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ORIGINAL ARTICLE

Dual Functional Copper(II) Isophthalate Coordination Polymer for Selective Sensing of Fe^{3+} Ions and Methanol

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Abstract
Fluorescent chemosensors are becoming attractive due to their unique features of sensitivity and selective detection of analytes in the aqueous and vapour phases. Inorganic and organic pollutants are of major concern because of their adverse health effects. In this work, a coordination compound $[Cu(ISO)_2(H_2O)_2]\cdot DMF$ **1** (where $H_2ISO = 2,4$-benzenedicarboxylic acid) was synthesized by oven-heating at 100 °C. Compound **1** was characterized using elemental analysis, UV/Visible and Fourier transform infrared (FTIR) spectroscopies, Thermogravimetric analysis (TGA), and Scanning Electron Microscopy (SEM). The compound is thermally stable up to 356 °C. Compound **1** showed excellent fluorescent properties with emission centred at 435 nm. This is attributed to ligand to metal charge transfer (LMCT) arising from the π-electron rich backbone of the free ligand. The compound was further studied for detection of metal cation and volatile organic compounds (VOCs). Results show selective recognition of Fe^{3+} ions and methanol by drastic quenching of fluorescent intensity. High Stern Volmer constants and low limits of detection makes the compound a viable material for ion and vapour detection.

Keywords Chemosensors · Coordination · Emission · Luminescence · Quenching

1 Introduction

The detection and analysis of specific ions and organic molecules play a crucial role in various scientific and technological fields, ranging from environmental monitoring to biomedical diagnostics [1, 2]. With the rapid growth of modern industry and a plethora of human activities, a number of inorganic pollutants have been released, causing detrimental effects to the environment while also posing a constant threat to human health [3, 4]. Effective detection, monitoring and removal of these pollutants is important to ensure the sustainability of humans and the environment [5–7]. Coordination polymers have emerged as promising materials for the detection of these target analytes, owing to their unique structural and functional properties. They are composed of metal ion or clusters coordinated to organic ligands. These materials exhibit high porosity, large surface areas and tunable pore sizes making them excellent candidates for gas storage, catalysis and sensing applications. By carefully selecting the metal ion and ligand components, coordination polymers can be tailored to exhibit specific chemical and physical properties, enabling their use as sensors for various analytes [8].

Fe^{3+} ions are of particular interest due to their crucial roles in biological processes and their widespread occurrence in natural systems. Detection and quantification of Fe^{3+} ions are important for understanding iron metabolism, monitoring water quality and diagnosing diseases associated with iron dysregulation. Similarly, the detection of methanol, a volatile and toxic organic solvent, is significant in various contexts. Methanol is widely used in industrial processes, laboratories and as a fuel source. However, its accidental release or ingestion can lead to severe health problems, including organ damage and even death [9, 10].

Traditional detection methods such as colorimetric assays, electrochemical method, spectroscopic techniques, inductively coupled plasma mass spectrometry, may exhibit limitations in terms of sensitivity, selectivity,

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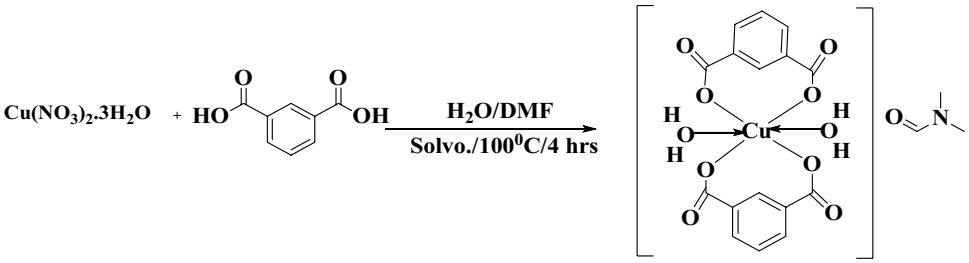
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Scheme 1 Preparation of $[Cu(ISO)_2(H_2O)_2]\cdot DMF$



ease of use or high cost. They may require complex sample preparation, lack of specificity towards target analyte or suffer interference by other compounds present in the sample matrix. In contrast, coordination polymers offer promising alternative by addressing these limitations and providing enhanced performance in terms of sensitivity, selectivity, fast response and versatility [11]. The design and synthesis of coordination polymers can be tailored to incorporate binding sites capable of interacting with the analytes. Upon binding to the analytes, they undergo structural or optical changes that can be easily monitored, enabling the sensitive and selective detection and quantification of the target analytes.

In the past, Song et al. [12], prepared a Copper coordination polymer based on isophthalate bridging ligand and imidazole capping ligand. The structural analysis revealed 1D zig-zag chains connecting into 3D framework by hydrogen bonding and π–π interactions. Recently, Chen et al. [11] reported the selective sensing of Fe^{3+} using an anionic metal organic frameworks (MOFs) having nitrogen and carboxylate oxygen sites. It was observed that Fe^{3+} could selectively quench the emission intensity of the MOF over other metal ions whose effects were negligible. The high Stern–Volmer (K_{SV}) obtained also suggest strong interactions between $Fe(NO_3)_3$ and the host framework. Yousuf et al. [13] also reported a novel Cu(II) 1D coordination polymer based on 1,2-diamminocyclohexane for sensing inorganic oxo-anions (CrO_4^{2-} and $Cr_2O_7^{2-}$) with high quenching efficiency as stipulated by the high Stern–Volmer constants. The compound was found to be stable after several usage. Ridhi and his group used a copper phthalocyanine thin films for vapour sensing. These films exhibit maximum response towards methanol compared to the other VOCs [14].

As continuation of our work on luminescent detection of nitroaromatic compounds [15] and other environmental pollutants [16] we report herein the synthesis of a multifunctional coordination polymer assembled from aromatic carboxylate ligand for selective detection of Fe^{3+} ion and methanol vapour. The novelty of the study lies in the ability of the coordination polymer to selectively detect Fe^{3+} ion in aqueous solution and methanol in the vapour phase.

2 Materials and Methods

2.1 Materials

All reagents and chemicals are of analytical grade and were used as received without further purification. 2,4-Benzenedicarboxylic acid was purchased from Sigma Aldrich, Germany. All metal salts were supplied by Alfa Aesar, UK. Solvents: ethanol (98.8%), *N,N*-dimethyl formamide (99.0%) were obtained from Central Drug House (P) Ltd New Delhi India. The samples were characterized by elemental analysis using an Exeter Analytical CE-440 Elemental Analyser. The infrared spectra were recorded using a Bruker Alpha diamond module FTIR spectrometer with attenuated total reflectance (ATR) attachment for solid samples. Powder X-ray diffraction (PXRD) patterns were measured on a PANalytical X'Pert PRO diffractometer with a Cu-Kα light source (40 kV, 40 mA) with a step size of 0.02° and a 50 s time step. UV–Vis absorption spectra were measured in the solid-state in the range 200–800 nm using an Agilent Cary Spectrophotometer with a slit width of 2 nm. The fluorescence emission spectra were recorded on an Edinburgh FL980 Photoluminescence Spectrometer set at 2 nm slit width for both the excitation and emission. TGA was performed using an SDT-Q600 TA instrument. The samples were heated in air with a heating rate of 10 °C min^{-1} and the scan was recorded within the temperature range of 30–600 °C.

2.2 Methods

2.2.1 Preparation of $[Cu(ISO)_2(H_2O)_2]\cdot DMF$ (1)

2,6-Benzenedicarboxylic acid (ISO) (0.166 g, 1 mmol) and $Cu(NO_3)_2.3H_2O$ (0.242 g, 1 mmol) were dissolved in two separate 50 mL beakers with 10 mL dimethylformamide (DMF) and 10 mL distilled water respectively. The solution mixture was stirred for 30 min and thereafter heated in the oven at 100 °C for 4 h (Scheme 1). The clear blue solution formed was kept standing on the bench undisturbed until the formation of a blue precipitate after 2 days. The

precipitate was centrifuged, and the powder obtained was washed in DMF then dried in the oven at 60 °C for 30 min.

2.2.2 Sensing Experiment

The method described by Shi et al. was modified and adopted for the detection and sensing experiment [17]. In a typical fluorescence experimental set-up, 3 mg of the finely ground sample was dispersed in 3 mL aqueous solution of 0.5 mM nitrate or chloride salts of Fe^{3+}, Cr^{3+}, Ba^{2+}, Cu^{2+}, Na^{+}, Li^{+}, Co^{2+}, Mg^{2+}, Ni^{2+}, K^{+}, Zn^{2+} and Ca^{2+} respectively. Each of the suspension was ultrasonicated for 30 min before fluorescence data collection. To further investigate the sensitivity of the luminescence studies towards the recognized metal cation, monitoring of a series of suspensions were performed by gradually increasing the concentration of analytes ranging from 0.25–2.00 mM. For vapour sensing, the method described by Mehlana [18] was modified and adopted for the vapour sensing studies. 3 mg of the compound was first activated by heating under vacuum for 5 h at 120 °C. The activated compounds were placed in small vials and then these small vials were placed in previously evacuated larger vials containing various dry solvents (ethanol, methanol, chloroform, acetone, dimethylformamide, dichloromethane). The large vials were sealed tightly and left to stand at room temperature for 24 h. The sensitivity of the compound towards the different vapours detected were examined by exposing the compounds to different concentrations of the analytes ranging from 0.5–4.0 vol%.

3 Results and Discussion

The physicochemical properties of the synthesized $[Cu(ISO)_2(H_2O)_2]\cdot DMF$ (**1**) are presented in Table 1. The melting point of (**1**) is slightly higher than that of the precursor, isophthalic acid. CHN analysis of the compound gave the percentage of carbon, hydrogen, and nitrogen that are present. The compound was found to be insoluble in ethanol, dimethylformamide but soluble in dimethylsulfoxide.

3.1 FTIR

The IR spectra of compound **1** was recorded between 4000–400 cm^{-1}. In the spectrum of compound **1** (Figure S1), asymmetric and symmetric vibrations were seen in the region 1573 cm^{-1} and 1381 cm^{-1} respectively with the separations ($\Delta\nu$) 192 cm^{-1} confirming bidentate coordination mode of the carboxylate group [19, 20]. The broad band ranging from 2542–2813 cm^{-1} in the spectrum of ISO ligand was not found in the spectrum of compound **1**, this was as a result of deprotonation suggesting chelation of the carboxylate group to the Cu(II) ion [21]. The absorption bands at 3102 cm^{-1} and 3388 cm^{-1} are ascribed to ν(N–H) of DMF in the lattice and ν(OH) stretching of coordinated water molecules, respectively. Additionally, the sharp peaks in the region 822 cm^{-1} and 727–742 cm^{-1} are attributed to rocking and wagging bending vibrations indicating the presence of coordinated water [22]. The new sharp peak found at 484 cm^{-1} is designated to ν(Cu–O).

3.2 UV–Vis Spectroscopy

The electronic data of compound **1** and its ligand were recorded in the solid state at room temperature in the range 200 to 800 nm as shown in Figure S2 and Table 2. The parent ligand displayed absorption bands at 211 nm and 324 nm which are characteristic of the carboxylic acid chromophores. The bands are assigned to $\pi \rightarrow \pi^*/n \rightarrow \pi^*$ electronic transitions. The spectrum of compound **1** shows distinct absorption bands in the UV-region at 272 nm assigned to $\pi \rightarrow \pi^*/n \rightarrow \pi^*$ transition and 350 nm assigned to LMCT [23]. In the spectrum of the parent ligand of **1** absorption bands in the UV region (210 nm and 253 nm) are assigned to $\pi \rightarrow \pi^*$ of the carboxylic group and aromatic ring chromophore. The spectrum of compound **1** revealed a distinct band in the visible region (498 nm) attributed to ^2E_g–$^2T_{2g}$ electronic transition [24]. The shoulder observed in the UV region (349 nm) is assigned to LMCT.

Table 2 Electronic data of compound 1 and its ligand

Compound/ligand	λ_{max}, nm (E, cm^{-1})	Assignment
ISO	210 (47,619), 253 (39,526)	$\pi \rightarrow \pi^*$
1	349 (28,653), 498 (20,080)	LMCT, ^2E_g–$^2T_{2g}$

Table 1 Physicochemical properties of $[Cu(ISO)_2(H_2O)_2]\cdot DMF$ (1)

Compound	Appearance	Melting point (°C)	Molecular weight (g/mol)	Yield (%)	C% Calc. (found)	H%	N%
ISO	White	341	166		–	–	–
1	Blue powder	356	500.91	80.3	45.56 (44.74)	3.82 (3.89)	2.80 (2.27)

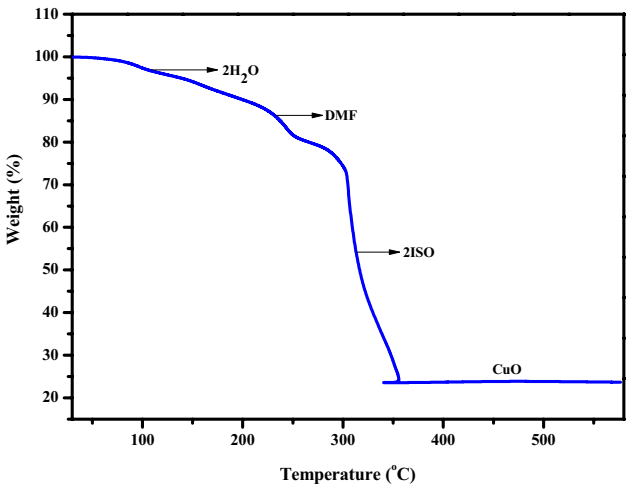


Fig. 1 TGA curve of compound **1**

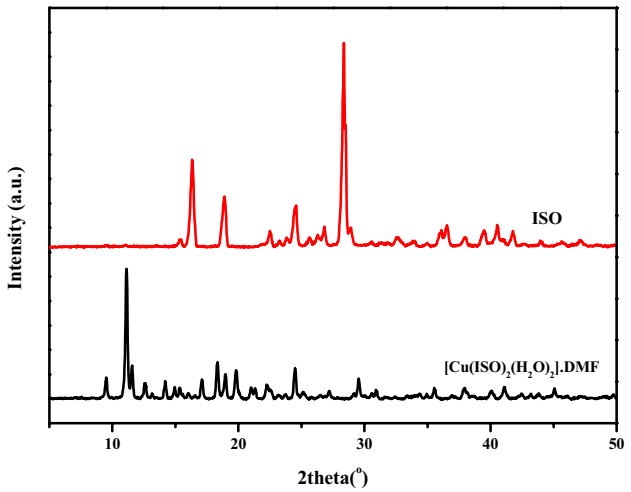


Fig. 2 PXRD patterns of **1** and its ligand

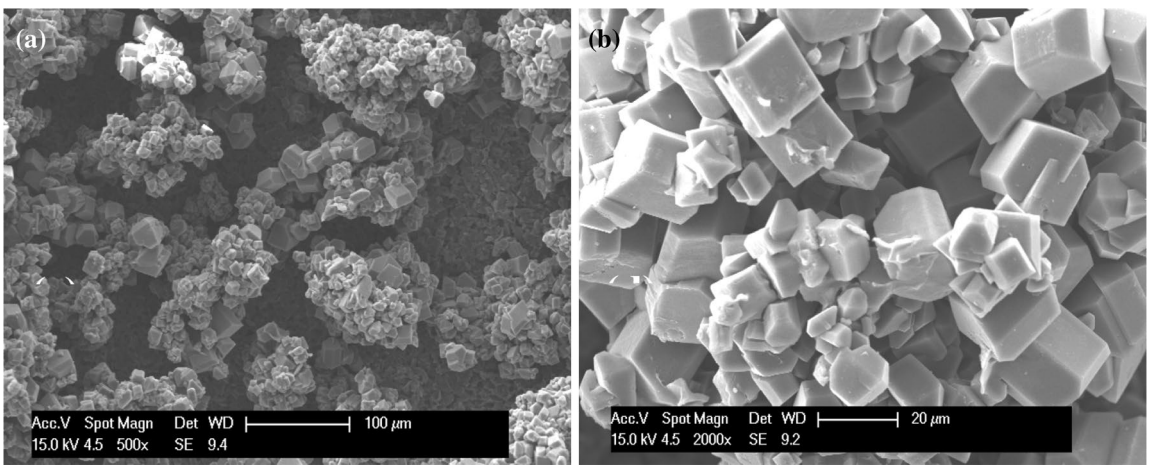


Fig. 3 SEM images of **a 1** scanned at 100 µm and **b 1** scanned at 20 µm

3.3 Thermal Analysis

In the curve of **1** (Fig. 1), the first weight loss (6.44% found (calc.7.19%)) is attributed to the evaporation of coordinated water in the temperature range of 89.66–111.04 °C. The second decomposition step is ascribed to evaporation of *N,N*-Dimethyl formamide molecule in the lattice at temperature ranging between 234.94–255.13 °C resulting to a total weight loss of 18.48% found (calc.15.72%). The drastic loss between 303.82–356.95 °C depicts the melting of the ISO ligand. A residue of 23.57% found (calc. 21.3%) CuO was left.

3.4 Powder X-Ray Diffraction Pattern

The PXRD pattern of the compound **1** and its ligand are shown in Fig. 2. The diffraction patterns revealed sharp crystalline peaks suggesting that the complex exist in a crystalline phase. The characteristic diffraction angles of **1** and ISO are ****2θ = [9.5°, 11.1°, 14.2°, 18.3°, 24.5°, 29.5°] and 2θ = [16.3°, 18.9°, 22.5°, 24.6°, 28.4°] respectively. The distinct PXRD pattern of **1** differs significantly from those of the ligand while new peaks were also observed at 9.5° and 11.1° diffraction angles confirming presence of Cu(II) ion in the PXRD of compound **1** [25].

3.5 Scanning Electron Microscopy (SEM)

The SEM image of compound **1** showed a cubical-shaped agglomerates of uniform particle sizes as shown in Fig. 3a, b.

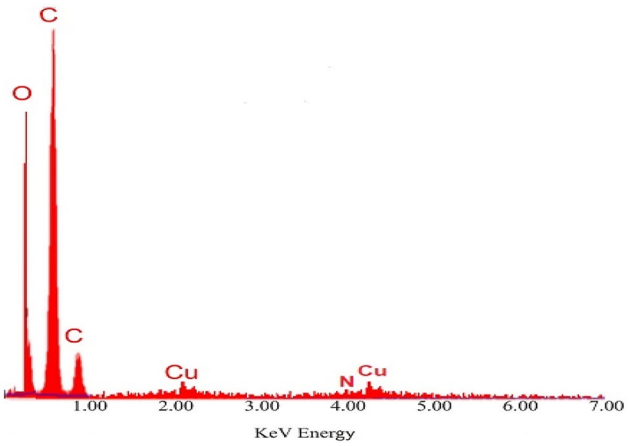


Fig. 4 Energy dispersive X-ray analysis of **1**

Table 3 EDX analysis of compound 1

Element	Weight %	Atomic %
C	47.76	49.51
O	36.49	34.26
Cu	13.43	18.24
N	2.22	2.54

3.6 EDX Analysis

The EDX spectrum of the compound as displayed in Fig. 4 and Table 3 confirmed the presence of copper (Cu), oxygen (O), carbon (C) and nitrogen (N). The percentage composition of the elements contained in the compound are found in the following order: C > O > Cu > N. Additionally, the reported EDX data (Table 3) is almost identical to that of the theoretically calculated and as well as by CHN elemental data.

3.7 Photoluminescent Property

Photoluminescent measurements were carried out on the compounds as-synthesized in the solid state and at room temperature. The photoluminescent (PL) spectra of **1** and its free ligand are shown in Figure S4. The ISO ligand shows an emission centered at 419 nm when excited at 365 nm which is ascribed to $\pi \rightarrow \pi^*$ transitions. The luminescence of **1** at 435 nm ($\lambda ex = 365$ nm) is attributed to intraligand emission arising from the π-electron rich backbone of the free ligand. Notably, the emission band of **1** is red-shifted (16 nm) compared to the luminescence of the ligand suggesting ligand to metal charge transfer (LMCT) while a reduction in luminescence intensity observed in **1** indicates coordination interaction between the ligand and the metal ion [26].

3.7.1 Sensing of Metal Cations

The sample was immersed in 3 mL of distilled water for 24 h prior to metal ion sensing, after which it was filtered and air-dried before PXRD data collection. The characteristic diffraction peaks observed in the As-synthesized sample matches well with that of sample immersed in distilled water and the sharpness of the peaks was also preserved (Figure S4). This suggests that the compound is stable even after immersion in distilled water; this observation is consistent with studies in literature [27, 28].

The effect of different metal ions in aqueous solution on the luminescence intensity of compound **1** is shown in Fig. 5a. The metal cations reduced the fluorescence intensities in the following order: $Fe^{3+} > Cr^{3+} > Mg^{2+} > Na^{+} > Zn^{2+} > Ba^{2+} > Co^{2+} > Li^{+} > Ca^{2+} > Ni^{2+}$. Interestingly, the introduction of Fe^{3+} into the suspensi on presented notable quenching effect with a quenching percent of 90.06% at a concentration of 0.5 mM (Figure S5). With the addition of Fe^{3+} ions, the emission intensity of **1** dispersed in distilled water steadily decreased (Fig. 5b). Additionally, the selective detection of Fe3 + in the presence of other interfering metal ions was investigated. The result showed the presence of interfering metal ions did not significantly alter the quenching effect of the Fe^{3+} ion (Figure S6). As the absorption band of Fe^{3+} overlaps with the emission band of **1**, the quenching mechanism might be attributed to energy transfer between the complex and the ion (Fig. 5c). [26]

3.7.2 Sensing of VOCs

The photoluminescence response of compound **1** towards various organic compounds is shown in Fig. 6a. The spectra clearly showed that the luminescent intensities of compound 1 is highly dependent on the recognition of the solvent molecules, for example in the PL spectrum of 1@MeOH, which displayed the most significant quenching effect. The luminescent selectivity is in the order DCM < $CHCl_3$ < DMF < Acetone < EtOH < MeOH and the calculated quenching efficiency for methanol was found to be 81% (Figure S7). To investigate the effect of methanol on the luminescent intensity of compound **1**, detailed tests were carried out by varying the concentration of methanol exposed to the complex, and the results revealed that as the concentration increases, the luminescent intensity decreases up to 61% (at 4.0 vol% of MeOH), as shown in Fig. 6b. The quenching effect may arise from hydrogen bonding interaction between the uncoordinated O atom of the ligand or coordinated water molecules and the methanol molecule which could reduce the luminescent intensity [29]. Furthermore, solvent loss/incorporation affects

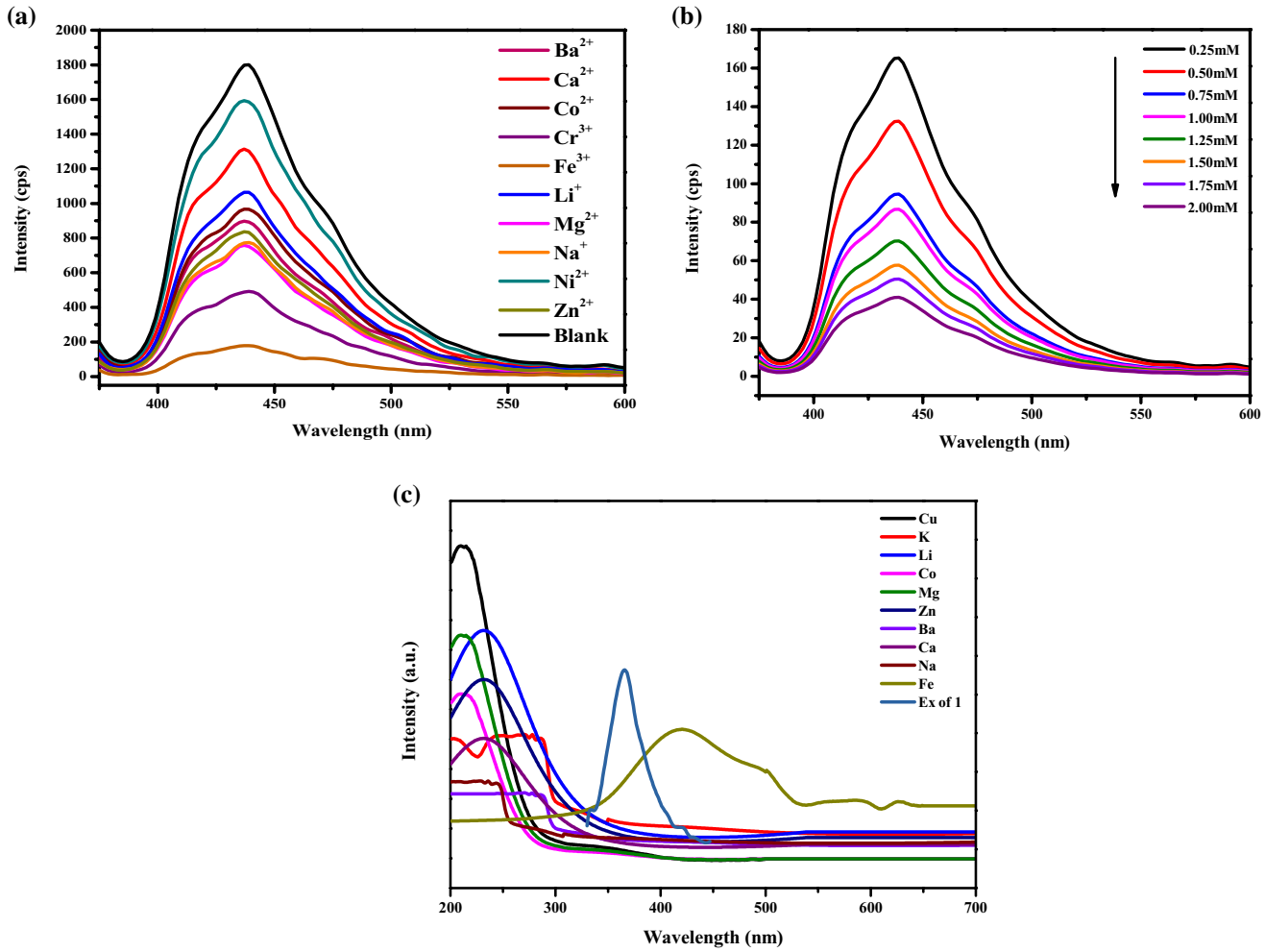


Fig. 5 **a** The emission intensities of compound **1** suspension after addition of $M(NO_3)_x$ or $M(Cl_2)_x$ (M^{x+} = Cr^{3+}, Ni^{2+}, Na^+, Mg^{2+}, Li^+, Fe^{3+}, Zn^{2+}, Co^{2+}, Ca^{2+}, Ba^{2+}). **b** PL of compound **1** immersed in different concentrations of Fe^{3+}. **c** Spectral overlap between the absorption bands of metal salts and excitation band of **1**

thermal stability. The TGA curve of 1@Activated was stable up to 170 °C compared to that of 1@As-synthesized which showed a 2.64% weight loss at 102 °C due to evaporation of water while the TG curve of 1@MeOH revealed a loss in weight (14.6%) at 62 °C attributable to loss of methanol and confirming inclusion of methanol (Fig. 6c). Fourier transform infrared (FTIR) spectroscopy was also used to monitor the uptake of methanol and the spectra are presented in Figure S8. In the FTIR spectrum of 1@Activated, disappearance of the broadband at 3466 cm^{-1} assigned to ν(OH) stretching vibration of water observed in the spectrum 1@As-synthesized was observed confirming removal of lattice water upon activation. Appearance of absorption bands in the region 2834 to 3347 cm^{-1} in the 1@MeOH spectrum, assigned to ν(OH) stretching vibration of alcohol indicates the presence of MeOH.

3.7.3 Stern–Volmer Plot and Detection Limit

The Stern–Volmer plot provides insight into the quenching mechanism and dynamics. Deviations from linearity or changes in the slope of the plot may indicate the presence of multiple quenching pathways. The Stern–Volmer equation is as follows:

$$Io/I = K_{SV}[Q] + 1,$$

where Io is the fluorescence intensity in the absence of the quencher, I is the fluorescence intensity of the quencher, K_{SV} is the stern–Volmer quenching constant and [Q] is the concentration of the quencher.

The S–V plot of relative intensity of compound **1** against concentration of Fe^{3+} shown in Fig. 7a demonstrated a good linear correlation (R^2 = 0.9919) and quenching constant, K_{SV}

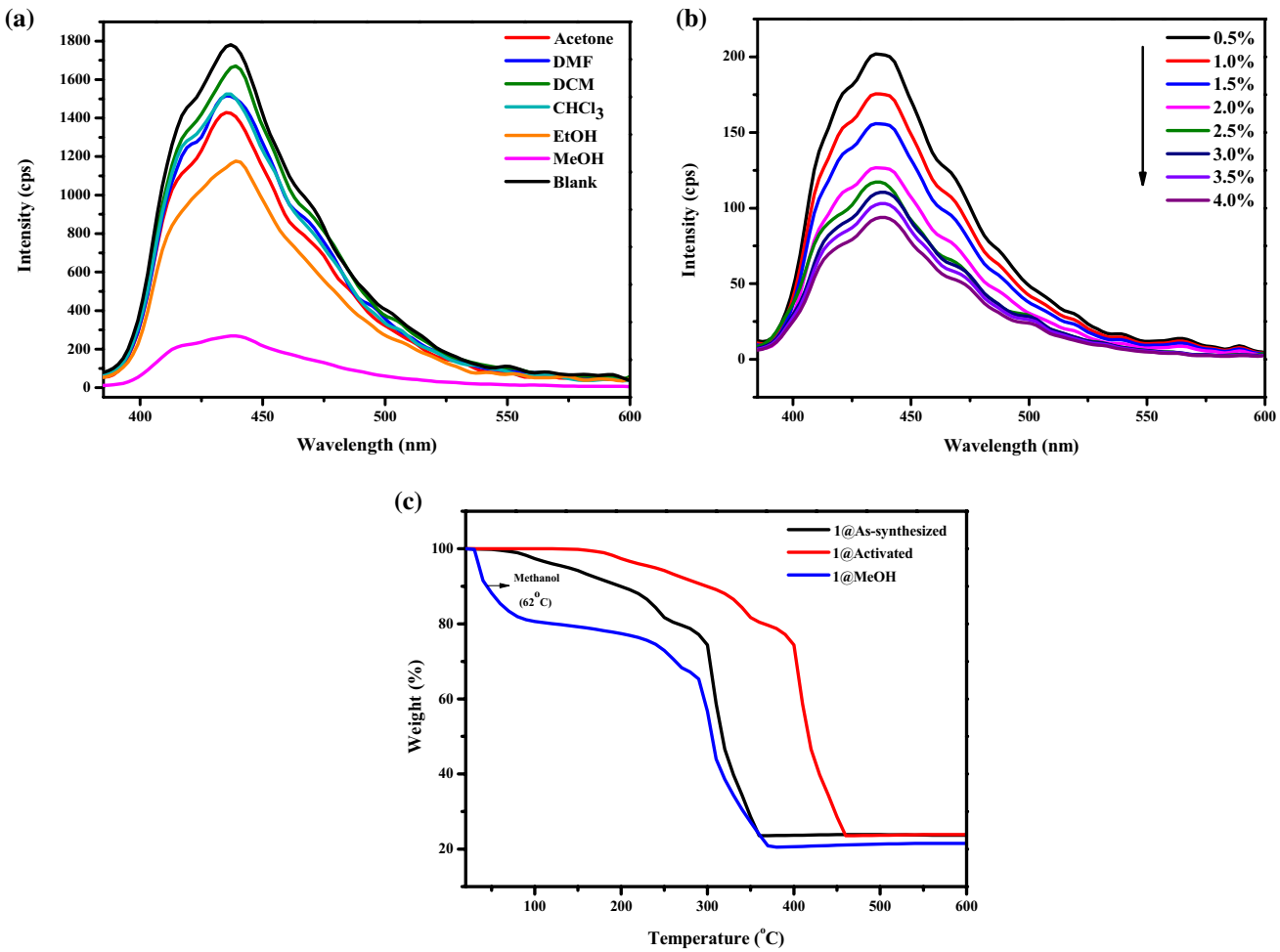


Fig. 6 **a** Fluorescence quenching spectra of compound **1** when exposed to different organic molecules. **b** Luminescence intensities of compound **1** upon incremental content of methanol. **c** Comparison of TGA curves of 1@As-synthesized, 1@Activated and 1@MeOH

of 2.038×10^4/M which revealed that **1** have high selectivity and sensitivity towards Fe^{3+} [17]. The limit of detection (LOD) was estimated to be 9.9×10^{-5} M which is quite lower than other reported sensors [30–32]. In the case of 1@MeOH, linear relationship ($R^2 = 0.9888$) was also observed and K_{SV} was 3.68×10^3/vol% (Fig. 7b). The limit of detection (LOD) was calculated to be 0.00084 vol%. The LOD 1@MeOH was found to be below the standard occupational exposure limit for methanol (0.025 vol%) [33]. Comparison of Stern–Volmer constants and limits of detections of this work to previous works is shown in Table S1 and S2.

4 Conclusion

In conclusion, this study presents an innovative approach for the design of a Cu(II) Isophthalate coordination polymer that has a dual functional ability to detect Fe(III) ions in aqueous solution and methanol vapour by drastic fluorescent turn off response. The compound was characterized by FTIR spectroscopy, XRD, SEM, EDX and Photoluminescence spectroscopy. The high selectivity towards the target analytes was attributed to the synergistic effect of energy transfer, competitive absorption of excitation energy and the host–guest interactions between the complex and the analytes. Compared to previous works high Stern–Volmer constant obtained indicates stronger analyte-probe interactions. This is crucial for achieving a significant fluorescence quenching response, even at low analyte concentrations. The high Stern–Volmer constants and low limits of detection achieved through this approach demonstrate its potential for sensitive and reliable analyte detection. This advancement contributes to the ongoing progress in fluorescence-based sensing and hold promise for future applications in various industries and scientific disciplines.

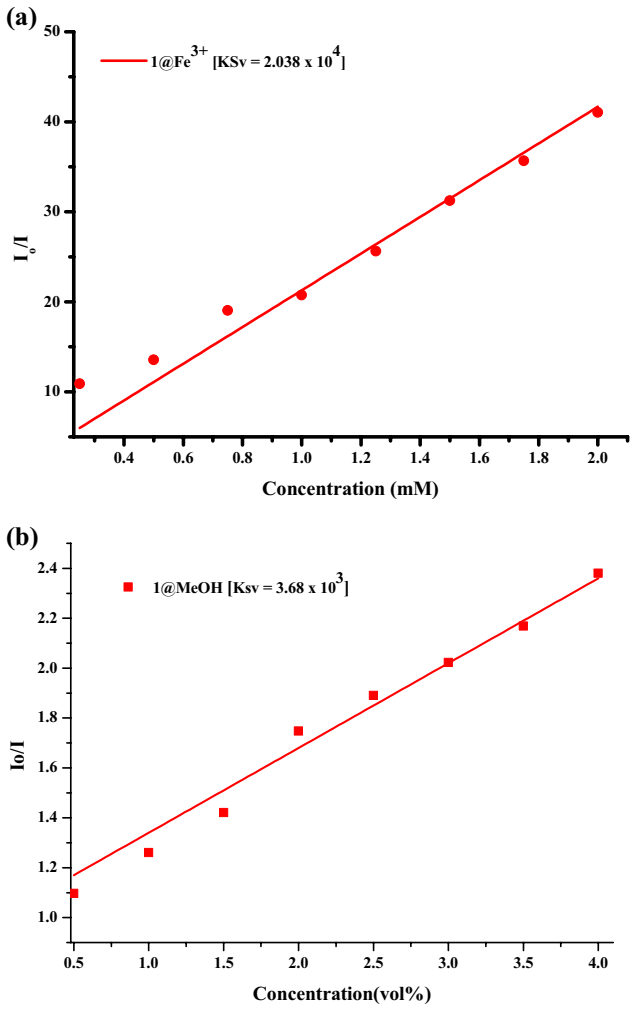


Fig. 7 **a** Stern–Volmer plots of **1**@Fe^{3+}. **b** Stern–Volmer plots of **1**@MeOH

Supplementary Information The online version contains supplementary material available at https://doi.org/10.1007/s42250-023-00746-9.

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Data Availability Statement Data will be made available upon request.

Declarations

Conflict of interest On behalf of the authors, the corresponding author states that there is no conflict of interest.

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