



A fluorescent copper(II) complex based on 4,4'-oxybisbenzoic acid and benzimidazole for selective detection of nitroaromatic compounds

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Abstract

Nitroaromatic compounds (NACs) are electron-deficient aromatic molecules extensively used in explosives, dyes, and pesticides, and their high toxicity and environmental persistence pose serious risks to human health and ecological systems. Consequently, the development of sensitive, selective, and portable sensing platforms for NAC detection is of significant importance. In this work, a copper-based coordination complex, $[Cu(Oba)_2(Beim)(H_2O)]$ (1) (H_2Oba = 4,4'-oxybisbenzoic acid; Beim = benzimidazole), was synthesized via a conventional method and fully characterized using standard instrumental techniques. Infrared spectroscopy confirmed the coordination environment, with characteristic bands observed at 1666 cm^{-1} ($\nu C=O$), 1243 cm^{-1} ($\nu C-N$), and 3061 cm^{-1} ($\nu N-H$). The complex exhibits strong fluorescence emission at 490 nm and good thermal stability. Fluorescence sensing studies revealed a significant quenching response in the presence of NACs, particularly 4-nitroaniline. The sensor demonstrated a low limit of detection (LOD) of 1.46 ppm and a high Stern-Volmer quenching constant ($K_{SV} = 9.16 \times 10^4\text{ M}^{-1}$) towards 4-nitroaniline, indicating excellent sensitivity and selectivity. Furthermore, recyclability experiments showed that the complex could be reused for up to four sensing cycles without significant loss of performance. These results highlight the potential of the copper coordination complex as an efficient and reusable fluorescent sensor for nitroaromatic compounds.

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1. Introduction

Nitroaromatic compounds (NACs), including nitrobenzene, 4-nitrophenol, 2,4,6-trinitrotoluene (TNT), and dinitrotoluene (DNT), represent major environmental and security risks due to

their toxicity, tenacity, and explosive nature [1]. Fluorescence-based detection techniques have grown in popularity for monitoring NACs due to their high sensitivity, fast response time, and on-site deployment capability [2]. Metal complexes, particularly transition metal complexes, have demonstrated great promise due to their tunable photophysical properties, structural versatility, and strong quenching responses to electron-deficient nitroaromatics [3]. As fluorescent sensors for nitroaromatic compounds (NACs), transition-metal complexes

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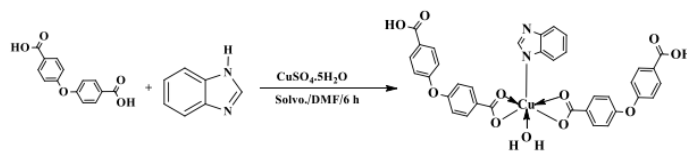
have been thoroughly investigated because of their strong interactions with electron-deficient analytes, structural diversity, and variable photophysical characteristics. Tsai *et al* synthesized Zn(II) coordination complexes that demonstrated strong emission quenching upon exposure to nitrobenzene, with PET identified as the main mechanism [4]. Another study used hydrothermal conditions to prepare a copper(II)-triazole complex containing a 5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazole-4-carboxylic acid ligand. A compound of the composition $[Cu(L)_2][Cu(L)_2(H_2O)_2]$ was produced (where L is the triazole ligand). Both the free ligand and its copper(II) complex demonstrated substantial fluorescence quenching in response to 2,4,6-trinitrophenol (TNP) [5]. Dutta and colleagues designed a Cd-based coordination polymer with high emission in both solid and suspension. Nitroaromatic compounds drastically reduced fluorescence, with detection limits in the micromolar range. Mechanistic studies indicated that the sensing response was caused by $\pi - \pi$ stacking and electron transfer interactions between the Cd-ligand framework and the nitroaromatic analytes [6]. In continuation of our previous studies on coordination complexes for luminescence, and chemical sensing [7], we report herein the application of a Cu(II) complex for detection of nitroaromatic compounds by fluorescence quenching mechanism.

2. Materials and methods

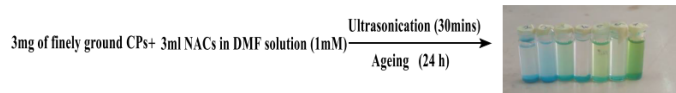
4,4'-Oxybisbenzoic acid, benzimidazole, and nitrobenzene, nitrophenol, nitroaniline from Sigma Aldrich, Germany. $CuSO_4 \cdot 5H_2O$ from Alfa Aesar, England. Methanol (99.8 %), Ethanol (97.0 %), N,N-dimethyl formamide (DMF), Dimethyl sulfoxide (DMSO) and Benzene from Central Drug House (P) Ltd New Delhi India.

2.1. Synthesis of $[Cu(OBA)_2(Beim)(H_2O)]$ (1)

The copper-based coordination complex $[Cu(OBA)_2(Beim)(H_2O)]$ (1) was synthesized via a solvothermal method using dimethylformamide (DMF) as the reaction medium. 4,4'-Oxybisbenzoic acid (H_2OBA , 0.254 g, 1 mmol) was dissolved in 10 mL of DMF under continuous stirring at room temperature to afford a clear solution. Separately, $CuSO_4 \cdot 5H_2O$ (0.250 g, 1 mmol) was dissolved in 10 mL of DMF, and benzimidazole (Beim, 0.059 g, 0.50 mmol) was dissolved in 5 mL of DMF with gentle heating to ensure complete dissolution. The three solutions were then combined in a single beaker under constant stirring, resulting in a homogeneous green solution. The reaction mixture was transferred to a sealed glass vessel and heated in a temperature-controlled oven at 90 °C for 6 h (Scheme 1), allowing gradual coordination between the Copper(II) ions and the organic ligands. After completion of the heating period, the reaction vessel was allowed to cool to room temperature, leading to the formation of a blue solid precipitate. The resulting product was collected by centrifugation at 4000 rpm for 10 min and washed several times with fresh DMF to remove unreacted ligands. The pure solid was then dried in an oven at 60 °C for 30 min to give the final copper coordination complex as a blue powder.



Scheme 1: Reaction pathway for the synthesis of $[Cu(OBA)_2(Beim)(H_2O)]$.



Scheme 2: Experimental set-up for sensing of NACs.

2.2. Instrumentation

A Bruker Alpha FTIR spectrometer coupled with a diamond ATR module was used to collect infrared data. X'Pert PRO diffractometer (PANalytical) with a Cu-K α radiation (40 kV, 40 mA) was used to generate X-ray diffraction patterns. Solid-state UV/Visible spectra were measured in the range of 200–800 nm. Fluorescence data were collected using an FL980 Spectrometer, with both excitation and emission slit widths set to 2 nm. A Thermal Analyzer SDT-Q600 equipment was used to perform thermogravimetric analysis (TGA). The measurements were made between 30 and 600 °C while the samples were heated in air at a rate of 10 °C per minute.

2.3. Sensing experiment

A modified version of the procedure reported by Lu *et al* [8] was employed for detection and sensing of nitroaromatic compounds. In a typical procedure, 3 mg of the sample in DMF and immersed in 3 mL of 1 mM solutions of various organic solvents (ethanol, methanol, benzene), including nitroaromatic compounds, nitrobenzene (NB), nitroaniline (NA), and nitrophenol (NP). The mixtures were sonicated for 30 minutes and then left to stand for 24 hours to form stable suspensions prior to fluorescence analysis (Scheme 2). To investigate the sensitivity of the compound towards nitroaromatic analytes, a batch of suspensions of the detected nitro-analytes in the sample were prepared by gradual increment of the nitroaromatics content.

3. Results and discussion

Table 1 summarizes the physicochemical properties of the compound as-synthesized. The compound has a molecular weight of 793.5 g/mol with a yield of 79.8 %. The compound displayed moderate thermal stability with a melting point of 348 °C and it was soluble only in dimethylsulfoxide (DMSO). The elemental analysis shows good concordance between the theoretical and experimental values obtained; this shows that the compound is pure.

Table 1: Physicochemical properties of $[Cu(OBA)_2(Beim)(H_2O)]$ (1).

Compound/ Ligand	Appearance	Solubility	Mp ($^{\circ}C$)	Mw (g/mol)	Yield (%)	C%			H%			N%		
						Calc.(Found)			Calc.(Found)			Calc.(Found)		
1	Blue powder	DMSO	348	793.5	79.8	58.87 (58.89)	3.67 (3.64)	3.92 (3.94)						
H_2Oba	White powder	DMF, DMSO	329	253.23	-	-	-	-						
Beim	White powder	DMF, DMSO	170	118.14	-	-	-	-						

Mp = Melting point and Mw = Molecular weight.

3.1. Spectroscopic studies

3.1.1. UV-visible spectroscopy

Figure 1 shows the electronic data of $[Cu(OBA)_2(Beim)(H_2O)]$, H_2OBA and Beim ligands respectively. The imidazole and aromatic ring chromophores bands of the free ligands are shown in the region 253 nm and 271 nm ascribed to $\pi \rightarrow \pi^*/n \rightarrow \pi^*$ transitions [9]. An absorption band was seen in the visible region of compound 1 at 490 nm, assigned to the $2E_g - 2T_{2g}$ of d-d transition [10].

3.1.2. Fourier transform infrared

The FTIR spectra of H_2Oba , Beim and compound 1 is displayed in Figure 1. In the spectrum of H_2Oba , a prominent absorption band at 1675 cm^{-1} correlates to the $\nu(C=O)$ stretching vibration. The spectra of compound 1 showed this band shift to 1666 cm^{-1} , indicating chelation with the Cu(II) ion [11]. Furthermore, the $\delta\nu(COO^-)$ value of 194 cm^{-1} obtained from the antisymmetric (1606 cm^{-1}) and symmetric (1412 cm^{-1}) stretching vibrations of the carboxylate group of compound 1 indicates that the H_2OBA ligand coordinates to the Cu(II) ion in a bidentate manner [12]. At 1243 cm^{-1} and 3061 cm^{-1} , respectively, the C–N and N–H stretching vibrations of the imidazole ring were visible in the spectra of Beim ligand. The spectrum of compound 1 revealed slight shifts in these bands to 1244 cm^{-1} and 3066 cm^{-1} , indicating that the nitrogen atom is involved in the coordination process [13]. The O–H stretching vibration of water accounts for the broad absorption band seen between 3386 and 3588 cm^{-1} . The in-plane bending vibrations of the C–H bonds in the benzene ring of the H_2OBA ligand correspond to peaks that appear in the 1160 – 852 cm^{-1} range. New peaks of M–O and M–N stretching vibrations are found at 475 cm^{-1} and 667 cm^{-1} respectively.

3.1.3. XRD Pattern

The XRD pattern of the complex along with its ligands are displayed in Figure 2. The appearance of distinct diffraction peaks suggests that the compound has an ordered structure [14]. The significant diffraction angles of H_2Oba , and Beim are as follows: $2\theta = [7.2^{\circ}, 11.3^{\circ}, 17.8^{\circ}, 20.6^{\circ}, 25.2^{\circ}, 33.2^{\circ}]$, $2\theta = [13.0^{\circ}, 17.9^{\circ}, 19.9^{\circ}, 24.0^{\circ}, 29.4^{\circ}, 32.7^{\circ}]$ and $2\theta = [13.3^{\circ}, 14.9^{\circ}, 18.5^{\circ}, 19.6^{\circ}, 22.6^{\circ}, 27.1^{\circ}, 29.0^{\circ}]$ respectively. The

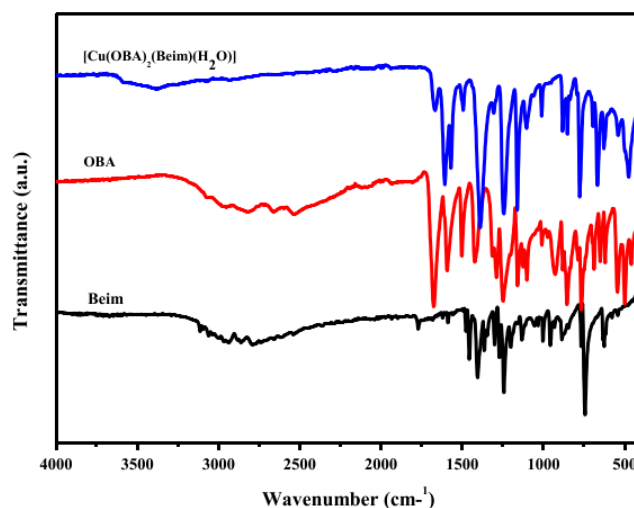


Figure 1: IR Spectra of compound 1 and its ligands.

complex exhibits characteristic diffraction peaks which are absent or significantly shifted compared to the parent ligands providing evidence for the formation of an entirely new compound [15]. These diffractions can be assigned to the (100), (110), (200), (210), (220), and (310) planes, respectively, consistent with a low-symmetry coordination framework commonly observed for copper(II) carboxylate-based complexes [16].

3.1.4. Thermal properties

The thermal decomposition of compound 1 occurred in two stages and it is presented in Figure 2. The first stage was the weight loss (18.47 % found (calc.17.10 %)) due to evaporation of a water molecule and decomposition of Beim ligand. The thermal curve was stable from $225^{\circ}C$ to $290^{\circ}C$, after which there was a drastic weight loss of 90.44 % found (calc. 90.34 %) assigned to the decomposition of H_2Oba ligand leaving a residue of CuO [17].

3.1.5. Scanning electron microscopy/electron dispersion xray spectroscopy

The scanning electron micrograph of $[Cu(OBA)_2(Beim)(H_2O)]$ showed a honeycomb shape,

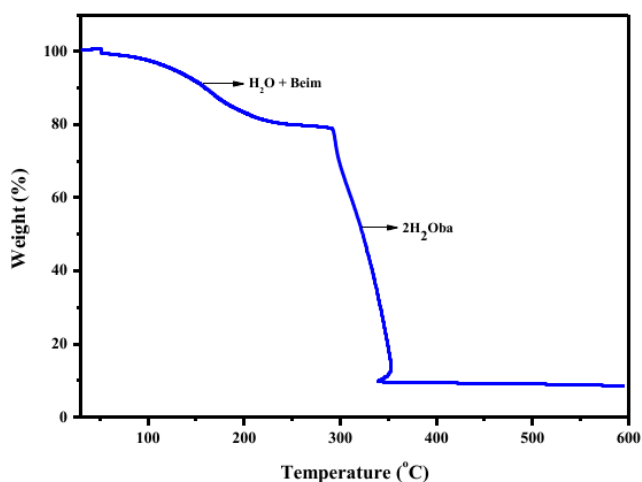


Figure 2: TG curve of compound 1.

with porous channels generating irregular hollow formations. The micrographs are displayed in Figure 3a at magnifications of 2000x and 1250x respectively. The EDX spectrum (Figure 3b) reveals the presence of Cu, O, N, and C in the material. The intense peaks observed at approximately 0.93 keV and 8.0 - 8.9 keV confirm copper as the dominant element, consistent with a copper-based coordination complex. A distinct signal at 0.52 keV corresponds to oxygen, while the peak at 0.39 keV is assigned to nitrogen, indicating the presence of O- and N-donor ligands coordinated to the copper center. A weak peak at 0.28 keV is attributed to carbon, confirming the organic nature of the complex. The comparatively low carbon weight percentage (2.0 wt %) relative to CHN elemental analysis is expected and arises from the limited sensitivity of EDX toward light elements and its surface-sensitive nature, particularly in metal-rich matrices. Overall, the EDX analysis supports the successful formation of a Cu-containing coordination polymer incorporating nitrogen- and oxygen-based organic ligands, with no evidence of elemental impurities.

3.1.6. Photoluminescence spectra

Upon excitation at 364 nm, the ligands exhibited distinct bands at 471 nm for Beim and 480 nm for H₂OBA. The observed emission is attributed to $n \rightarrow \pi^*/\pi \rightarrow \pi^*$ transitions resulting from intraligand fluorescence [18]. The emission band of compound 1 at 490 nm is red-shifted with respect to those of the ligands, which may be due to coordination-induced changes influencing ligand-to-metal charge transfer (LMCT) [19]. Figure 4 displays the luminescence spectra of [Cu(OBA)₂(Beim)(H₂O)], H₂OBA and Beim ligands.

3.1.7. Structure of the Cu(II) complex

The molecular structure consists of a mononuclear Cu(II) center coordinated by two carboxylate oxygen atoms derived from the 4,4'-oxybisbenzoate (OBA²⁻) ligands, one nitrogen atom from a benzimidazole ligand, and one oxygen atom from a coordinated water molecule, giving a distorted square-pyramidal coordination geometry around the copper ion (Figure

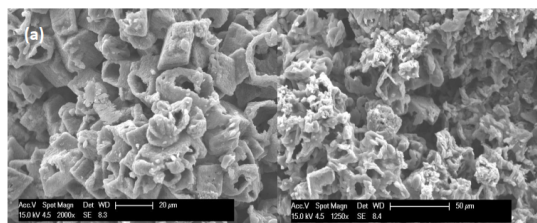
5). The equatorial plane is defined by the carboxylate oxygen donors and the benzimidazole nitrogen atom, while the axial position is occupied by the coordinated water molecule. Each (OBA²⁻) ligand coordinates to the Cu(II) center in a monodentate fashion through one carboxylate oxygen atom, with the remaining carboxylic acid group remaining protonated and non-coordinating. The benzimidazole ligand acts as a neutral monodentate N-donor, contributing to stabilization of the metal center and influencing the overall electronic environment.

3.1.8. Fluorescence quenching analysis

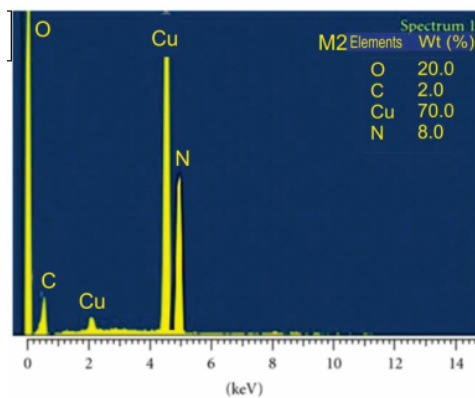
The influence of nitroaromatic compounds on the intensity of emission of compound 1 was examined, and the results showed that fluorescence intensity decreased in the order of 4NA > 4NP > NB (Figure 6). Significant quenching of fluorescence intensity was observed in the nitroaromatics up to 90 %, 69 % and 55 % at concentration of 1 mM for 4NA, 4NP and NB respectively. Moreover, non-nitroaromatics have insignificant effect on the luminescent intensity indicating that the quenching effect is largely dependent on the nitro substituent groups (Figure 4). To further examine the sensitivity of 1 towards 4NA, it was suspended in varying concentrations of the nitro analyte (0.5 – 3.0 mM). Incremental concentrations led to gradual reduction in luminescent intensity (Figure 7). The quenching mechanism involves photoinduced electron transfer (PET) from π - electron rich ligands to 4NA adsorbed on the surface of 1 [20]. The reversal in sensing selectivity as compared to the previously reported work [7] arises from differences in coordination environment and available interaction sites. In this Cu(II) complex, reduced hydrogen-bonding accessibility and enhanced donor-acceptor interactions favor 4-nitroaniline over 4-nitrophenol, while nitrobenzene remains the weakest due to the absence of specific interaction functionalities. At lower concentrations, the Stern-Volmer plot of 1 showed an essentially linear relationship with $R^2 > 0.9$ (Figure 5, 6 and 7). The limit of detection (LOD) was estimated using $LOD = 3\delta/k$, and the quenching constant (KSV) was determined from the stern volmer equation; $I_0/I = KSV[Q] + 1$. The quenching constants (KSV) were found to be $1.7 \times 10^4 M^{-1}$, $9.16 \times 10^4 M^{-1}$ and $7.93 \times 10^3 M^{-1}$ for 4NP, 4NA and NB respectively while the LODs were 1.46 ppm, 1.51 ppm and 1.58 ppm for 4NA, 4NP and NB respectively both of which are in line with literature findings [21]. The low detection limit and high quenching constant for 4-NA indicate that the complex is highly sensitive and capable of detecting even trace amounts of 4-NA [22]. The fluorescent photograph of the compounds under UV-light before and after immersion in nitroaromatic compounds is displayed in Figure 8. It can be observed that the luminescence of the compounds decreases upon immersion in nitroaromatic compounds.

3.2. Recyclability test

The recyclability of compound 1 for nitroaromatics sensing was investigated in four cycles by washing several times in DMF and thereafter contacted with the NACs. As depicted



(a)



(b)

Figure 3: (a) SEM images of compound 1 showing honeycomb morphology with scans at different magnifications. (b) EDX spectrum of $[\text{Cu}(\text{OBA})_2(\text{Beim})(\text{H}_2\text{O})]$.

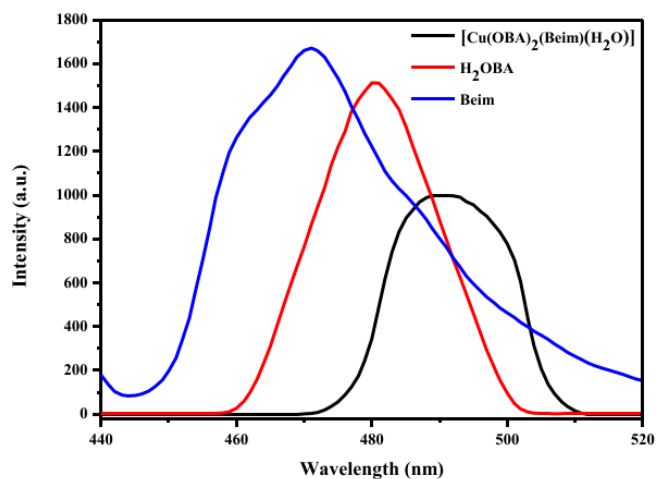


Figure 4: Photoluminescence Spectra of $[\text{Cu}(\text{OBA})_2(\text{Beim})(\text{H}_2\text{O})]$ and its ligands.

in Figure 9, the quenching efficiency of 1 for all the four cycles were almost uniform at 90 %, 87 %, 85 %, 84 % respectively. To further examine the stability and structural integrity of compound 1 after immersion in various NACs in several cycles, PXRD pattern were collected after each cycle. There were no significant changes in the PXRD patterns confirming the stability of the compounds (Figure 8).

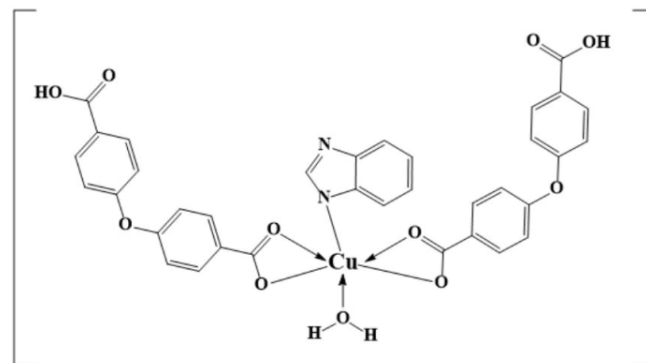


Figure 5: Proposed structure of $[\text{Cu}(\text{OBA})_2(\text{Beim})(\text{H}_2\text{O})]$.

4. Conclusion

A copper-based coordination complex was successfully synthesized and characterized, and its luminescent sensing performance toward nitroaromatic compounds in aqueous solution was investigated. The complex exhibited a pronounced fluorescence quenching response upon exposure to nitroaromatic analytes, with exceptional sensitivity toward 4-nitroaniline (4-NA). Significant attenuation of the emission intensity was observed even at low analyte concentrations, indicating strong interactions between the electron-deficient nitroaromatic molecules and the electron-rich copper coordination framework. The fluorescence quenching mechanism is predominantly attributed to a photoinduced electron transfer (PET) mechanism, facilitated

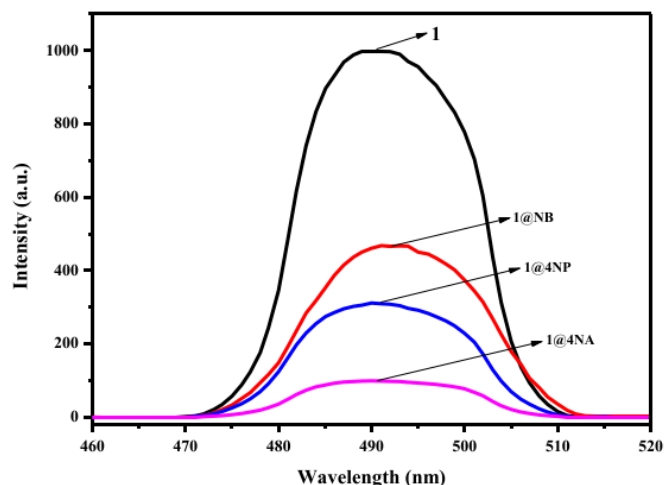


Figure 6: Luminescence intensities of 1, 1@4NA, 1@NB and 1@4NP.

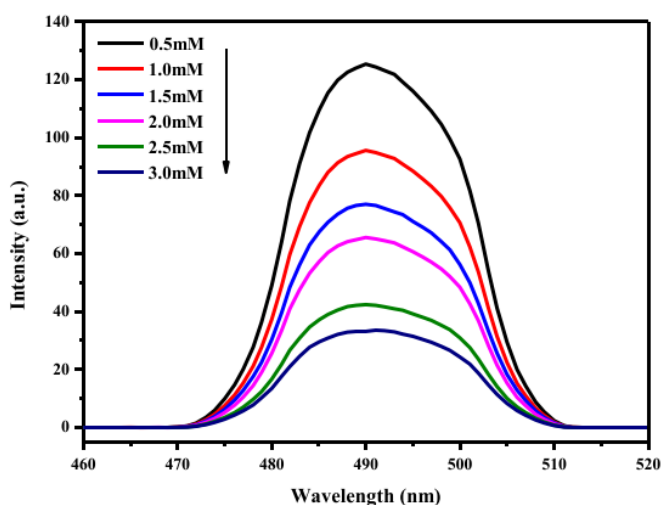


Figure 7: Emission spectra of $[\text{Cu}(\text{OBA})_2(\text{Beim})(\text{H}_2\text{O})]$ with incremental addition of 4NA.

by favorable energetic alignment between the excited-state fluorophore and the electron-accepting nitro groups. Under ambient aqueous conditions, the sensor displayed rapid response kinetics, good repeatability, and excellent selectivity for nitroaromatic compounds over other potentially interfering organic species. Importantly, the coordination complex retained its structural integrity and sensing efficiency after multiple sensing cycles, demonstrating high chemical stability and reusability. These results highlight the potential of copper-based coordination materials as robust and efficient luminescent sensors for the selective and real-time detection of nitroaromatic explosives and related hazardous pollutants.

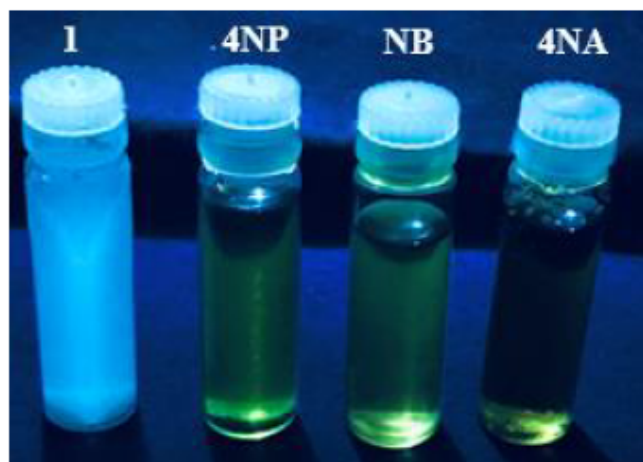


Figure 8: Photograph of $[\text{Cu}(\text{OBA})_2(\text{Beim})(\text{H}_2\text{O})]$ after immersion in various NACs when exposed to UV light.

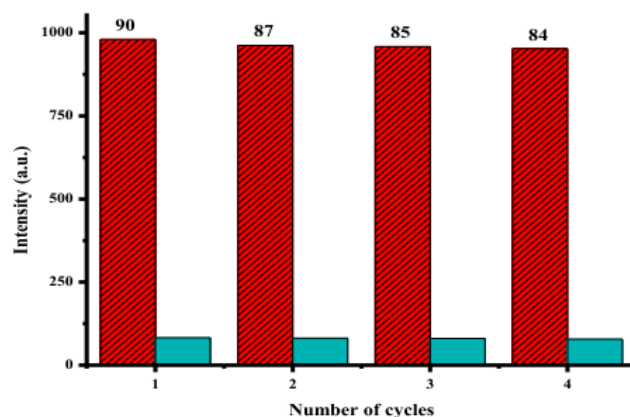


Figure 9: Emission intensities of 1@4NA up to four recycles.

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Data availability

The dataset used in this study is available upon request to the corresponding author.

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