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Design, Synthesis, And Fluorescence Sensing Performance of A Quinoline-Based Schiff Base Toward Zn^{2+} Ions

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ABSTRACT

FT-IR, 1H NMR, 13C NMR and elemental analysis were used to synthesize and characterize a novel quinoline-based Schiff base, (E)-2-((quinolin-8-ylmethylene)amino)quinolin-8-ol (H_5L). The photophysical behavior of H_5L was studied in different homogeneous solvents and showed that the fluorescence behavior was strongly dependent on the solvents. The free ligand was weakly fluorescent but when it was chelated with Zn^{2+}, it showed a significant enhancement in emission intensity. This phenomenon is called chelation-enhanced fluorescence (CHEF). A 1:1 binding ratio of H_5L:Zn^{2+} was confirmed from fluorescence titration studies. pH also affected the sensing performance: the complex H_5L–Zn^{2+} exhibited the highest fluorescence response between the pH range of 6.5–10.5. The results obtained in this case show that H_5L is an appropriate chemosensor to be used as a selective and sensitive fluorescent sensor for detection of Zn^{2+} ions in solution.

Keywords: *Quinoline, Schiff Base, Fluorescent, Chemosensor, Ethanol.*

1. INTRODUCTION

A chemosensor is an abiotic molecule that may attach to an analyte selectively and reversibly while simultaneously changing one or more system characteristics, such absorbance or fluorescence spectra, redox potentials, or both. One key aspect of chemosensors is their ability to transduce signals upon analyte binding, which allows for the potential of real-time and spatial monitoring of concentration. The widespread use of chemosensors has piqued the interest of scientists across many fields, particularly in the fields of chemistry, biology, physics, and materials science. When compared to other types of chemosensors, those that rely on fluorescence have numerous benefits, such as being very sensitive, inexpensive, easy to execute, and adaptable; they can also provide spatial resolution of subnanometers, with visualisation of submicrons, and temporal resolution of submilliseconds. Modulating the photophysical properties of a fluorophore is possible through various means, including proton-, energy-, and electron-transfer processes, heavy-atom effects,

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changes in electronic density, and the destabilisation of a non-emissive nπ* excited state. Many other opportunities are also available. Luminescent chemosensors may be fine-tuned in a variety of ways thanks to this. Concurrently, developments in photochemistry laid the groundwork for the creation of devices that could detect analyte complexation by changes in absorbance and/or fluorescence bands.

Developing chemosensors for transition metal ions is a current area of focus among various analytes. While these metals are generally considered a threat to the environment when present in excessive amounts, essential elements like iron, zinc, copper, and cobalt are present in living systems. Among these metal ions, Zn2+ is involved in a wide variety of cellular processes, including neurotransmission, metalloenzyme catalysis, gene expression control, and apoptosis. An imbalance in Zn2+ metabolism is linked to a number of serious neurological disorders, such as epilepsy, Alzheimer's disease, and cerebral ischaemia. Consequently, the study of neurobiology relies heavily on Zn2+ measurements. Since Zn2+ and Cd2+ have comparable chemical characteristics and frequently react in tandem with similar spectrum changes, there is an urgent need to create Zn2+ selective sensors that can differentiate Zn2+ from other transition metal ions, particularly Cd2+.

It is well-known that Schiff bases, which are imines, may effectively bind to metal ions. Several Schiff base metal complexes have desirable electrical and photophysical characteristics, as well as antioxidative and anticancer actions. Another promising method for optical detection of metal ions are Schiff base derivatives that have a fluorescent moiety. Complexes formed by tetradentate ligands, including pyridine- or salen-type symmetrical Schiff bases, and certain metal ions can display remarkable coordination, excellent thermal stability, fluorescence, and biological activity. The self-assembly of these compounds is influenced by various factors, such as the ligand's chemical structure, the metal's preferred coordination geometry, pH, the ratio of metal to ligand, reaction temperature, and solvent system, making controllable synthesis a formidable challenge. The development and fabrication of highly selective and sensitive fluorescence sensors for metal ions is a dynamic and essential area of research at the moment. Commercial metal ions sensors are widely available, but chemists are always working to enhance their sensitivity, selectivity, and reliability. This is all in an effort to meet the many demands that arise from the widespread presence and significant role of metal ions in organisms.

II. REVIEW OF LITERATURE

Kumar, Ram & Pandey, Shwetank. (2023). Because of its paramount role in living things, zinc has garnered a lot of attention from the scientific community in recent decades. All living things rely on zinc, a trace metal that is critical for many bodily functions, including immune system function, wound healing, cell division, and growth. Hence, it is very necessary to create and improve chemosensors for Zn2+ determination that are easy to use, highly efficient, selective, and affordable. Because of its one-of-a-kind characteristics, chemosensors can detect a wide range of metal ions with remarkable precision. Several research groups created and synthesised Schiff base chemosensors in 2018. This review summarises their work. This review has also provided a brief overview of how these probes interact with zinc metal ions. In addition, the future lead molecule for determining Zn2+ ions may be Schiff base probe 34, which has two benzothiazole moieties and the lowest detection limit (0.00028 µM), as shown by comparing the detection limits of various probes.

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Liu, Hongmei et al., (2022) By using colorimetric or fluorescence analyses, this study presents a straightforward and flexible Schiff base chemosensor (L) that can detect four neighbouring row 4 metal ions (Co2+, Ni2+, Cu2+, and Zn2+). Even in solutions with a broad variety of additional cations and anions, L was able to detect the target ions. Using a Job's plot, HR-MS tests, and 1H NMR titration studies, the recognition processes were confirmed. Following that, L was used to create Co2+ colorimetric test strips and TLC plates. At the same time, L could accurately quantify the concentrations of target ions in both municipal and river water samples. Remarkably, L's potential biological uses were proven when it was utilised to image Zn2+ in HepG2 cells, zebrafish, and tumor-bearing animals. This suggests that L has potential as a multipurpose instrument for the detection of the target metal ions in biological and environmental contexts.

Yan, Jun et al., (2016). Zn2+ selective recognition was achieved using the development and synthesis of a novel resumable Schiff-base fluorescent probe, 7-methoxychromone-3-carbaldehyde-(3'-hydroxy-2'-naphthaleneformyl) hydrazone (L). We found that the binding ratio of L to Zn2+ was 1 using data from the titration and the ESI-MS spectra. In the presence of Zn2+ in an ethanol environment, the sensor's fluorescence increased significantly (430 nm excitation and 498 nm emission). The improvement might be a result of photoinduced electron transfer (PET) and C=N isomerisation.

Singh, Takhellambam et al., (2013). N, N'-bis(salicylidene)-1,2 phenylenediamine (LH2), a Schiff-base fluorescent molecule, was developed and tested as a chemoselective Zn(2+) sensor. A significant increase in the fluorescence intensity and a red shift were seen when Zn(2+) was added to an ethanol solution of LH2. In addition, additional typical ions of alkali metals, earth metals, and transition metals did not elicit any reaction or even minor shifts in the spectrum. The ability of this chemosensor to differentiate between Zn(2+) and Cd(2+) is noteworthy. Quantum yield is dramatically enhanced by coordination, according to fluorescence experiments on both the free Schiff base ligand LH2 and the LH2-Zn(2+) complex. The Benesi-Hildebrand relation, which yields a stoichiometric ratio of 1:1, was used to assess the association constant and stoichiometric ratio. According to Job's plot analysis, this provided more evidence of 1:1 complex creation.

III. MATERIALS AND METHODS

Materials and reagents

Sigma-Aldrich India Pvt. Ltd. supplied the 2-aminoquinolin-8-ol and quinoline-8-carbaldehyde. Merck Life Science Pvt. Ltd. and Sisco Research Laboratories (SRL) in Mumbai, India, were sourced for the spectroscopic-grade solvents, which included ethanol, methanol, chloroform, dimethyl sulfoxide (DMSO), and acetonitrile. The study relied on analytical reagent (AR) grade chemicals sourced from three different companies in India: Loba Chemie Pvt. Ltd. of Mumbai, Himedia Laboratories Pvt. Ltd. of Mumbai, and SRL Pvt. Ltd. of India. All aqueous solutions were prepared using double-distilled deionised water. Using conventional analytical-grade chemicals, buffer solutions with varying pH values were produced. Unless otherwise noted, all tests were conducted at room temperature in the lab (298 ± 2 K).

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Synthesis and characterization of (E)-2-((quinolin-8-ylmethylene) amino) quinolin-8-ol (H5L)

Prepared a solution of 2-aminoquinolin-8-ol in absolute ethanol (20 mL) using 0.150 g and 0.94 mmol. There was a separate dissolution of quinoline-8-carbaldehyde in ethanol (20 mL) containing 0.125 g and 0.80 mmol. A yellowish reaction mixture was produced by gradually adding the aldehyde solution to the amine solution while stirring continuously. Afterwards, the reaction mixture was refluxed for 3 hours after adding 2-3 drops of glacial acetic acid as a catalyst. Vacuum filtration, three washes with cold ethanol to remove unreacted contaminants, and reduced pressure drying were all performed on the yellow precipitate that resulted from the reaction. Pure yellow crystals of the Schiff base ligand (E)-2-((quinolin-8-ylmethylene)amino)quinolin-8-ol (H_5L) were obtained by recrystallisation from an ethanol/chloroform combination (1:3, v/v) to purify the crude product. A yield of around 85% was achieved with the purified product.

FT-IR (vmax, cm-1, KBr): 3377(υOH), 3192(υas(C- H), 3047(υs (C-H),), 1596(υC=N), 1572(υC=C), 1240(υ (C-O)), 1128(υ(C-N)), 787(υ(C-H)); 1H NMR (400 MHz, CDCl3, TMS, δ, ppm): 6.93 (d, H-2), 7.28 (t, H-3), 7.32 (d, H-4), 8.25 (d, H-6),7.93 (d, H-7), 7.50 (s, H-9), 7.6 (d, H-11) 7.4 (t, H-12) 7.7 (d, H-13) 8.0 (d, H-15) 7. 3 (t, H-16), 8.8 (d, H-17); 13C NMR (400 MHz, CDCl3, TMS, δ, ppm): 112.0, 120.3, 120.8, 121.8, 122.7, 126.1, 126.2, 127.6, 129.3, 131.3, 135.4, 135.7, 138.9. 141.6, 149.7, 150.0, 152.7, 153.0, 163.7; Anal. Calc. for C19H13N3O (299.3): C, 76.25 %; H, 4.36 %; N, 14.04 %. Compounds identified: C, 76.40%; H, 4.32%; N, 14.10%.

Physical Measurements

Using the KBr pellet approach, FT-IR spectra were obtained using a PerkinElmer Spectrum Two spectrometer in the 4000-400 cm^{-1} range. Bruker Avance III 400 MHz NMR spectra were acquired on a $CDCl_3$ solvent with tetramethylsilane (TMS) serving as the internal standard for the ´H and ´³C NMR spectra. Parts per million (δ, ppm) was used to represent chemical changes. An elemental analyser from PerkinElmer, the 2400 Series II CHNS/O, was used to measure carbon, hydrogen, and nitrogen.

A Shimadzu UV-1800 UV-Visible spectrophotometer was used to record the electronic absorption spectra. A PerkinElmer LS-55 fluorescence spectrometer was used to measure the emission and excitation spectra of the fluorescent compounds. Quartz cuvettes of superior quality with an optical path length of 10 mm were used for all spectroscopic experiments. The study's fluorescence measurements were conducted with excitation and emission slit widths kept at 5 nm. Every experiment followed the same protocol and was carried out at room temperature.

The comparable approach was used to estimate the fluorescence quantum yield (Φf) of the synthesized compounds, with quinine sulphate in 1 M H_2SO_4 serving as the reference standard (Φf = 0.546). The following equation was used to compute the quantum yield by comparing the sample's integrated fluorescence intensity with the standard's:

$$\Phi^{i}_{f} = \Phi^{s}_{f.} \cdot \frac{F^{i}}{F^{s}} \cdot \frac{1-10^{-A^{s}}}{1-10^{-A^{i}}} \cdot \left(\frac{n^{i}}{n^{s}}\right)^{2}$$

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In this context, F represents the overall fluorescence intensity throughout the complete fluorescence spectral curve, A^i and A^s denote the sample's and standard's optical densities, respectively, and η^i denotes the solvent's refractive index at 300 K.

IV. RESULTS AND DISCUSSION

Steady state spectral properties

A variety of homogenous solvents were used to record the absorption spectra of H5L. Two distinct peaks were seen at 280 nm and 335 nm in the absorption spectra. The fact that the molar extinction coefficient is 1.5 X 105 M-1 cm-1 suggests that the ππ* transition of the benzene ring is responsible for the absorption. Because the azomethine C=N group has a double bond, the peak at 280 nm may be seen. Table I shows that following excitation at 335 nm, the fluorescence emission spectra of H5L exhibit a single peak around 400-525 nm. Because of variations in solvent polarity, fluorescence spectra exhibit distinct characteristics in pure homogeneous fluids (Figure -1). Thus, this fluorescent probe has further applications as a polarity sensor probe. We may explain the low value of fluorescence quantum yield (Φf) by considering the non-radiative decay transition to the lying nπ* state of nitrogen.

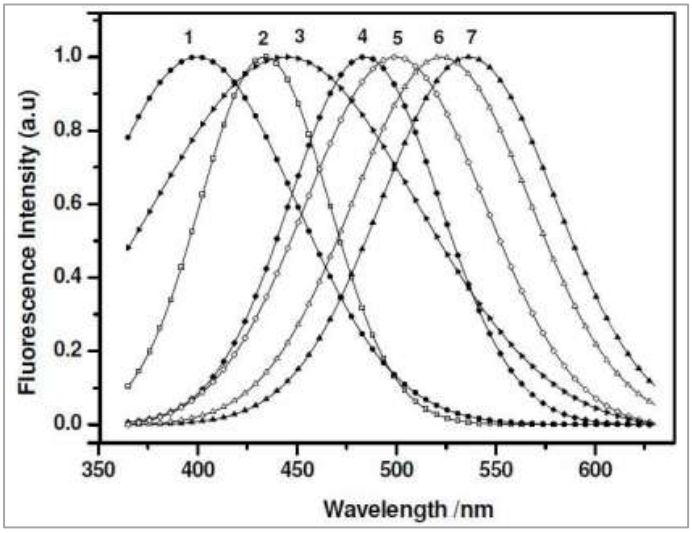

Figure 1: Solvent-Dependent Fluorescence Emission Spectra of H₅L in Different Homogeneous Solvents

Table 1: Steady-State Spectroscopic Characteristics of H₅L In Different Homogeneous Solvent Media

Solvents	λabs a(nm)	λ b(nm) fl	Δνssc(cm-1)	Φ d(10-3) F
Ethanol	266	500	10303	4.1
	330			
Methanol	252	520	10530	2.6
	336			
Acetonitrile	253	495	10140	3.9
	330			

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1,4-Dioxane	255 335	401	4974	3.0
N,Ndimethylformamide	270 328	504	10645	2.8
Toluene	268 333	439	7249	3.2
Dimethylsulfoxide	260 337	484	9010	2.9
Chloroform	253 332	472	8932	5.6
Ethyl acetate	265 332	425	6590	4.5
Tetrahydrofuran	257 338	430	6220	5.1

Influence of Zn2+ concentration

The influence of Zn2+ concentration on fluorescence properties of H5L was carried out in ethanol solution. The 335 nm and 500 nm peaks for excitation and fluorescence emission, respectively, were measured. The isomerisation of the C=N double bond resulted in a very faint fluorescence signal for H5L. Zn2+ binds strongly to the nitrogen atom of the H5L. Thus, a red shift of the emission peak from 500 to 520 nm and an increase in fluorescence intensity are observed as the concentration of Zn2+ is gradually increased from around 60 µM to H5L. Isomerisation cannot occur because of binding of metal ions. According to Figure-2. The addition of Zn2+ causes a significant complexation with H5L, which is responsible for the rise in fluorescence. Despite the existence of additional metal ions, not even at extremely high concentrations (~ 100µM) was this impact noticed. In both the presence and absence of Zn2+, the quantum yield of H5L was determined. With the addition of the Zn2+ complex, the quantum yield of H5L rose from 3.8X10-3 to 5.6X10-1. The picture below shows the equation for 1:1 complex creation in the inset, which is a double reciprocal plot.

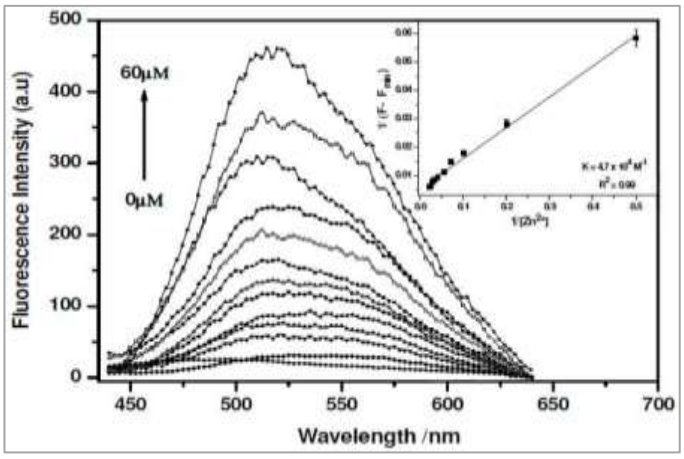

Figure 2: Variation in Fluorescence Emission Intensity of H_5L upon Incremental Addition of Zn^{2+} Ions

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Effect of pH

It was tested for pH sensitivity both with and without Zn2+. Over a wide range of pH, the fluorescence intensity of the unbound ligand remained constant (Figure-3). In contrast, H5L demonstrated pH dependence when Zn2+ was present. In an acidic environment, H5 L exhibited a modest fluorescence response to Zn2+, which led to a weak coordination ability of Zn2+. The molecule's Zn2+ sensing capabilities, however, improved when the pH was raised to 6.5-10.5. In this graph, black dots denote the presence of Zn2+ while white dots denote its absence.

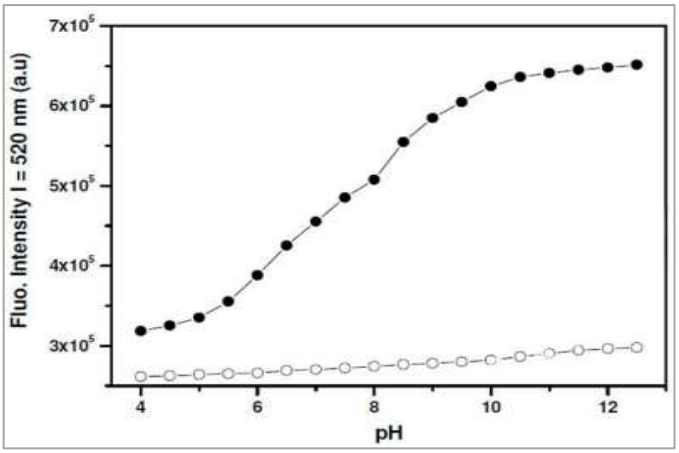

Figure 3: pH-Dependent Fluorescence Response of H_5L with and without Zn^{2+} Ions

V. CONCLUSION

Spectroscopic and elemental analytical methods were used to effectively synthesise and characterise a new quinoline-based Schiff base ligand, (E)-2-((quinolin-8-ylmethylene)amino)quinolin-8-ol (H_5L). Its sensitivity to environmental changes was demonstrated by the photophysical tests, which showed that H_5L's absorption and fluorescence characteristics are significantly solvent-dependent. The ligand's solvatochromic behaviour was clearly seen in its fluctuating quantum yield and fluorescence emission over a range of homogenous solvents. H_5L is shown to be a selective and effective chemosensor for Zn^{3+} ions in fluorescence sensing tests. The chelation-enhanced fluorescence (CHEF) effect was responsible for the noticeable increase in fluorescence intensity and red shift in emission wavelength that occurred when the compound was coordinated with Zn^{2+}. A stable 1:1 H_5L-Zn^{2+} complex was verified by binding studies. In addition, it was discovered that the sensing response was pH-dependent, and the H_5L-Zn^{3+} system showed its best fluorescence performance within the pH range of 6.5-10.5.

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