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History of Physiochemical and Structural Hydroxyquinoline Complexes

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Abstract
In this review, we studied a group of studies on the complexes of hydroxyquinoline ligands and their derivatives with divalent or trivalent metals, their physical, spectral, and structural properties, such as X-ray, UV-vis, infrared, fluorescence, luminescence, absorption, and emission properties.

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1. Introduction

Hydroxyquinoline ligands—particularly 8-hydroxyquinoline (often abbreviated 8-HQ or oxine) and its derivatives—have long occupied an important place in coordination chemistry because of their strong chelating ability, with nitrogen (from the quinoline ring) and oxygen (from the hydroxyl) donors providing stable binding to many metal centers [1]. The history of their physiochemical and structural characterization spans nearly a century and has moved through phases of analytical, spectroscopic, crystallographic, and computational methods.

Early in the 20th century, hydroxyquinoline was used largely as an analytical reagent. Its ability to form colored complexes with metal ions enabled it to serve in spectrophotometric determinations and gravimetric analysis [2]. Over time, as synthetic coordination chemistry matured, systematic studies of stability constants, ligand substitution, metal-oxidation state effects, and geometries became common. Physicochemical parameters such as solubility, redox potentials, luminescence, and thermal stability were measured to understand how ligand modifications (e.g., substituents on the quinoline ring) influence the electronic structure and reactivity of complexes [3].

Structural investigations (X-ray crystallography, powder XRD, single crystal studies) augmented this, confirming the chelate geometry (often bidentate N,O binding), coordination numbers, and the influence of steric and electronic substituents on geometry (e.g. distortion from ideal octahedral or tetrahedral, bridging modes, polynuclear clusters). Infrared, UV-Vis, NMR, and later computational DFT/TD-DFT studies have enriched understanding of the bonding, charge transfer transitions, and photophysical behavior (e.g. fluorescence, electroluminescence) of hydroxyquinoline complexes.

In recent decades, interest has also grown in applications—antibacterial, anticancer, material science (OLEDs etc.)—which has spurred more refined structural studies (single-crystal X-ray, mixed ligand complexes, etc.), and computational work to rationalize observed properties [5].

2. History of complexes (1956-1999)

In 1956, Charles et al. [6] have studied the spectra of infrared absorption have been examined for solid 8-hydroxyquinoline, 2-methyl-8-hydroxyquinoline, 4-methyl-8-hydroxyquinoline, and several metals chelates that are obtained from these substances. It was discovered that the metal chelates' spectra, which were obtained from a particular reagent, were generally rather similar. However, there were a number of notable variances that could be helpful in an analytical sense. The spectra of the chelates of all three reagents identified an absorption peak at roughly 9 μ. The C-O vibrations in these molecules are thought to be connected to this peak. For the transition-metal 8-hydroxy-quinolates, it is proposed that this could either show that 3d orbitals are involved in metal-ligand bonding or that the two classes of chelates have different crystal structures.

In 1984, Satake et al. [7] have developed iron (II) can be determined spectrophotometrically using a method that involves extracting its ternary complex with 2,2′-dipyridyl and tetraphenylborate into molten naphthalene. 2,2′-dipyridyl and iron (II) combine to generate a color complex that is soluble in water. When sodium tetraphenylborate is present, this complex cation forms a stable ternary complex that is insoluble in water. This complex can be readily extracted into molten naphthalene in the pH

range of 2.8–7.6 by shaking vigorously for a short while. Filtration is used to separate and dissolve the solid naphthalene that contains the iron 2,2′-dipyridyl tetraphenylborate complex in acetonitrile. A reagent blank is used to measure the absorbance at 521 nm. The concentration range of 2.2 to 65.5 µg of iron in 10 ml of acetonitrile solution complies with Beer's law. Sandell's sensitivity and molar absorptivity at 521 nm are 0.006 3 µg cm–2 and 8.89 × 103 l mol–1 cm–1, respectively. A great deal of research has been done on the interference of different ions. The technique has been used to measure the amount of iron in common metallic samples, and the outcomes are contrasted with those of the 1,10-phenanthroline approach.

In 1999, das Cracas et al. [8] have proposed a 7-(4-nitrophenylazo)-8-hydroxyquinoline-5-sulfonic acid (p-NIAZOXS), an 8-hydroxyquinoline derivative, is suggested as a novel spectrophotometric reagent for the quick measurement of zinc (II) in a sensitive and selective manner. The response of the p-NIA. At pH 9.2 (borax buffer), ZOXS and zinc (II) are immediate, and the absorbance stays constant for more than 24 hours. With a molar absorptivity of 3.75x104 L mol-1 cm-1 and a detection limit of 15 ng mL-1, the method enables the determination of zinc over the range of 0.05-1.0 µg mL-1. The suggested technique has been effectively used to measure the amount of zinc in a number of copper alloys and pharmaceutical formulations. The accuracy and precision (R.S.D. < 2%) were both satisfactory.

In the same year, Johansson et al. [9] have studied the tris-8-hydroxyquinoline electrical structure! Both in its pristine molecular solid form and after contact doping, aluminum (Alq3) has been examined! together with lithium and potassium. Based on a combination of quantum-chemical calculations at the density functional theory level and x-ray and ultraviolet photoelectron spectroscopies, we present the findings of a combined theoretical and experimental study. Every electron that is transferred from an alkali metal atom to one of the three ligands of the Alq3 molecule is stored upon doping, creating a new spectral characteristic called ~peak! in the valence band, which changes consistently as the number of metal atoms per Alq3 molecule increases from one to three.

3. History of complexes (2000-2010)

In 2000, Brinkmann et al. [10] have high light levels The path to designing inexpensive large area displays and illuminators was paved by low-voltage driven devices based on tris(8-hydroxyquinoline) aluminum (III) (Alq3). Even though this material has been the subject of several studies, little is known about its fundamental structural and optical characteristics in the solid state. Thus, in a variety of Alq3 systems, such as solution, amorphous thin films, and various crystalline forms, we have examined the structure or structures and the relationship between intermolecular interactions and optical characteristics. The crystalline structures of two new unsolvated polymorphs of Alq3, α-Alq3 and β-Alq3, have been synthesized and identified using X-ray diffraction data on powders (α) and single crystals (β).

By using absorption, fluorescence excitation, fluorescence, and Raman spectroscopy to investigate the various crystal phases as well as amorphous thin films and solutions, it was possible to clarify the nature of the photoexcitations, highlight the vibrational fingerprints of the α and β crystalline forms, and determine how the molecular packing affected the emission properties. As a result of distinct dispersive and dipolar interactions as well as distinct π–π orbital overlaps, the fluorescence's spectral position is found to be connected with the molecular density of the packing and the length of inter ligand contacts between nearby Alq3 molecules (the shorter the contacts, i.e., the denser the crystal, the more the fluorescence is red-shifted).

In 2001, Shen et al. [11] have studied Tris (8-hydroxy quinoline) aluminum and magnesium (Mg) and aluminum (Al) interfaces are investigated using x-ray photoemission spectroscopy, ultraviolet photoemission spectroscopy, and current-voltage measurements to determine the chemistry, electronic structure, and electron injection properties at these interfaces.

Interfaces between Alq3 and metal as well as between metal and Alq3 are examined. To get rid of extrinsic effects linked to interface contamination, all interfaces are constructed and tested in ultrahigh vacuum. The tendency of magnesium and aluminum to generate covalent metal-carbon bonds when the metal is placed on the organic film, bonding result in wide and highly reactive surfaces. We suggest the creation of an organometallic structure for this deposition sequence in which a single metal atom binds to the pyridyl side of the molecule's quinolate ligand and coordinates with an oxygen atom of either a neighboring molecule or another ligand.

In the same year, Raj et al. [12] have found that the NiII ion is coordinated by the N and quinolinate-O atoms of HQS and four water mol¬ecules, exhibiting a distorted octahedral geometry. They have also found that the overall structure is stabilized by π–π* stacking interactions between the ring moieties of HQS in the neighboring complex molecules.

In the same year, Albuecht et al. [13] have used a series of amide- and urea-substituted 8-hydroxyquinoline ligands 1/6-H are used for the formation of zinc (II) complexes. They have also

showed that only the derivatives of 7-amino-8-hydroxyquinoline 4-H and 5-H form trinuclear hexa-helical 6:3 complexes which exhibit interesting structural and NMR and fluorescence spectroscopic properties.

In 2002 Sahin and his group [14] have prepared a zinc complex with nicotine amide Dibromobis (nicotinamide) zinc (II) with the formula $[ZnBr_2(na)_2]$ which the structure in Fig. (1) and studied the complex by UV-Visible spectroscopy, FT-IR and X-ray diffraction in addition to Accurate analysis of (C.H.N) elements and measurement of bromine content by potentiometric titration using silver nitrate.

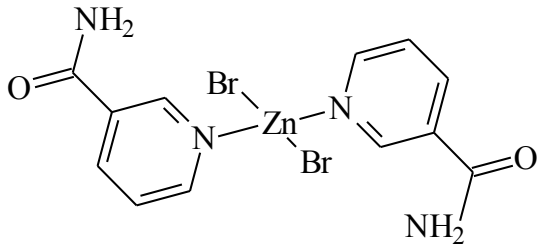

Fig. (1) Structure of $[ZnBr_2(na)_2]$ complex

In 2003, Hairong et al. [15] have synthesized and purified organic electroluminescent material (Alq3) for OLEDs. They have found that the coordinate interaction between Al^{3+} and O ion is more intensive than that between Al^{3+} and N ion. A very intensive chelation of aromatic ring was indicated. Fluorescence data confirmed the existence of a fluorescent species formed between Al ion and 8-hydroxyquinolinol.

In the same year, Gurnani et al. [16] have immobilized the hydroxyquinoline on cellulose via a moderate size -NH-CH2-CH2-NH-SO2-C6H4-N=N- linker and the resulting macromolecular chelator (and intermediates) was characterized. It has been used for enrichment of Cu (II), Zn (II), Fe (III), Ni (II), Co (II), Cd (II) and Pb (II) prior to their determination by flame atomic absorption spectrometry. The tolerance limits of electrolytes NaCl, NaBr, NaNO3, Na2SO4, Na3PO4 and cations Ca2+ and Mg2+ (added as chloride and sulphate, respectively) in the sorption of all these metal ions were reported.

In 2005, Xie et al. [17] have synthesized a novel 8-hydroxyquinoline derivative with a carbazole group substituting in the 5-position of quinoline, where carbazole acted as a hole transporter. They have obtained single crystal of this 8-hydroxyquinoline derivative and also synthesized its coordination complex with Al (III). This complex with the ability of whole transportation and electron transportation simultaneously was soluble in common organic solvents and showed yellow luminescence with high quantum yield.

In the same year, complexes of some transitional element ions with nicotine amide and thiocyanate were prepared and diagnosed [18] with the chemical formula [M(na)2(SCN)2] as [M(II)= Co (II), Cu (II), Fe (II) and Cd (II)].

In 2008, Jiang et al. [19] have prepared the beta-cyclodextrin (beta-CD) inclusion complex containing bis(8-hydroxyquinoline) magnesium. They have indicated that the formation of inclusion complex in which the quinoline rings of the guest were encapsulated within the beta-CD cavities. The association constant calculated with the modified Benesi-Hildebrand equation at 25°C was determined. The thermal stability and solubility of bis(8-hydroxyquinoline) magnesium were improved when forming inclusion complex.

In the same year, Khaorapapong et al. [20] have formed bis(8-hydroxyquinoline) Zn in the interlayer spaces of smectites by solid-solid reactions between Zn (II)-smectites and 8Hq at ambient condition. The photoluminescence intensity of the Znq2 complex in synthetic saponite was higher than that of the complex in montmorillonite, suggesting the very low content of quenching impurities in synthetic saponite. The difference in the luminescence bands were thought to be caused by the different molecular structure and molecular packing of the complex formed in the interlayer spaces.

In 2009, Yan et al. [21] have firstly synthesized a fully conjugated ligand B8QPC containing hole- and electron-transporting groups through the Wittig condensation polymerization. Corresponding polymeric complexes, B8QPC (1) with Cu(II) (2) and Zn(II) (3), were synthesized and characterized. UV-visible and fluorescence spectra at room temperature revealed that both the polymeric complexes 2 and 3 emit blue luminescence at wavelength 473 and 513 nm in DMSO solution and blue/green luminescence at 522 and 531 nm in solid state, respectively. Thermal property measurements show that they have good thermal stability.

In 2010, Wang et al. [22] have synthesized large-scale and high-purity bis(8-hydroxyquinoline) magnesium nanoribbons via a facile solvothermal route. The room-temperature photoluminescence spectrum of the products showed a strong and stable blue-green emission centered at 482 nm under excitation at 378 nm. As the light source was switched on and off, the currents could be reversibly switched between high and low values, which might be useful in the fabrication of photosensor and photo switch microdevices or nanodevices.

4. History of complexes (2011-until now)

In 2012, Fayad et. al. [23] have been synthesized and identification of the mixed ligands complexes of

M (II) Ions in general commonly abbreviated (LynH) as a primary ligand and 1,10-phenanthroline (C12H8N2) commonly abbreviated as" phen," as a secondary ligand. The ligands and the metal chlorides were brought in to reaction at room temperature in ethanol as solvent. The reaction required the following molar ratio [(1:1:2) (metal): phen:2 Lyn-] with M(II) ions, were M= Mn (II), Cu (II), Ni (II), Co (II), Fe (II) and Cd (II) as shown in Fig. (2).

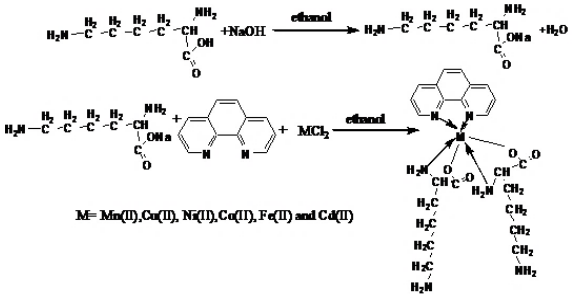

Fig (2) 4-hydroxyazobenzene identified by IR spectrum

In 2013, Fayad et. al. [24] have been prepared a new six mixed Ligand complexes of some transition metal ions, such as Mn (II), Fe (II), Co (II), Ni (II), Cu (II) and non-transition metal ion Cd (II)) with L-valine (Val H) as a primary ligand and 1,10-phenanthroline (phen) as a secondary ligand with method as shown in Fig. (3).

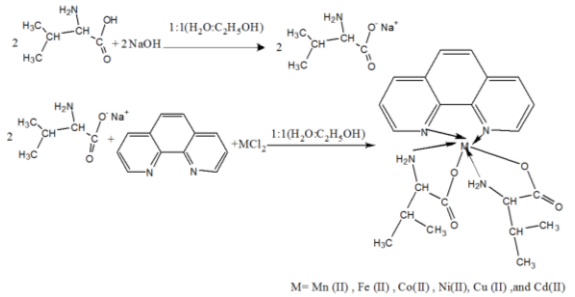

Fig. (3) Schematic representation preparation of the complexes [M(Val)2(phen)]

In 2014, Pimchan et al. [25] have incorporated a luminescence metal complex (Znq2), into the interlayer space of smectites (a natural montmorillonite and a synthetic saponite) by two different ways; colloidal processes via the in-situ formation of bis (Znq2) in CTA-smectites and the adsorption of Znq2CTA into hydrophilic smectites. The photoluminescence bands of Znq2CTA@montmorillonite and Znq2@CTA-montmorillonite (498 nm) were blue shifted in comparison with the weak emission bands of Znq2CTA@saponite and Znq2@CTA-saponite (510 nm), indicating the formation of different nanostructures and/or packing of bis(8-hydroxyquinoline) zinc (II) in the two types of smectites.

In 2016, Li et al. [26] have used the reaction of BiCl3 with 2-(2-hydroxy-3-methoxyphenyl) benzimidazole (HL) in tetrahydrofuran (THF) under reflux to prepare mononuclear complex of formula [Bi(HL)2Cl3•H2O]. They have investigated the binding interaction of the complex with bovine serum albumin (BSA) using the fluorescence quenching method. Their experimental results showed that the binding of the complex to BSA could change the microenvironment and conformation of BSA.

In the same year, Shohei et al. [27] have presented a treatment of anodic porous alumina (APA) plates in heated water containing 8-Hq to produce crystalline tris (Alq3) micro belts. These micro belts were found to aggregate to form flower-like structures on the surface and they exhibited green photoluminescence with a peak at around 520 nm. This reaction was induced at the APA surface by the reaction between the HQ and amorphous Al2O3 species.

In 2017, Petrova et al. [28] have synthesized novel luminescent organic-inorganic hybrid materials based on 8-Hq metal complexes (Liq, Kq, Naq, Rbq, Mgq2, Srq2, Znq2, Scq3, Alq3, Gaq3 and Inq3) by a high temperature exchange reaction with 80PbF2–20B2O3 inorganic low-melting glass. All hybrid materials showed a wide luminescence band in the range 400–700 nm.

In 2018, Gaml [29] has studied the effect of UV irradiation on the optical constants of -5-Sulfono-7-(4-Cloro phenyl azo)-8-Hydroxy Quinoline (SAHQ-Cl) compound, which has amorphous thin film structure for the pristine film and partially crystallizes upon UV irradiation. They have found that the UV irradiation increased the band gap from 2.44 to 2.8 eV, increased the fluorescence emission intensity, and UV irradiation remarkably changed the material dispersion parameters. They also have calculated the nonlinear optical parameters and found to increase upon increasing UV irradiation time.

In the same year, Shinde et al. [30] have investigated the photophysical behavior of Znq2 chelate under various environments such as temperature, humidity, UV radiation, acidic and basic solvents and polymer matrix in order to explore the effect of these parameters on the photoluminescence spectra. Their results prove that the emission from the synthesized metal complex can be tuned in the wavelength range of 433-524 nm by selecting the solvent or the polymer according to the requirement, inferring its potential as emissive material for the fabrication of OLED displays as

well as in other optoelectronic applications where green light is essential.

In the same year, Keshmiri et al. [31] have investigated an organo-metallic complex based on zinc ions (Znq2). They have produced ZnO nanoparticles and added them to the Znq2 complex. They have found that the complexes have a high thermal stability in the air atmosphere.

The maximum intensity of the photoluminescence spectrum of Znq2 occurred at 565 nm and showed a blue shift to 511 nm by adding ZnO nanoparticles to the Znq2 complex. The optical and electrical properties of the Znq2 and the mixture of Znq2 and ZnO nano powders were studied in order to find any possible applications in organic light emitting devices.

3. Conclusion

In conclusions, hydroxyquinoline and its derivatives have played a significant role in coordination chemistry since the early 20th century, owing to their strong chelating ability through both nitrogen and oxygen donor atoms. The earliest studies in the 1920s and 1930s highlighted the compound 8-hydroxyquinoline (oxine) as a versatile analytical reagent, especially in gravimetric and spectrophotometric determination of metal ions. Over time, extensive physiochemical investigations—including solubility, stability constants, and spectroscopic properties—demonstrated the remarkable affinity of hydroxyquinoline for transition and rare earth metals. By the mid-20th century, X-ray crystallography and later advanced spectroscopic methods provided detailed insights into the structural features of these complexes, confirming their bidentate coordination modes and varied geometries. Today, hydroxyquinoline complexes continue to attract interest not only for their historical importance in analytical chemistry but also for their applications in catalysis, medicine, and material science.

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