-1} are associated with C–H stretching vibrations. The band at 1973 cm^{-1} falls within the combination and overtone region and is not assigned to a specific functional group. The strong absorption band at 1635 cm^{-1} is attributed to the C=O stretching vibration of the carboxylic acid groups in H_2bqda. The intense band at 844 cm^{-1} is characteristic of the P–F stretching vibration of the PF_6^- counterion [21]. The bands observed at 653 and 557 cm^{-1} may be associated with metal–ligand coordination-related vibrations involving the Ru–N bonds [22].

The ^{1}H NMR spectrum of $[Ru(dmphen)_2(H_2bqda)]^{2+}$ complex displays a broad resonance at δ 11.13 ppm attributeable to the carboxylic acid protons of H_2bqda (Fig. 3a). The doublet at δ 8.22 ppm represents H^3, doublet at δ 8.03 ppm represents H^4, doublet at δ 7.9 ppm indicates H^5, doublet at δ 8.00 ppm depict H^6, doublet at δ 7.19 ppm indicates $H^{6'}$ and $H^{13'}$, doublet at δ 8.32 ppm corresponds to $H^{7'}$ and $H^{12'}$, doublet δ 7.92 ppm indicates $H^{9'}$ and $H^{10'}$, consists of multiple overlapping signals. The sharp singlet at δ 2.53 ppm corresponds to $H^{15'}$ and $H^{16'}$ of the methyl groups present in dmphen ligand. The observed downfield shifts and peak broadening are consistent with coordination of the polypyridyl ligands to Ru(II) complex. The chemical shift value of the ligands ranges from δ 2.53-11.13 ppm.

The ^{13}C NMR spectrum of $[Ru(dmphen)_2(H_2bqda)]^{2+}$ shows a chemical shift δ 167.7 ppm indicates the presence of C=O group of the carboxylic acid (Fig. 3b). The chemical shift at δ 24.86 ppm indicates the presence of methyl group. The signals range δ 126.68-150.08 ppm indicates the carbon atoms present in the benzene and pyridine moieties. The carbon signal present in pyridine rings at δ 165.97 ppm represents C^{11}. The carbon atoms of the benzene rings show characteristic chemical shift with C^2 and $C^{2'}$ appearing at δ 150.08 ppm, C^3, C^5 and $C^{5'}$ at δ 127.62 ppm, C^4 at δ 129.18 ppm, C^6, C^7 and $C^{6'}$ at δ 126 ppm and another C^4 signal observed at δ 137.07 ppm.

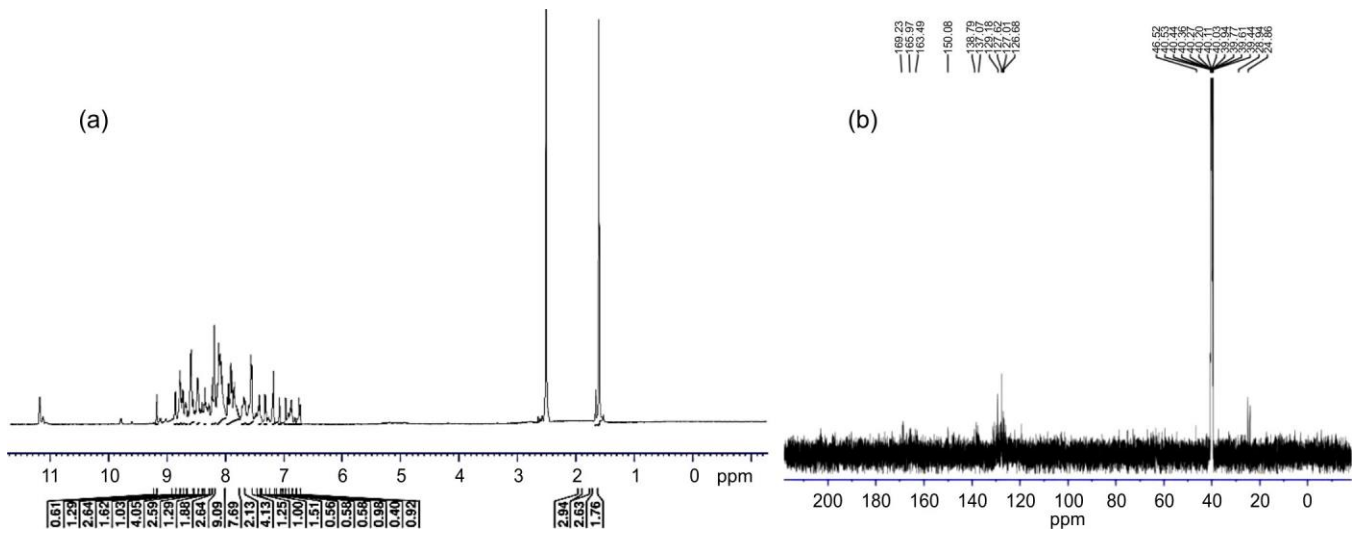


Fig. 3. (a) ^{1}H NMR and (b) ^{13}C NMR spectra of $[Ru(dmphen)_2(H_2bqda)]^{2+}$ complex

The HRMS mass spectrum of $[Ru(dmphen)_2(H_2bqda)](PF_6)_2$ depicts the M^+ peak at 1162.97. The M^+ peak with elimination of 1 and 2 PF_6^- counter ion is at 1018.01 and 857.16, respectively (Fig. 4). Elemental analysis revealed the percentage composition of carbon (50%), hydrogen (3.1%), oxygen (5.6%) and nitrogen (7.24%). These results, together with the HRMS data, were consistent with the proposed molecular formula of the synthesized complex, $C_{48}H_{36}O_4N_6Ru_1P_2F_{12}$. The experimental results showed agreement with the corresponding theoretical values, which further support the proposed composition of the complex (Table-1).

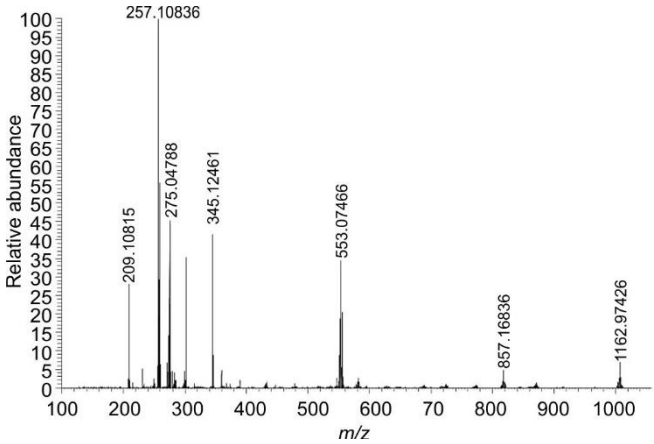


Fig. 4. HRMS spectrum of $[Ru(dmphen)_2(H_2bqda)](PF_6)_2$ complex

TABLE-1
ELEMENTAL DATA OF
$[Ru(dmphen)_2(H_2bqda)](PF_6)_2$ COMPLEX

Element	Experimental (%)	Calculated (%)
C	50.0	50.05
H	3.1	3.15
N	7.24	7.3
O	5.60	5.56

Cation sensing studies: The sensing capacity of $[Ru(dmphen)_2(H_2bqda)]^{2+}$ complex towards various cation are identified by visual investigation of the cations induced colour change of the receptors in 1% aqueous CH_3CN and HEPES buffer (7.2 pH) solution. The colorimetric titration of the $Ru(dmphen)_2(H_2bqda)]^{2+}$ complex with Cu^{2+}, Fe^{3+}, Co^{2+}, Ba^{2+}, Cd^{2+}, Li^+, Mg^{2+}, Ni^{2+}, Zn^{2+} and Zr^{2+} shows significant colour change in Cu^{2+}, Fe^{3+} and Co^{2+}. With incremental addition of Cu^{2+}, Fe^{3+} and Co^{2+} cations, the colour varies from brown to blue, yellowish orange and pink. The cations such as Ba^{2+}, Cd^{2+}, Li^+, Mg^{2+}, Ni^{2+}, Zn^{2+} and Zr^{2+} does not show any change in colouration (Fig. 5). The binding interactions between the complex and the metal cations were investigated using UV-Visible spectrophotometric titrations. Upon the gradual addition of metal cations, an increase in absorbance was observed in the metal-to-ligand charge-transfer (MLCT) region, indicating interactions between the complex and the added cations. A slight bathochromic shift was observed from 428 to 448 nm. New absorption bands appeared at 617, 448 and 638 nm in the presence of Cu^{2+}, Fe^{3+} and Co^{2+}, respectively (Fig. 6a-c). In contrast, no comparable new bands were observed for the other cations investigated. The observed bathochromic shifts and changes in the absorption profiles may be attributed to alterations in the electronic environment of the $Ru(dmphen)_2(H_2bqda)]^{2+}$ complex upon interaction with the target cations [23].



Fig. 5. Titration of $[Ru(dmphen)_2(H_2bqda)]^{2+}$ complex with cations displaying colour change in Cu^{2+}, Fe^{3+} and Co^{2+}cations

The binding constants (K_b) for the interactions between the receptor and metal cations were evaluated using the Benesi-Hildebrand equation. Among the cations investigated, Cu^{2+} exhibited the highest binding constant of 3.18×10^4 M^{-1} (Table-2). For the investigated cations, plots of $1/\Delta A$ against 1/[C] showed good linearity, supporting a 1:1 binding model under the experimental conditions employed (Fig. 7a-b). The limits of detection (LOD) and quantification (LOQ) for Cu^{2+}, Fe^{3+} and Co^{2+} were determined from the corresponding absorbance calibration curves obtained by the incremental addition of each cation to the $Ru(dmphen)_2(H_2bqda)]^{2+}$ complex. The calculated values are shown in Table-3.

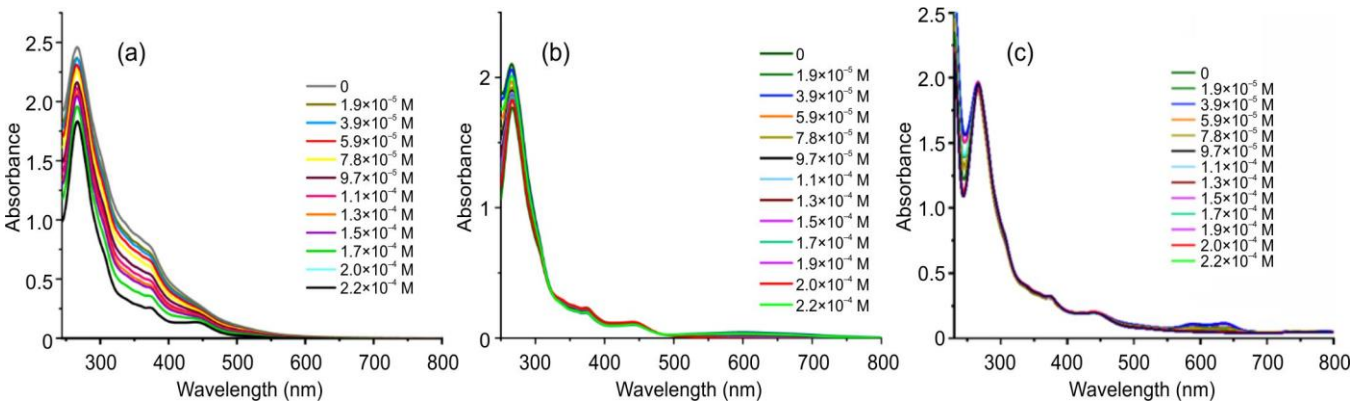


Fig. 6. Absorption spectra of $[Ru(dmphen)_2(H_2bqda)]^{2+}$ complex with incremental addition of (a) Fe^{3+} (b) Cu^{2+} and (c) Co^{2+} cation

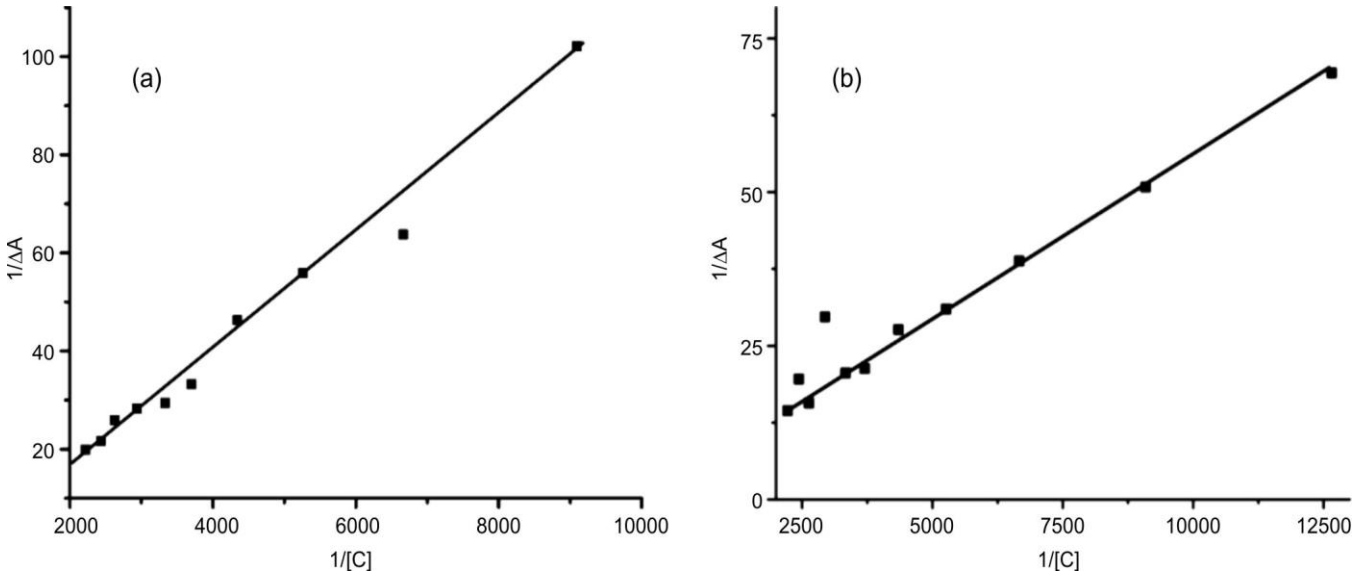


Fig. 7. Benesi-Hildebrand plots of $[Ru(dmphen)_2(H_2bqda)]^{2+}$ complex with Cu^{2+} ion (a) and (b) Co^{2+} ion (b)

TABLE-2
BINDING CONSTANTS (K_b) OF $[Ru(dmphen)_2(H_2bqda)]^{2+}$ COMPLEX WITH CATIONS

Cations	Binding constant (K_b) (M^{-1})	Cations	Binding constant (K_b) (M^{-1})
Cu^{2+}	3.18×10^4	Li^+	0.20×10^4
Fe^{3+}	1.14×10^4	Mg^{2+}	0.08×10^4
Co^{2+}	1.68×10^4	Ni^{2+}	0.29×10^4
Ba^{2+}	0.47×10^4	Zn^{2+}	0.53×10^4
Cd^{2+}	0.03×10^4	Zr^{2+}	0.04×10^4

TABLE-3
DETECTION AND QUANTIFICATION LIMITS OF STUDIED CATIONS WITH $[Ru(dmphen)_2(H_2bqda)]^{2+}$ COMPLEX

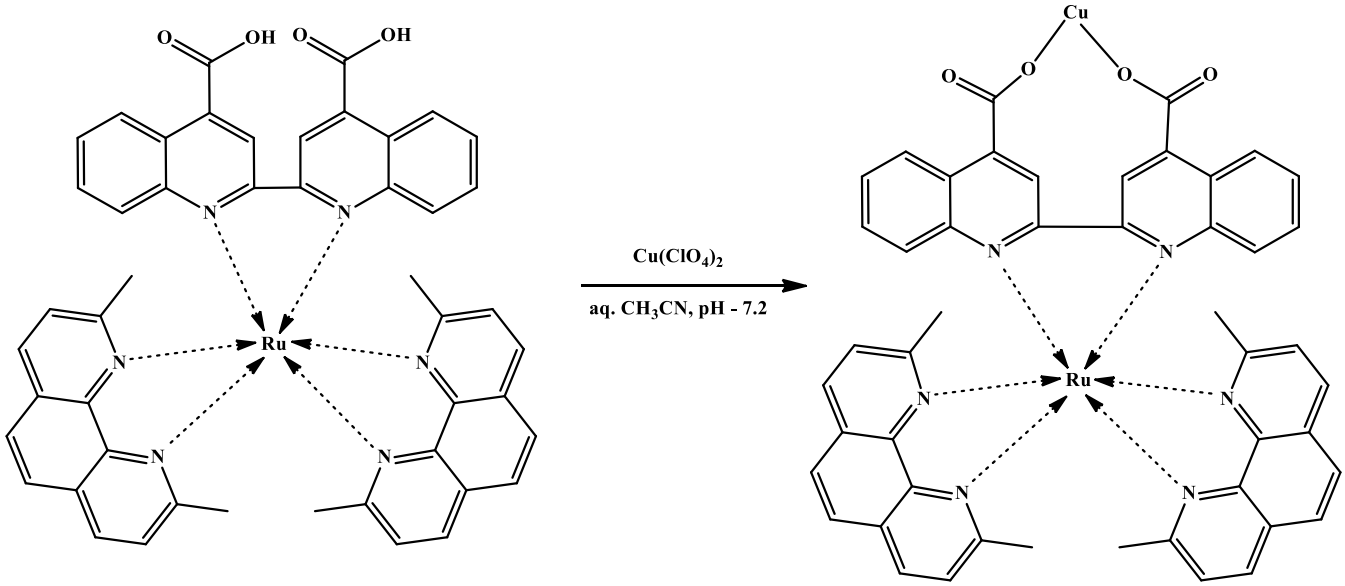
Cations	Concentration (M^{-1})	
	Detection limit	Quantification limit
Cu^{2+}	7.15×10^{-5}	2.16×10^{-4}
Fe^{3+}	2.03×10^{-5}	6.13×10^{-5}
Co^{2+}	1.22×10^{-5}	3.69×10^{-5}

Although measurable binding constants were obtained for Ba^{2+}, Cd^{2+}, Li^+, Mg^{2+}, Ni^{2+}, Zn^{2+} and Zr^{2+}, their binding affinities were considerably lower than those observed for Cu^{2+}, Fe^{3+} and Co^{2+}. Moreover, only Cu^{2+}, Fe^{3+} and Co^{2+} exhibited distinct colorimetric responses and characteristic spectral changes under the experimental conditions employed. Therefore, the selective response of the complex is based on the combination of relatively higher binding affinities and distinguishable spectroscopic and colorimetric changes rather than the complete absence of interactions with other cations. The sensing performance of the synthesized complex was also compared with that of previously reported Ru(II) complexes, and the comparative results are shown in Table-4.

The proposed mechanism of interaction between the metal cations and the $Ru(dmphen)_2(H_2bqda)]^{2+}$ complex is shown in **Scheme-I**. The carboxylic acid groups of H_2bqda may undergo deprotonation in the aqueous HEPES buffer at pH 7.2, generating carboxylate groups that can participate in interactions with the target cations. In case of Cu^{2+}, coordination with the available carboxylate oxygen atoms may alter the electronic environment of the Ru(II) complex and influence its metal-to-ligand charge-transfer (MLCT) transitions. These changes may account for the observed bathochromic shift and the appearance of a new absorption band. Similar interactions may occur in the presence of Fe^{3+} and Co^{2+}, which result in their respective spectroscopic responses.

TABLE-4
COMPARISON OF SELECTED Ru(II)-BASED METAL-ION SENSING COMPLEXES WITH $[Ru(dmphen)_2(H_2bqda)]^{2+}$ COMPLEX

Ru(II)-based sensing system	Target ion	Sensing mode/Medium	Binding constant	Detection limit	Ref.
Quinoline-appended Ru(II)-polypyridyl complex	Cu^{2+}	Luminescence ON/OFF; colorimetric Aqueous medium	–	50.67 nM	[24]
Dinuclear Ru(II)-azacrown ether complex	Hg^{2+}	Luminescence quenching H_2O/CH_3CN (1:1)	5.02×10^4 M^{-1}	2×10^{-7} M	[25]
Ru(II)-bipyridine/imidzole complex	Cu^{2+}	Luminescence quenching Aqueous solution	–	83.3 nM	[26]
$[Ru(dmphen)_2(H_2bqda)]^{2+}$	Cu^{2+}	Colorimetric + UV-Visible	3.18×10^4 M^{-1}	7.15×10^{-5} M	Present study
	Fe^{3+}	spectrophotometric	1.14×10^4 M^{-1}	2.03×10^{-5} M	
	Co^{2+}	HEPES buffer/1% CH_3CN	1.68×10^4 M^{-1}	1.22×10^{-5} M	



Scheme-I: Proposed mechanism for the coordination of Cu^{2+} with $[Ru(dmphen)_2(H_2bqda)]^{2+}$ complex

Conclusion

In conclusion, the $[Ru(dmphen)_2(H_2bqda)]^{2+}$ complex was successfully synthesized and characterized using UV-Visible and FTIR, ^1H and ^{13}C NMR spectroscopies, HRMS and elemental analysis. The results supported the proposed molecular composition and coordination structure. The complex exhibited a characteristic metal-to-ligand charge-transfer (MLCT) band at 428 nm, which showed distinct spectral changes upon interaction with selected metal cations. Cation-sensing studies revealed distinguishable colorimetric and spectrophotometric responses toward Cu^{2+}, Fe^{3+} and Co^{2+}, accompanied by a bathochromic shift from 428 to 448 nm and the appearance of additional absorption bands at 617, 448 and 638 nm, respectively. Benesi-Hildebrand analysis supported a 1:1 binding model for these cations, with binding constants of 3.18×10^4, 1.14×10^4 and 1.68×10^4 M^{-1} for Cu^{2+}, Fe^{3+} and Co^{2+}, respectively, which indicate the highest apparent binding affinity toward Cu^{2+}. The corresponding limits of detection were 7.15×10^{-5} M for Cu^{2+}, 2.03×10^{-5} M for Fe^{3+} and 1.22×10^{-5} M for Co^{2+}. These findings demonstrate the potential of the synthesized Ru(II) complex as a colorimetric and spectrophotometric probe for the recognition of Cu^{2+}, Fe^{3+} and Co^{2+} under the experimental conditions investigated.

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CONFLICT OF INTEREST

The authors declare that there is no conflicts of interest regarding the publication of this article.

DECLARATION OF AI-ASSISTED TECHNOLOGIES

During the preparation of this manuscript, the authors used an AI-assisted tool(s) to improve the language and no data, results or scientific conclusions were generated by AI. All analyses, interpretations and conclusions are the authors' own. The authors reviewed and edited the content and take full responsibility for the published work.

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