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ROBUST AND SELECTIVE ANALYSIS OF THE Co^{2+} IONS USING NEWLY PREPARED QUINOLINE-BASED CHEMOSENSORS AND DFT OPTIMIZATION

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ABSTRACT

A chemosensing method focused on sensitivity, selectivity, and reproducibility has been developed to identify Co^{2+} ions in various materials. Two new chemosensors (QHZ and QEDA) have been prepared by introducing the strong ligand groups (hydrazine and ethylenediamine) into quinoline molecules and characterized with spectroscopic techniques. High turn-off-fluorescence sensitivity, specifically to Co^{2+} ions, was demonstrated by both chemosensors. The fluorescence emission of QHZ and QEDA was at 507 and 502 nm, respectively, and they demonstrated outstanding photoluminescence properties. The sensing ability of the QHZ and QEDA toward Co^{2+} ions was tested by analyzing different samples from different sources at a nanomolar scale. The effect of the various functional groups in chemosensors was investigated in chemosensing analysis. The lowest energy structures and HOMO-LUMO orbitals of QHZ and QEDA were developed by performing DFT calculations. The interactions of Co^{2+} ions with chemosensors (QHZ and QEDA) were investigated by developing 3D structures of complexes using DFT.

Keywords: D-Amino Acid, Derivatives of Quinoline, Metal Ion Detection, Fluorescence Spectroscopy, Environmental Samples.

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INTRODUCTION

Cobalt is an essential element and is significant in biological and industrial applications.[1-3] Cobalt is a core metal ion in vitamin B_{12} (cobalamin), and its isotopes are used to detect and treat disease (cancer).[4] Cobalt is also considered an environmental pollutant in different aspects. As per WHO guidelines, 1.7 micromoles is the highest permissible amount of cobalt that can be present in drinking water.[5,6] The higher cobalt concentration causes adverse effects in the body, such as asthma, dermatitis, lung disease, thyroid enlargement, vasodilation, and heart disease.[7,8] At the same time, an inadequate amount of cobalt in the body might cause pathological disorders. In industries, cobalt is used in the preparation of catalysts, steels, magnetic materials, pharmaceuticals, the preparation of inorganic dyes, and cosmetics.[1-3,9] The development of sensitive, robust, and specifically selective fluorescent probes for detecting cobalt from various samples is essential due to the metal's extensive usage in industries and natural processes, and its advantages and disadvantages. The novel luminescent molecule development has become an active field of

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study in recent times due to the growing need for adaptable, versatile, and efficient luminous systems for chemical, biological, and material applications.[10-17] Substituted quinoline received the most excellent attention among the chromophores employed to create luminous materials due to its unique coordination and photophysical properties.[18] Quinolines are aromatic compounds that naturally arise as fused rings that belong to a heterocyclic class. Numerous industrial and medical uses have been found for quinoline and its derivatives.[11,12,14] Because of their aromatic structure of fused rings, quinolines exhibit exceptional luminescence and fluorescence. As a result, the development of quinolone-based luminous and fluorescent chemosensors has attracted much attention. Due to the nitrogen atom (electro-donor) in the quinoline structure, quinolone-based chemosensors have a high binding capacity with various analytes and provide susceptible analyte detection in multiple scenarios. Until now, quinoline has been derivatized at the second and eighth positions to create a variety of quinoline-based fluorophoric chemosensors.[11,12] Chemosensors based on quinoline have been applied to other areas, such as chromatographic applications, metal cation detections, and clinical applications.[12,14,15,19] The present report describes the synthesis of two novel chemosensors prepared on the basis chemical structure of quinoline (Scheme-1). The structure of quinoline was modified by introducing amino acid (D-valine) and hydrazine (HZ)/ethylenediamine (EDA) molecules, and this turned quinoline into highly sensitive chemosensors toward metal ions. A fluorophore chemosensing methodology for detecting metal ions from laboratory and environmental samples was developed using the synthesized chemosensors. Additionally, the stable structures, metal complexes, HOMO-LUMO, and energy gap optimisations were carried out using DFT simulations. The system's sensitivity and selectivity towards the cobalt ion were determined.

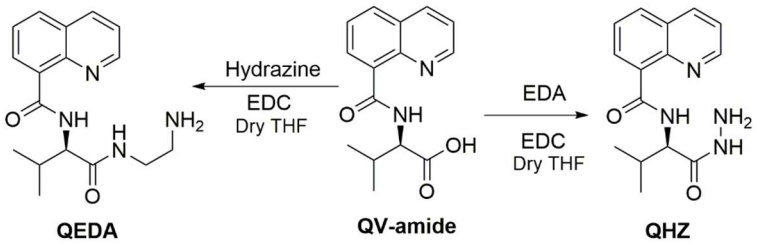
EXPERIMENTAL

Materials and Methods

D-valine, oxalyl chloride, hydrazine (HZ), Ethylene diammine (EDA), and quinoline derivative were ordered from Sigma-Aldrich. Environmental water samples were gathered in the surrounding areas. Avra Chemicals (India) supplied the investigation's solvent and other reagents.

Instruments and Equipment

The following instruments were utilised to characterise the synthesis products: FT-IR spectrophotometer (Agilent), Fluorescence spectrometer (Agilent), UV-spectrophotometer (UV-2450 Shimadzu), HR-MS (Bruker HD), NMR instrument (Jeol 500 MHz), and CHNS-elemental analyser.



Scheme-1: Synthesis of Quinoline-Based Chemosensors [QHZ and QEDA]

Synthesis of D-valine amide of 8-quinolinecarboxylic acid (QV-amide)

The catalytic amount of pyridine as a base and excess oxalyl chloride turned the carboxylic group of the quinolone-8-carboxylic acid into a highly reactive acyl chloride.[19-21] After that, an amide molecule was produced by substituting the acyl chloride from quinolone-8-carboxylic acyl chloride with the $–NH_2$ group of the chosen amino acids (D-valine).[21,22]

QV-Amide

Colour: white solid; yield: 98%; ^{1}H NMR (500 MHz, $CDCl_3$-*d6*) δ ppm: 0.93-0.98 (6H, m), 2.09-2.16 (1H, m), 4.25-4.30 (1H, m), 7.56-7.68 (2H, m), 7.93-7.94 and 7.95-7.96 (1H, dd), 7.97 (1H, d), 8.04-8.07 (1H, d) and 8.94-8.95 (1H, d). IR (KBr): 3833, 3656, 3115, 2983, 2036, 1927, 1821, 1741, 1712, 1414, 1345, 1301, 1032, 818 and 622 cm^{-1}. HRMS [$C_{15}H_{16}N_2O_3$] 273.11 ($M+H^+$); Anal. calcud. (found) % for $C_{15}H_{16}O_3N_2$: C, 66.17 (65.14); H, 5.93 (6.37); N, 9.18 (8.34).

Synthesis of Chemosensors (QHZ and QEDA)

Excess of EDA (361 mg, 0.006 mol) was mixed with the QV-amide (545 mg, 0.002 mol) and dissolved in 40 mL anhydrous THF in a dry N_2 atmosphere. In this mixture, a dry THF solution containing EDC (466 mg, 0.003 mol) and 4-DMAP (250 mg, 0.002 mol) was mixed dropwise and then left it be at room temperature for three hours with continuous stirring.[15] After the reaction, the excess of the reagents, DMAP and diurea product of EDC, were removed by extraction with aqueous 1N HCl solution and ethyl acetate, and pure product QEDA was obtained. Similarly, the chemosensor (QHZ) of quinoline with HZ (hydrazine) was prepared (Scheme-1). QHZ and QEDA were characterised using spectroscopic techniques, and the characterisation data are given below.

QHZ

Colour: pale-yellow sticky solid; yield: 99%; ^{1}H NMR (500 MHz, $CDCl_3$-*d6*) δ ppm: 0.90-0.91 (3H, d), 0.92-0.93 (3H, d), 2.07-2.13 (1H,m), 3.99 (1H, -NH_2), 4.38-4.43 (1H, m), 5.72 (1H, -NH-C=O), 7.56-7.63 (2H, m), 7.84 (1H, O=C-NH-), 7.93 (1H, d), 8.06 (1H,d), 8.24 (1H, d) and 8.94 (1H, d). IR (KBr): 3659, 3614, 3491, 3116, 2982, 2928, 2032, 1937, 1819, 1774, 1704, 1414, 1330, 1280, 1032, 872 and 678 cm^{-1}. HRMS [$C_{15}H_{18}N_4O_2$] 287.14 ($M+H^+$); Anal. calcud. (found) % for $C_{15}H_{18}N_4O_2$: C, 62.92 (62.46); H, 6.34 (7.11); N, 19.57 (19.89).

QEDA

Colour: yellow sticky solid; yield: 99%; ^{1}H NMR (500 MHz, $CDCl_3$-*d6*) δ ppm: 1.01-1.02 (3H, d), 1.03-1.04 (1H, d), 2.26 (1H,m), 2.98 (2H, m), 3.25 (2H, t), 3.34 (2H,m), 6.31 (1H, -NH-C=O), 7.56-7.62 (2H,m), 7.89 (1H, O=C-NH-), 7.93-7.95 (1H, d), 8.05-8.07 (1H, d), 8.24-8.26 (1H, d) and 8.94-8.95 (1H, d). IR (KBr): 3672, 3118, 2980, 2936, 2033, 1935, 1820, 1703, 1367, 1340, 1142, 1046, 882, 784 and 688 cm^{-1}. HRMS [$C_{17}H_{22}N_4O_2$] 315.18 ($M+H^+$); Anal. calcud. (found)% for $C_{17}H_{22}N_4O_2$: C, 64.95 (65.42); H, 7.05 (7.28); N, 17.82 (17.18).

RESULTS AND DISCUSSION

Under S_N2 substitution, the acid group of quinoline-8-carboxylic acid was transformed to acyl chloride in the presence of chlorinating reagents (oxalyl chloride and pyridine). The acylation of carboxylic groups is thought to benefit significantly from using oxalyl chloride as a chlorinating reagent and pyridine as a catalyst.[21,23] This combination produces over 100% of the desired product. Acyl chlorides are very sensitive towards nucleophilic reactions. When appropriate nucleophiles are present during the substitution reaction, it facilitates the facile synthesis of the amide bond and produces an impressive amount of products.[24-26] The acyl-chloride of quinoline-8-carboxylic acid was then converted to an amide with the D-valine in the presence of a catalytic quantity of pyridine; in this, the amino group of D-valine acts as a nucleophile and replaces chlorine from acyl-chloride. Nearly 100% of the required amide (QV-amide in Scheme 1) was produced under a fast reaction of the reactants. Then, using spectroscopic techniques, the produced and purified QV-amide was characterized.

Synthesis of Chemosensors

The carboxylic group of the QV-amide was modified to an amide of hydrazine or ethylene diammine in a condensation coupling reaction, where EDC and DMAP (non-nucleophilic base) were used as coupling reagents. EDC is an excellent reagent used in coupling reactions. It removes the hydrogen from amines or alcohol and the hydroxyl group from carboxylic acid groups, and functions as a dehydrating reagent. Thus, it yielded the desired chemosensors (QHZ and QEDA) with a high yield (about 98-99% yield).[14,15] The produced and purified chemosensors were then characterised using spectroscopy and chromatography (HPLC). Compounds based on quinolé are known to exhibit intriguing properties related to photoluminescence.[27-29] Therefore, the fluorescence properties of QHZ and QEDA (5×10^{-5} M) were investigated in a combination of buffer (HEPES) (0.5:9.5%, v/v) and polar organic solvent (MeOH). For the chemosensors QHZ and QEDA, the highest emission of the fluorescence intensity was measured at 507 and 502 nm, respectively. The fluorescence emission shown by QHZ and QEDA was arranged as follows: QHZ > QEDA. The electronic effect of the connected ligand group (HZ and EDA) significantly influences the fluorescence properties of the two chemosensors (QHZ and QEDA).

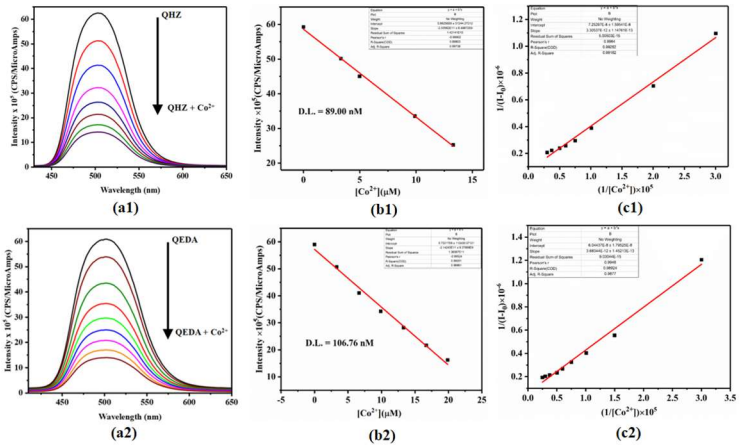


Fig.-1: Fluorescence Spectra of QHZ (a1) and QEDA (a2) Upon Incremental Addition of Co^{2+} Conc; the Limit of Detection of QHZ (b1) and QEDA (b2) with $Co^{2+,}$ respectively; and Binding Constant of QHZ (c1) and QEDA (c2) with Co^{2+} Respectively

Due to the presence of appropriate binding sites, the ability of chemosensors (QHZ and QEDA) for numerous metal ions such as Ag^{+}, Al^{3+}, Hg^{2+}, Na^{+}, Cr^{3+}, Zn^{2+}, Ni^{2+}, Pb^{2+}, Fe^{3+}, Cd^{2+}, Co^{2+}, and Cu^{2+}.were investigated. Several of these metal ions are known to be dangerous environmental pollutants that can be hazardous to human health when they are exhibited in excess. The fluorescence intensities of the QHZ and QEDA solutions did not change substantially when these metal ions were added, except for Co^{2+}. The peak intensity in the fluorescence spectrum at 507 and 502 nm, respectively, was quenched considerably upon the addition of Co^{2+} to the QHZ and QEDA solutions (Fig.-1(a1) and 1(a2) display the typical fluorescence spectra of QHZ and QEDA). The produced chemosensors (QHZ and QEDA) for Co^{2+} ions had their detection limit (LOD) determined, followed by IUPAC guidelines. Ten blank samples were also collected and quantified to estimate the relative standard deviation in the current investigations. As shown in Fig.-1(b1) and 1(b2), the determined LOD values of QHZ and QEDA towards Co^{2+} were 89.00 nM and 106.76 nM, respectively.[30] Compared to QEDA, QHZ has the lowest LOD towards Co^{2+}. The Benesi-Hildebrand method was used to determine binding constants of QHZ and QEDA towards Co^{2+} [21.9×10^3 M^{-1} and 16.4×10^3 M^{-1}, respectively; Fig.-1(c)]. Additionally, the constants of quenching for QHZ and QEDA towards Co^{2+} were calculated to be 96.6×10^3 M^{-1} and 87.4×10^3 M^{-1}, respectively, using Stern-Volmer plots. The upward bend and non-linear features in Fig.-2(a1) and 2(a2), which show the QHZ Stern-Volmer plot, suggest that a mix of static and dynamic quenching is accountable for the fluorescence quenching.[14]

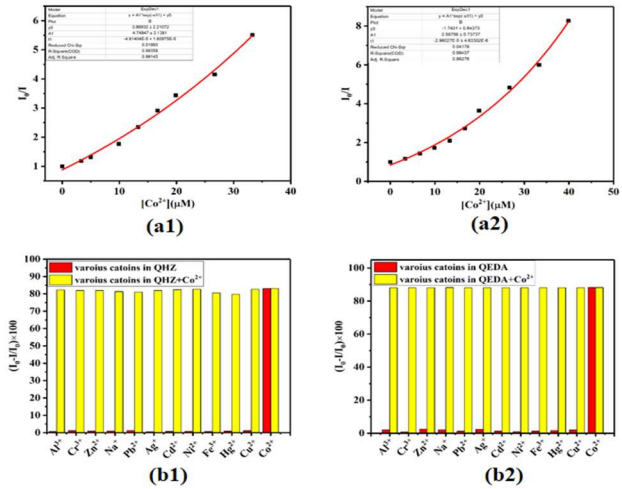


Fig.-2: Stern-Volmer Plot of QHZ (a1) and QEDA (a2) with Co^{2+} respectively, and Fluorescence Response of QHZ (b1) and QEDA (b2) after the Addition of Various Cations (Red Bars), Followed by the Addition of Co^{2+} (yellow bars)

To evaluate the exceptional selectivity of QHZ and QEDA towards Co^{2+} ions, we conducted comparative investigations employing a range of cations (Ag^{+}, Al^{3+}, Na^{+}, Cr^{3+}, Ni^{2+}, Pb^{2+}, Fe^{3+}, Cd^{2+}, Hg^{2+}, Zn^{2+}, and Cu^{2+}) present under similar experimental circumstances. These studies demonstrated that the capacity of QHZ and QEDA to detect Co^{2+} was not considerably hindered by other metal cations Fig.-2(b1) and 2(b2). As a result, QEDA and QHZ are incredibly selective fluorescence chemosensors for Co^{2+} identification.

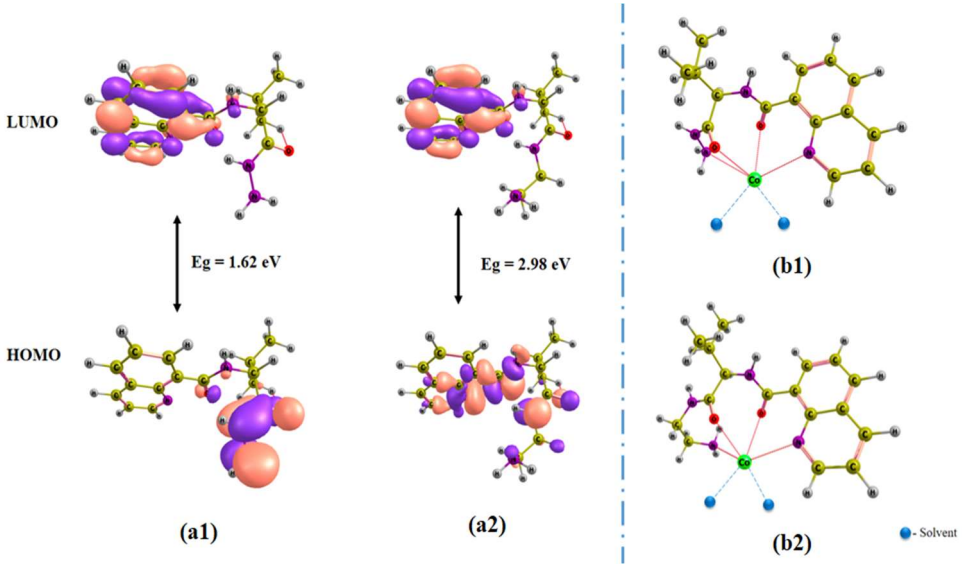


Fig.-3: DFT Optimised HOMO-LUMO Orbitals Diagrams of the Chemosensor Molecules [(a1) QHZ and (a2) QEDA] and DFT Optimised Energy Minimised Structures of the Metal Complex of Cobalt with (b1) QHZ and (b2) QEDA

The stoichiometry and mechanism of QHZ and QEDA interaction with Co^{2+} were confirmed by performing Job's plot analysis. Job's plot shows that the fluorescence intensity (λem) of the QHZ and QEDA varied with the mole fraction of Co^{2+}.[11,31] At 0.5-mole fraction, the two compounds' most incredible peak intensity in the fluorescence spectrum was obtained, demonstrating a 1:1 stoichiometry ratio. The DFT calculations were carried out using Gaussian software, and the minimized-energy stable structures of the prepared chemosensors (QHZ and QEDA; Fig.-3) and the Co^{2+} ions complexes of the chemosensors (Fig.-3) were generated.[14,32] The Homo-Lumo orbitals energy gaps for QHZ and QEDA were found to be 1.62 eV and 2.97 eV, respectively. QHZ chemosensor demonstrated better reactivity with Co^{2+} ions due to a reduced energy gap in Homo-Lumo orbitals.[14,27] Binding sites lead to the formation of chelating rings with Co^{2+} in the complex and form six-coordinated complexes, Fig.-3(b1 and b2). In the complex structure, the two vacancies were stabilised using the solvent that was utilised.

River Water Analysis

Two sample solutions were prepared to do this study: sample A, which is an environmental water sample obtained from a river that has been heated for five minutes, and sample B, which is an intentionally doped Co^{2+} river water sample.[33] In a H_2O: MeOH (0.5: 9.5%,v/v) solution, the QHZ chemosensor showed an excellent fluorescence emission at 507 nm. The addition of sample A to this solution did not appreciably change the QHZ fluorescence intensity. However, once sample B containing Co^{2+} ions was added, the intensity of the QHZ fluorescence was quenched Fig.-4(i).

Under the same experimental conditions, compound QEDA also showed the same results, suggesting that both compounds can detect Co^{2+} in river water samples. In river water, the detection limit for QHZ and QEDA towards Co^{2+} was calculated to be 78.51 nanomolar and 104.52 nanomolar, respectively. As a result, QHZ had the lowest LOD for Co^{2+} in river water compared to QEDA. The binding constants of QHZ and QEDA to Co^{2+} in river water were measured to be 29.8×10^3 M^{-1} and 12.7×10^3 M^{-1}, respectively. Furthermore, for QHZ and QEDA, the quenching constants towards Co^{2+} were measured to be 66.3×10^3 M^{-1} and 79.5×10^3 M^{-1}, respectively.

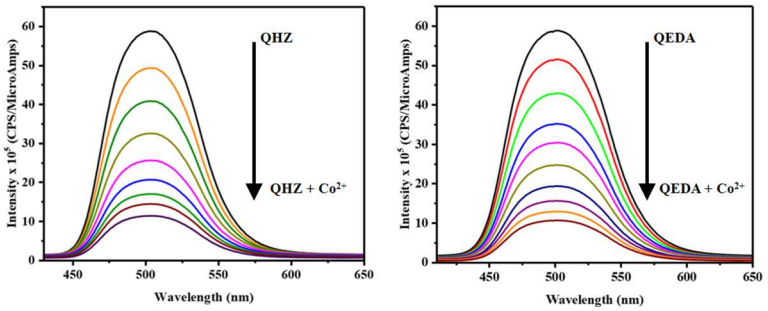


Fig.-4: Fluorescence Spectra of QHZ (i) and QEDA (ii) Upon Incremental Addition of Co^{2+} Concentrations

CONCLUSION

Quinoline-based chemosensors have been proposed and synthesized in this report. These chemosensors showed great affinity and fluorescence sensitivity for Co^{2+} and detected them at nanomolar concentrations. The research on quinoline-based class chemosensors showed that the substituent's electronic effects play an important role in controlling and manipulating the chemosensing and photoluminescence properties. Chemical analysis may be utilized with these chemosensors to detect Co^{2+} traces in rivers and other waste. In conclusion, tuneable chemosensors based on quinoline-8-carboxylic acid moieties have been generated for the first time, forming a unique class of well-defined chemosensors. In addition to the chemosensor applications under investigation in this work, previous research indicates that these compounds have significant promise as electroluminescent and electron-transport materials. Similar chemosensors can be developed with great potential for the aforementioned applications by analysing their features.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-9 Industry, Innovation and Infrastructure.* All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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