

Synthesis, Structure, and Fluorescence Features of a Dinuclear Zinc Complex Based on a Quinoline-Containing Schiff Base Ligand

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Abstract. Zinc coordination compounds contribute a lot to chemistry, acting in catalytic processes, reactions in biology, and the creation of photoluminescent materials. The type of ligands is necessary for making well-defined and used zinc coordination compounds. The research describes the synthesis and structural analysis and fluorescence behavior of zinc compound $[Zn_2L(OAc)_3]$ (**1**), which was synthesized from Schiff base ligand HL (HL=2-[(2-hydroxy-3-methoxybenzyl)imino]methyl}quinoline). The Schiff base ligand HL resulted from the condensation of quinoline-2-formaldehyde with o-vanillin. The photophysical properties of quinoline formaldehyde and the coordination flexibility of 3-methoxy-2-hydroxybenzylamine in compound **1** produce an asymmetric dinuclear structure that exhibits unique photoluminescent behavior. The characterization of compound **1** included FT-IR spectroscopy and elemental analysis, as well as single-crystal X-ray diffraction and powder X-ray diffraction. The fluorescence emission of compound **1** occurs in different solvents until nitrobenzene causes a significant reduction in its emission. Therefore, we can see that compound **1** can detect nitroaromatic hazardous substances through fluorescence.

Keywords: Zinc coordination complex; Schiff-based ligand; fluorescent sensors.

1. Introduction

Zinc metal is noted for its low poisonous effects, affordability, plenty of reserves, and friendly effect on the environment. Since zinc coordination compounds are adaptable and of interest to researchers, they are utilized everywhere. Right now, many catalytic reactions use zinc compounds instead of precious metals [1, 5], and these substances contribute to essential processes in living beings [6, 9]. Because they emit more light, zinc compounds are perfect choices for making luminescent materials [10, 25]. Preparing zinc compounds is best done by selecting facile-to-make ligands whose structures can be easily adjusted, which allows you to create zinc complexes with interesting shapes and specific functions. The condensation reaction between 3-methoxy-2-hydroxybenzylamine and quinoline-2-carboxaldehyde was used to make the Schiff base ligand HL (HL = 2-[(2-hydroxy-3-methoxybenzyl)imino]methyl}quinoline). After this, ligand HL was reacted with $Zn(OAc)_2$ under solvothermal conditions to create a compound $[Zn_2L(OAc)_3]$ (**1**). The ability of compound **1** to emit light with the help of light was also examined. The following section covers the making, structure, and light effects of compound **1**.

There has been a noticeable increase in interest in zinc-based luminescent materials because they perform well in various fields, for example, OLEDs, biological imaging, and detecting environmental changes. Post-transition metal zinc with a completely filled d^{10} electronic configuration draws extra attention to such materials. With this unusual electron configuration, the lights from transition metal complexes are much better, because fewer non-radiative pathways occur and more may enjoy greater fluorescence than many other similar complexes. Zinc ions, in addition, have strong coordination properties and they easily form bonds with nitrogen, oxygen, and sulfur. Thanks to their ability to be tetrahedral, trigonal bipyramidal, or octahedral, they enable the exact control of electrons and space around the metal part of the compound. Because of these properties and smart ligand creation, chemists can control the way absorption and light emission take place in the complexes. Among all ligands, those with a Schiff base core – especially when made from aromatic aldehydes and amino alcohols – are especially worth attention. They are straightforward to make and also have strong

binding properties and are very stable at high temperatures because of their several points of coordination. Coordination with metals usually leads to the formation of strong chelate rings in the complex, which increases the complex's stability and reduces vibrational relaxation. The addition of π -conjugated systems, for example, quinoline groups, to these zinc Schiff base complexes helps more electrons move across the different atoms, which improves absorption and using light and changes the way the compounds act physically. HL, an organic molecule made with a quinoline structure, methoxy, and hydroxyl groups, was selected for the current method. Since the quinoline unit is aromatic, it raises the transition chance in $\pi - \pi^*$. On the other hand, the two groups increase the ability of the compound to dissolve in various solvents and attach to metal ions. Such functional groups make it possible for secondary bonds to develop inside the molecule and protect the overall structure of the ligand complex. Sealed and high-temperature conditions ensure that crystals are formed, so they are of high quality and can be used in single-crystal X-ray diffraction analysis. With this approach, it is possible to find out the structure of the molecule, the way the atoms are linked, and how the molecule behaves optically, which are very important for the study. All in all, the study shows that using thoughtful design and the right techniques for synthesis leads to the creation of zinc coordination complexes with luminescent features. Because of this synergy, it becomes possible to produce future materials for advanced optoelectronics, sensors, or imaging agents in biology. This way of thinking also adds to our understanding of how metal-organic systems react when they are excited.

2. Experimental

2.1 Experimental Reagents and Instruments

All the reagents were of analytical quality and were used without any additional purification.

The analysis was done using an instrument called a Nicolet Magna IR 500 FT-IR spectrometer to collect infrared spectroscopic data, a Carlo Erba EA1110 CHNO-S microanalyzer for elemental analysis, a Bruker SMART APEX II CCD single-crystal diffractometer for crystal structure determination and an FLS 920 spectrophotometer for measuring fluorescence.

2.2 Synthesis of Schiff Base Ligand HL [26]

Dissolve the proper amounts of each chemical in 20 mL of methanol, stir the mixture at room temperature, and leave it overnight. The yellow solid that settles is filtered, separated, and cleaned with just a bit of anhydrous methanol to get the ligand with an 85% yield. The C, H, and N analyses of $C_{18}H_{16}N_2O_2$ indicate a formula of C, 73.95%; H, 5.52%; N, 9.58%. Copied: C accounts for 73.66 percent, H accounts for 5.59 percent, and N represents 9.78 percent of the total.

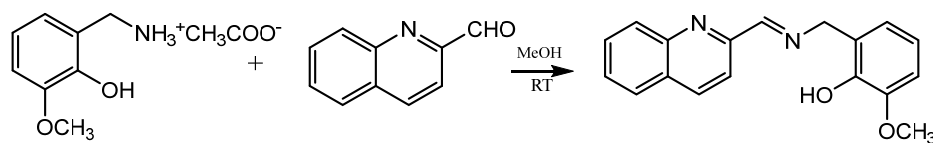


Figure.1 Scheme 1 Synthesis of the HL Ligand

2.3 Synthesis Routine of $[Zn_2L(OAc)_3]$ (1)

Accurately weigh 0.05 mmol of ligand (0.0175 g), and 0.1 mmol of $Zn(OAc)_2$ (0.0199 g), added to a long Pyrex glass tube, and 2 mL of anhydrous ethanol was then added to the glass tube. Seal the glass tube and place it in an 80 °C oven for 2 days. After cooling to room temperature, yellow block crystals suitable for X-ray diffraction analysis were obtained.

2.4 Structure Features by X-ray Diffraction

To obtain the crystal's X-ray diffraction data, Bruker SMART APEX II CCDX was used in the ω - 2θ scanning mode. SHELXT and SHELXTL, which are part of Olex2 [26, 28], were used to generate the crystal structures. The positions of hydrogen atoms were initially suggested theoretically and then refined by fitting the riding model. Results from crystallographic analysis of complex one can be seen in Table 1, and the most important bond lengths and angles are shown in Table 2.

Table 1 Crystal data and structure refinement for compound one.

| Parameter | Value |
|--|---|
| Empirical formula | C ₂₄ H ₂₄ N ₂ O ₈ Zn ₂ |
| Formula weight | 599.23 |
| Temperature/K | 298 |
| Crystal system | orthorhombic |
| Space group | <i>P</i> 2 ₁ 2 ₁ 2 ₁ |
| <i>a</i> /Å | 7.9749(3) |
| <i>b</i> /Å | 14.1228(6) |
| <i>c</i> /Å | 21.7631(8) |
| α /° | 90 |
| β /° | 90 |
| γ /° | 90 |
| Volume/Å ³ | 2451.13(17) |
| <i>Z</i> | 4 |
| ρ_{calc} g/cm ³ | 1.624 |
| μ /mm ⁻¹ | 2.009 |
| <i>F</i> (000) | 1224.0 |
| Crystal size/mm ³ | 0.9 × 0.2 × 0.1 |
| Radiation | MoK α (λ = 0.71073) |
| 2θ range for data collection/° | 5.866 to 49.99 |
| Index ranges | -9 ≤ <i>h</i> ≤ 8, -16 ≤ <i>k</i> ≤ 13, -25 ≤ <i>l</i> ≤ 25 |
| Reflections collected | 10377 |
| Independent reflections | 4327 [<i>R</i> _{int} = 0.0382, <i>R</i> _{sigma} = 0.0485] |
| Data/restraints/parameters | 4327/0/329 |
| Goodness-of-fit on <i>F</i> ² | 1.016 |
| Final <i>R</i> indexes [<i>I</i> ≥ 2 σ (<i>I</i>)] | <i>R</i> ₁ = 0.0313, <i>wR</i> ₂ = 0.0622 |
| Final <i>R</i> indexes [all data] | <i>R</i> ₁ = 0.0399, <i>wR</i> ₂ = 0.0660 |
| Largest diff. peak/hole / e Å ⁻³ | 0.35/-0.32 |
| Flack parameter | -0.012(9) |

Table 2 Selected bond length (Å) and angles (°) for compound one.

| | | | |
|--------|----------|-----------|------------|
| Zn1-O1 | 1.976(3) | O1-Zn1-O2 | 113.11(14) |
| Zn1-O2 | 1.979(3) | O1-Zn1-O3 | 97.80(13) |
| Zn1-O3 | 2.093(3) | O1-Zn1-N1 | 90.60(15) |
| Zn1-N1 | 2.189(4) | O1-Zn1-N2 | 116.89(16) |
| Zn1-N2 | 2.055(4) | N2-Zn1-O3 | 84.26(14) |
| Zn2-O3 | 2.010(3) | N2-Zn1-N1 | 78.30(16) |
| Zn2-O4 | 2.320(3) | O3-Zn2-O4 | 73.88(12) |
| Zn2-O5 | 2.332(4) | O4-Zn2-O5 | 82.83(15) |
| Zn2-O6 | 2.043(3) | O8-Zn2-O4 | 88.08(13) |
| Zn2-O7 | 2.020(3) | O8-Zn2-O5 | 59.46(14) |
| Zn2-O8 | 2.025(3) | O8-Zn2-O6 | 95.65(14) |

3. Results and Discussion

3.1 IR Spectrum and PXRD Pattern of Compound 1

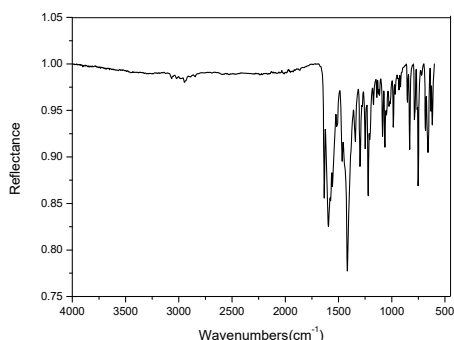


Figure. 2 IR spectrum of compound 1.

To describe Compound **1**, IR (Figure 2), X-ray powder diffraction analysis (Figure 3), and elemental analysis were carried out. No $\nu(\text{OH})$ band between $3000 - 3500 \text{ cm}^{-1}$ in **1** proves that the HL ligand has been deprotonated. Many bands can be found in the wavelength area between 1650 cm^{-1} and 1200 cm^{-1} . The region includes the vibrations of the CO_2 molecule, but it is hard to tell them apart because they overlap with the $\text{C} = \text{N}$ bands of the Schiff base ligand. At both 1594 and 1416 , strong bands are connected to the covalent stretching and bending vibrations of CO_2 . The difference Δ [$\Delta = \nu_{\text{as}}(\text{CO}_2)$ and $\nu_{\text{s}}(\text{CO}_2)$] is bigger than for the related NaO_2CMe salts (the amount for NaO_2CMe is 164 cm^{-1}), illustrating that acetate ions connect to the metals via a bidentate bridge. Figure 3 shows that the PXRD pattern for compound **1** looks similar to the one that was predicted by single-crystal X-ray data. The peaks in both the diffraction patterns line up nicely, proving that the sample consists of crystals with the same phase as the single one. Changes in peak intensities are often due to the way the sample's crystals are oriented and how many particles are in the powder used. Because the experimental and simulated results are close, it proves that the compound is pure and has the same structure. The experimental powder X-ray diffraction (PXRD) pattern of **1** is well matched with that of the simulated PXRD pattern, indicating that the phase of compound **1** is pure.

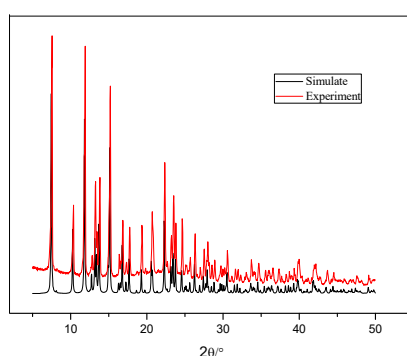


Figure. 3 Powder X-ray diffraction pattern of compound one

3.2 Description of Crystal Structure of 1

The results of single-crystal X-ray diffraction reveal that complex one is in the shape of an orthorhombic crystal with the space group $\text{P2}_1\text{2}_1\text{2}_1$. According to Figure 4a, the crystal has one single deprotonated ligand, two zinc ions, and three acetate ions. In Figure 4b, you can see that Zn1 and Zn2 sit in coordination environments made of polyhedrons. Figure 4(c) separates and draws attention to the metal coordination spheres with the help of a stick-and-sphere model. This image removes the overall structure to concentrate only on the atoms attached to Zn1 and Zn2 . Zn1 makes a five-coordinate structure by forming bonds with two nitrogen and three oxygen atoms, whereas Zn2 is surrounded by six oxygen atoms in its structure. It is obvious that O3 connects the Zn1 and Zn2 atoms in this molecule. It is visible that acetate coordinates with the metal ion in both monodentate and

bidentate ways by examining O4 to O8 atoms. This figure makes it easier to understand the bonding directions, type of coordination, and the way these factors play a role in building the overall structure. While Zn1 has five ligands around it, Zn2 takes on a six-ligand setup. In particular, Zn1 links with the hydroxyl oxygen O3, two nitrogen atoms N1 and N2, and two of the oxygen atoms O1 and O2 that are present in acetate groups. These five coordinating atoms make a square pyramid structure when linked to Zn1. Meanwhile, Zn2 coordinates with the hydroxyl oxygen atom O3 on the L⁻ ligand, O4 on the methoxy group, as well as O6 and O7 from two acetate groups, and O5 and O8 from another acetate group. The Zn2 atom has six surrounding atoms that make up a regular octahedron in shape. Also, the crystal structure reveals that the HL ligand gives up its proton to transform into an anion, and it functions by forming a $\mu^2 : \eta^1 : \eta^2 : \eta^1 : \eta^1$ -type bridging mode. The three acetate groups exhibit two different bidentate coordination modes, with two acetate groups showing bridging fashion and the other one displaying chelating mode.

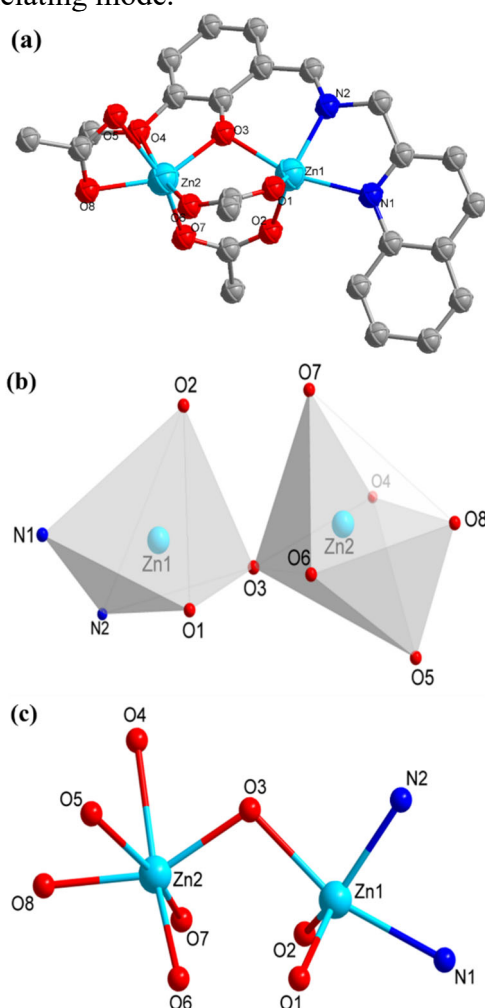


Figure. 4 (a) Molecular structure of **1**. Hydrogen atoms and solvents have been omitted for clarity. (b) Coordination polyhedrons of Zn1 and Zn2 ions in **1**. (c) The dinuclear [ZnOZn] core in **1**.

3.3 Photoluminescent Properties of **1**

Upon irradiating at 476 nm wavelength, the fluorescence emission spectrum of HL was measured and is displayed in Figure 5. The emission spectrum shows a strong peak around 520 nm location, which belongs to the green region of visible light rays. The emission pattern is standard for aromatic Schiff bases since $\pi - \pi^*$ transitions have more effect on how the base releases energy. Because of the quinoline ring, the molecule has an elongated π -system that eases the absorption and emission of light. The behavior of $[Zn_2L(OAc)_3]$ (compound **1**), on the other hand, is different from its other form. Upon exciting the complex with a 395 nm laser, it gives off a strong fluorescence and its peak emission is at 490 nm, as apparent from Figure 6. From these results, we can tell that the complex

gives a blue shift of approximately 30 nm as compared to the free ligand HL. A shift to shorter wavelengths implies that putting a ligand around a zinc center changes the location of the electrons. Metal – ligand bonding leads to the blue shift since the bonding of Zn(II) to the nitrogen and oxygen from HL shifts the density of the metal. In particular, when there are heteroatoms like nitrogen or oxygen in the ligand with lone pairs, these interact with the d-orbitals of the metal, bind to it, and minimize electron sharing across the π system. This leads to a larger difference between HOMO and LUMO and thus makes the emitted light energetic (it has a shorter wavelength). This way of interacting is usually associated with ligand-to-metal charge transfer (LMCT) or intrigant transitions that have been changed by coordination. Besides, the flexibility of the ligand lessens after coordination, and the assembly's effects can keep more energy in the excited state and alter the light emitted. Looking at the changes in fluorescence proves that associating the ligand with the protein affects its excited-state behavior, showing that even little structural changes can affect how a ligand fluoresces.

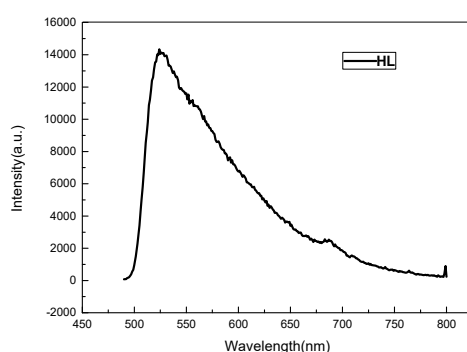


Figure. 5 Emission spectrum of the HL ligand at solid state

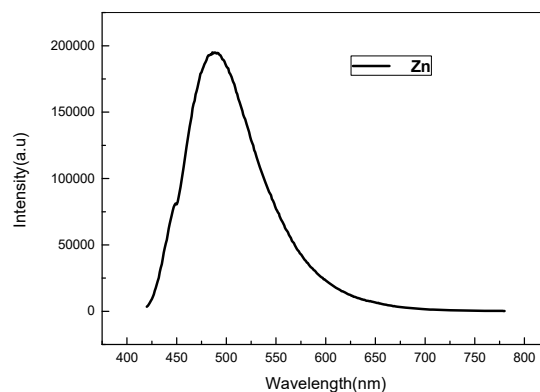


Figure. 6 Emission spectrum of compound 1 at solid state

Then, the emission fluorescence spectra of complex one were observed in many solvents such as H₂O, MeOH, EtOH, MeCN, DMF, DME, THF, 1,2-CH₂ClCH₂Cl, CH₂Cl₂, CHCl₃, toluene, acetone and nitrobenzene (Figure 7). It is evident from the experiments that complex one shows fluorescence in H₂O, MeOH, EtOH, MeCN, DMF, DME, THF, 1,2-CH₂ClCH₂Cl, CH₂Cl₂, CHCl₃, toluene and acetone. Bearing in mind that fluorescence quenching happens in nitrobenzene, complex **1** may be fit to detect harmful nitrobenzene in a fluorescent manner. Since compound **1** changes its luminescence depending on the solvent, it is clear that its photophysical traits react to the chemical environment. Because of beneficial hydrogen bonding, the increased emission from ethanol and methanol can be attributed to the way the excited state is stabilized and non-radiative ways of decay are reduced. Besides, the not-too-drastic solvatochromic shifts demonstrate that ligand field and electronic properties are affected by the changes in polarity associated with different solvents. Solvent molecules that do not engage in hydrogen bonding, such as CH₂Cl₂ and THF, reveal instead weak fluorescence. Of all of the solvents tested, nitrobenzene gave the most noticeable reduction in the

glow of the phosphor, reducing it nearly to zero. It is most likely that this significant decrease in fluorescence comes from strong $\pi - \pi$ interactions with the nitro group and the transfer of excited-state electrons to the nitro group. So, the compound works as an effective sensor that detects the presence of toxic, explosive, and persistent nitroaromatic substances. Finding out about these substances by measuring fluorescence makes it valuable for monitoring chemicals and for the environment. In addition, quenching happens preferentially in nitrobenzene, indicating the special reaction mechanism, and not just because of the general polarity of the other solvents. On this basis, investigations into compound **1** for portable sensing devices and organic materials detection in water can be conducted.

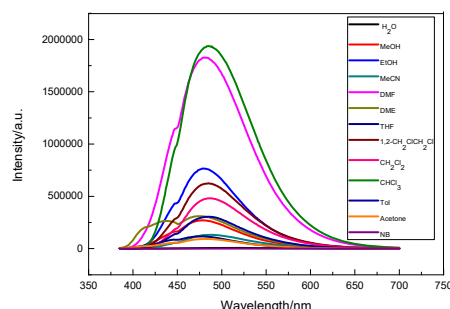


Figure. 7 Emission spectra of compound **1** in different solvent

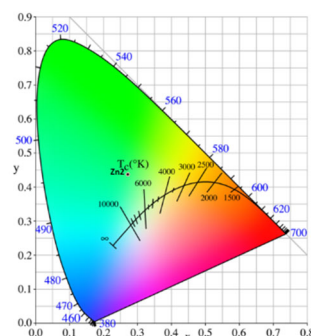


Figure. 8 The Commission Internationale de l'éclairage (CIE) color space chromaticity diagram of the complex **1** in the solid state ($x = 0.27$, $y = 0.43$).

Figure 8 is an illustration of the CIE 1931 chromaticity diagram that reveals the emission color of complex **1** when it is in the solid state. The luminescence is located in the green region of the visual spectrum since ($x = 0.27$, $y = 0.43$) are its emission coordinates. This proves the green fluorescence in UV light and suggests that complex **1** can be used in photonic or sensing applications. It shows how related the colors we see are to the wavelengths of light emitted by the source. The emission does not match the Planckian locus (blackbody radiation curve), therefore revealing that the emission comes from electronic processes instead of heat. This difference proves that the emission is the result of processes happening between the zinc and the Schiff base. Besides, the chromaticity coordinate of the emission falls within the range seen in photoluminescence findings, which indicate major emission in the area around 480 to 500 nanometers. This diagram makes it possible to see and discuss colour as well as numerically analyze and compare the properties of different compounds. In addition, the uniform green glow and the different intensities between compound **1** and nitrobenzene-induced quenching mean that compound **1** is a suitable pick as a specific probe. Collecting such information could play a role in improving future studies designed to change the colours emitted by dyes by changing their components.

4. Conclusions

To conclude, adding acetic acid to a mixture of Schiff base ligand (HL), formed by reacting 2-quinoline carboxaldehyde and 3-methoxy-2-hydroxybenzylamine in a solvothermal manner yielded the dinuclear zinc structure $[Zn_2L(OAc)_3]$ as anticipated. To make certain that the structure was pure and had the proper structure, the compound was studied using single-crystal X-ray diffraction, powder X-ray diffraction (PXRD), and Fourier-transform infrared (FT-IR) spectroscopy, and measured for its elements. According to the crystal structure, the compound is formed by two Zn atoms, where Zn₁ has a five-coordinated square pyramidal geometry while Zn₂ has a six-coordinated octahedral geometry. Complex formation by the Schiff base involves $\mu^2:\eta^1:\eta^2:\eta^1:\eta^1$ bridging patterns, shown by ligand and acetate coordination, which underline the easy and peculiar way Schiff base forms stable multinuclear structures. Apart from structure determination, researchers evaluated the light-related characteristics of the complex in detail. The emission from solid-state photoluminescence experiments was found to happen at shorter wavelengths than that of the free ligand. Such a blue shift is thought to be caused by how the electrostatic fields around the Zn(II) ions influence the ligand's behaviour. Especially, results showed that this compound gave strong fluorescence in many solvents such as H₂O, MeOH, EtOH, MeCN, DMF, DME, CHCl₃, CH₂Cl₂, THF, and acetone, which means the luminescent effect is maintained in various media. As confirmed by the results of CIE chromaticity analysis, which show that $x = 0.27$ and $y = 0.43$, the emission wavelength is situated in the green region, thus proving that the sample produces green light. Importantly, the fluorescence was turned off entirely when compound **1** was added to nitrobenzene, which is a deficient electron aromatic compound. The cool-down is probably caused by PET from the photosensitive complex to nitro. The particular quenching effect makes compound **1** an excellent fluorescent indicator for detecting nitroaromatic compounds connected to dangerous environmental pollution and potential explosions. Because nitrobenzene is preferred over other solvents, this complex may be important in chemical sensing by sensing dangerous materials in both solutions and mixtures. On the whole, the study shows how the researchers successfully created a dinuclear zinc complex that is structurally defined and active in light. Because of its strength, solvent-sensitive light-emission, and selective luminescence-suppression feature, this complex has great potential for more research in environmental monitoring, photoluminescent materials, and molecular sensing.

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