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Research Article

Synthesis and characterizations of Hg (II) Complex of Macrocyclic complexes compounds Tetradecahydrodibenzo hexaazacyclooctadecine with HgX2 (X= Cl, Br) by Hirshfeld analysis and antimicrobial activity

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Abstract

This study examines the synthesis and characterization of a macrocyclic complex molecule having the general formula [M-LX_2], where M =Hg (II) with schiff base Tetradecahydrodibenzo hexaazacyclooctadecine ligands. Resulted from the interaction between mercury and the diethyl triamine and benzene- 1, 2 diol in an ethanolic solution. Schiff base and its novel mercury (II) Complexes were studied vibrational in the solid state using spectral, ^{1}H-NMR, IR, UV, and antimicrobial activity approaches. We were able to establish the coordination mode of the metal in complexes by comparing the changes in the ligands' and complexes' FT-IR and UV-Visible spectra.

Keywords: MERCURY (II), Schiff bases, IR, ^{1}H-NMR, PXRD Spectroscopy.

1. Introduction

Mercury has contributed in a variety of ways to the field of bio-conjugate chemistry. Its special electrical characteristics have been helpful in developing electrochemical-based thiol test techniques [1.] Mercury metal has helped to characterize protein using electron microscopy [2]. Along with many other metals, it also creates useful amalgams that are used in a variety of contexts [3]. The fact that Schiff base ligands are easily produced by condensation of primary amines with carbonyl compounds, aldehydes, or ketones makes them preferred ligands. In honor of German chemist and Nobel Prize laureate Hugo Schiff, who made the discovery of Schiff bases in 1864, they bear his name [4]. Schiff bases are regarded as an extremely significant class of chemicals due to their capacity to form complexes with transition metal ions. Schiff base ligands are readily synthesized, and they combine with nearly all metal ions to form complexes. Azomethine nitrogen is used to coordinate with metal ions [5]. Schiff base ligands have been thoroughly researched in coordination chemistry, mostly due to their availability, ease of synthesis, and electrical characteristics.Recent years have seen a major increase in interest in Schiff base coordination chemistry because of its important functions in analytical chemistry, organic synthesis, metal refining, electroplating, metallurgy, and photography [6]. The physical characteristics of crystals, like bulk density and flowability, are closely linked to their appearance [7]. Despite its seeming simplicity, 1,2-dihydroxy-benzene (catechol) can interact with its surroundings in a variety of ways. This is because it has two hydroxyl groups and an aromatic ring, which can form strong covalent bonds as well as less obvious non-covalent interactions [8, 9]. Which has the ability to function as a chelating agent since it can act as a hydrogen donor or acceptor in an intra- or intermolecular hydrogen bond interchangeably [10, 11, 12]. A more thorough investigation of the distinctive characteristics of the catechol functionality, which includes two neighboring phenoxyl groups with chelating abilities, would, however, indicate the functioning of different interaction geometries in addition to the usual cation-π [13]. There are minute amounts of this colourless substance in nature [14]. Destructive distillation of the plant component catechin led to its initial discovery. Nowadays, synthetic catechol is generated in large quantities as a basic organic compound, primarily as a building block for insecticides, flavours, and scents [15-16]. Since then, it has been shown that catechol can be found in free form in beech wood tar and Kino.

Its sulfonic acid has been found in both human and horse urine [17]. Together with the enzyme polyphenol oxidase, also referred to as catecholase or catechol oxidase, small levels of catechol are found naturally in fruits and vegetables [18].

The colourless catechol oxidises to produce benzoquinone-derived reddish-brown melanoid colours. By adding an acid, such as the citric acid found in lemon juice, the enzyme is rendered inactive. The browning reaction is also stopped by excluding oxygen. But as the temperature drops, the enzyme's activity rises. According to claims, benzoquinone has antibacterial properties that reduce the decay of harmed fruits and other plant materials [19]. Catechols are abundant in nature and biologically reactive compounds that are extensively employed in nanotechnology and biomedicine [20, 21, 22]. Numerous branches of chemistry and materials science deal with catechol-functionalized molecules and materials [23]. Particularly for creating: (i) sticky functional hydrogels for biological applications [24] (ii) metallopolymer networks for water purification and (iii) polymers with antibacterial qualities [25]. Catechols are readily oxidised by photooxidation, chemical agents, enzymatic oxidants, autoxidation in the presence of molecular oxygen, and photooxidation [26, 27, 28]. According to reports, immunological, sensory, neurological, motor, and behavioral dysfunctions can be brought on by exposure to mercury (II) and propagate up the food chain [29, 30]. Thus, it is critical to create novel techniques for Hg (II) detection and quantification. The excellent sensitivity and selectivity, fast reaction time, and simple signal detection of Hg^{+2} probes have made them extremely helpful in recent times[31-36.] There is still a need for more probes for the analysis of this ion in various types of samples in terms of the sample matrix and concentration ranges, despite the introduction of numerous types of Hg^{+2} ion-sensing ionophores to detect mercuric ions in chemical and biological system[37,38.] Therefore the development of Hg^{+2} selective probes is still a challenge [39-41].

2. EXPERIMENTAL

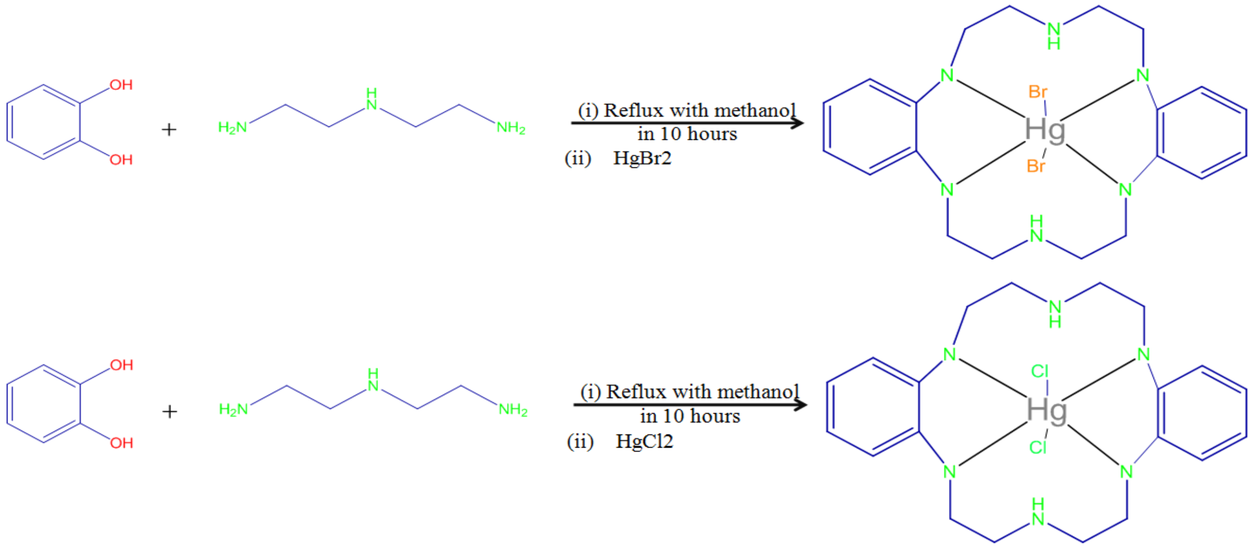
Standard processes were used to purify ligand and solvent precursors of reagent grade (BDH). E. Merck provided the mercury (II) chloride which was utilised unpurified. Every other chemical was obtained from commercial sources and utilised exactly as supplied. Metals and chloride were determined volumetrically and gravimetrically respectively. Mercury was estimated by standard method. Infrared (IR) spectra of ligands from 4000 to 500cm^{-1} of all prepared complexes in CsI pellets were recorded with a JASCO 350 FT-IR spectrophotometer. ^{1} H NMR spectra, recorded in $CDCl_3$ using a JEOL JNM-ECZ500R/S$_1$spectrometer with Me_4Si as an internal standard, were obtained at the IIT Kanpur, India. Powder X-ray diffraction (PXRD) was recorded on a ASCII CLASS PC using Cu-K β radiation .UV absorption spectra of the these compounds in different buffer solution at room temperature were recorded using SHIMADZU 3600 Plus UV-VIS NIR spectrophotometer.

SYNTHESIS

Synthesis of ligands. To ethanolic solution (20ml) of HgX_2 (X=Cl, CH_3COO^-, NO_3^-) (1mmol) a solution of (2mmol) benzene 1, 2 diol was added drop wise with constant stirring. This was followed by Dropwise addition of diethyl triamine (2mmol) in ethanol (25ml) with constant stirring for 8hrs. A brown solid appeared which was filtered, washed with ethanol and dried under vacuum over $CaCl_2$ $[HgL^1X_2]$scheme 1.

Synthesis of the Hg (II) Macrocyclic complexes compounds

To ethanolic solution (20 ml) of HgX_2(X=Cl, Br) (1 mmol) a solution of benzene 1,2 diol(2mmol) was added Dropwise with constant stirring. This was followed by Dropwise addition of diethyl triamine (2mmol) in ethanol (25 ml) with constant stirring for 8hrs.A brown solid appeared which filtered ,washed with ethanol and dried under vacum over $cacl_2$ $[HgL^2X_2]$.scheme.1



Scheme 1. Synthesis of macrocyclic complexes of Hg (II)

RESULTS AND DISCUSSION

These synthesised complexes were brown solid and stable at room temperature. All the complexes have showed high melting points.The compounds were prepared in good yield.

FT-IR Spectra

The IR spectral analysis of compounds is in good arrangement with the synthesis compounds. The infrared spectra of the metal complexes shown the absence of the stretching modes of functional groups (NH_2 and OH) and the for C-H bending vibrations appear at ca.4000-500cm^{-1}.The presence of new bands in the spectra of the metal complexes in the region at 7800-349cm^{-1} due to (Hg-N) vibration supports the coordination of the imine nitrogen to the mercury(II).In the spectrum of the complexes the bands at cm^{-1} are observed which may be assigned to coordinated chloro, acetate and nitrate group respectively. The additional distinctive peak of compounds in figure (1). These data are in agreement with reported for JASCO in MNNIT, Prayagraj, Uttarpradesh.

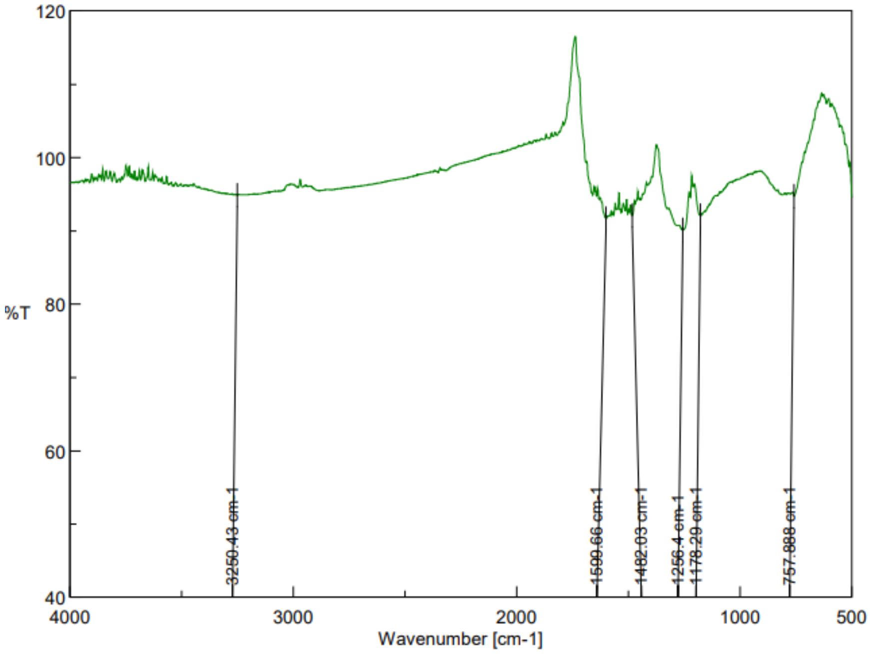
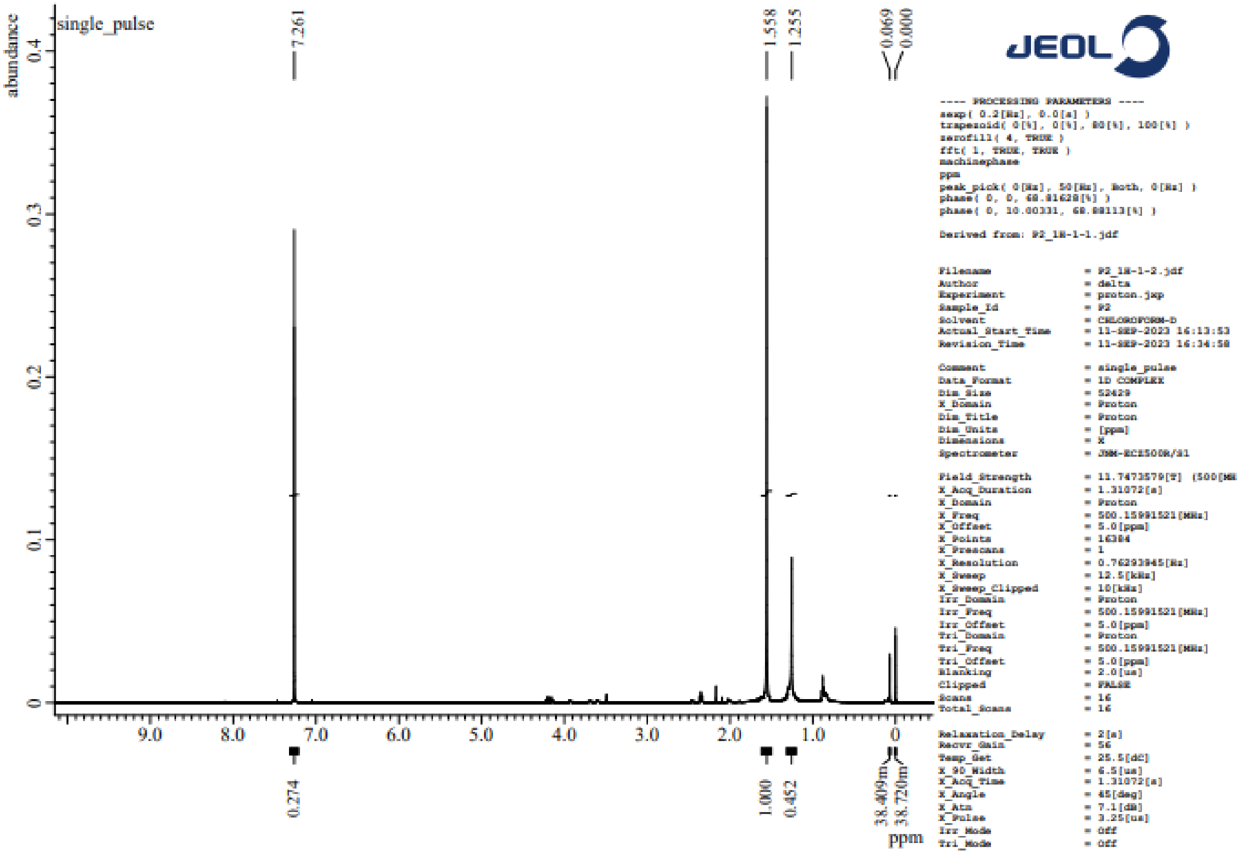


Figure 1: FT-IR spectra of mercury (II) complexes compounds

^{1}HNMR spectra

^{1}H-NMR spectra of the prepared mercury (II) compounds were performed in denaturated $CDCl_3$ solutions with tetramethylsaline as an internal standard. All these spectra showed a peak at 1.5 ppm which was due to $CDCl_3$ solvent and some spectra showed a sharp peak at 7.2 ppm due to dissolved water in $CDCl_3$. Figure 2. Represent the ^{1}HNMR spectra of the compounds the data of these (JEOL).



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Tri_Mode                  = Off
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Figure 2: ^{1}HNMR spectra of mercury (II) complexes compounds

Powdered X-RAY Diffraction Studies

PXRD is widely used to characterize a crystalline solid. XRD spectrum of clearly showed crystalline material. Diffraction pattern comprises a number of sharp Bragg's reflaction corresponding to different orientation of crystalline. Non-uniform broadening of lines is attributed to anisotropic crystallites, used here to calculate the crystallite size using Debye Scherrer formula. Fig.3 Using ASCII CLASS in GONIO Lab, prayagraj, uttarpradesh the generated materials' crystallite size is 1.54 Å.

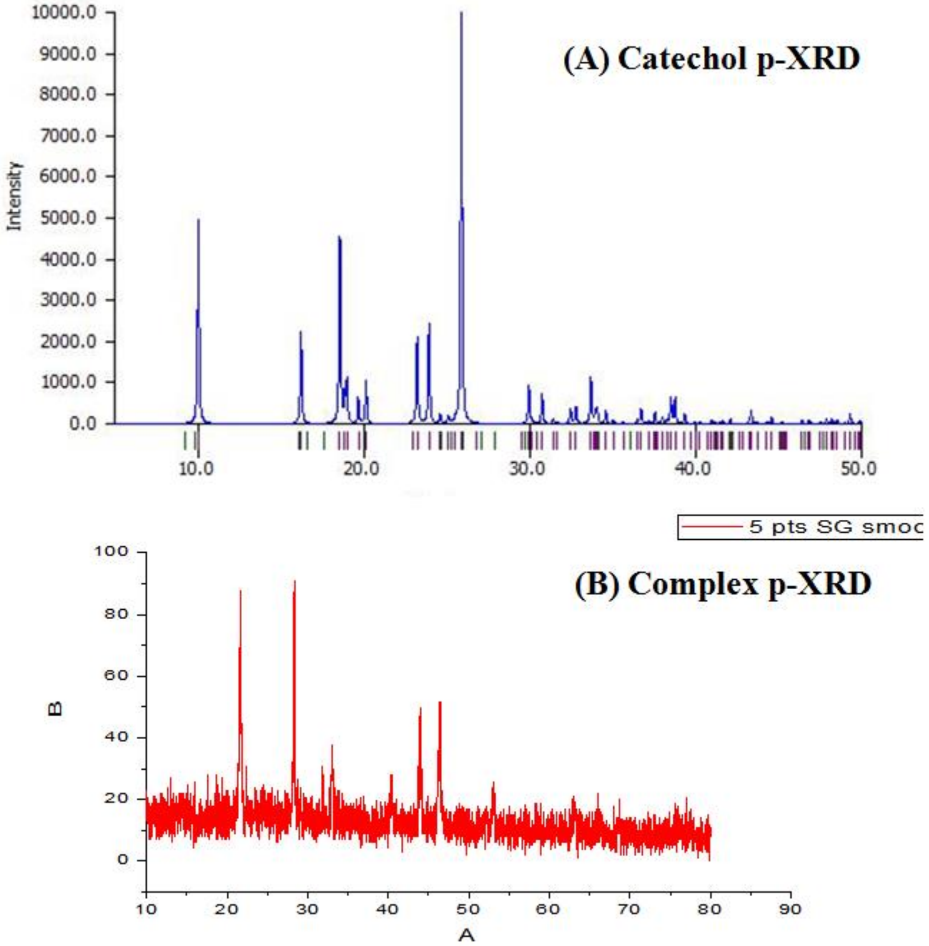


Figure 3: Powder-XRD of a) catechol derivative ligands peak, b) mercury (II) complex compounds peak.

Hirsh feld analysis of Catechol molecule

A Hirshfeld surface analysis was implemented for an outstanding qualification of the inter/ intra molecular interaction present in the crystal structure by using Crystal Explorer 17.5 program. The crystal structure of Molecule is an monoclinic crystal system in the P 21/c space group. It's a, b, and c values are 9.8326(18)Å, 5.5910(11)Å, and 10.467(2)Å and α = 90°, β= 114.988(4)° and ϒ= 90° in that order.[42] More scrupulous stabilizing interactions can be readily identifiable by means of 3D Hirshfeld surface and 2D molecular fingerprint analysis, which point to this occurrence of intermolecular interactions of different types and provides global insight into their key role in the packing. The descriptor d_{norm} is used to map the directions and intensities of intermolecular interactions (i.e. red speck) on the within the molecular crystal onto Hirshfeld surfaces, has demonstrated the existence of C-H··O and C-O···H intermolecular interactions. Intermolecular π··π interactions are shown by triangle (i.e., red circle) on shape index [43] (figure 4).

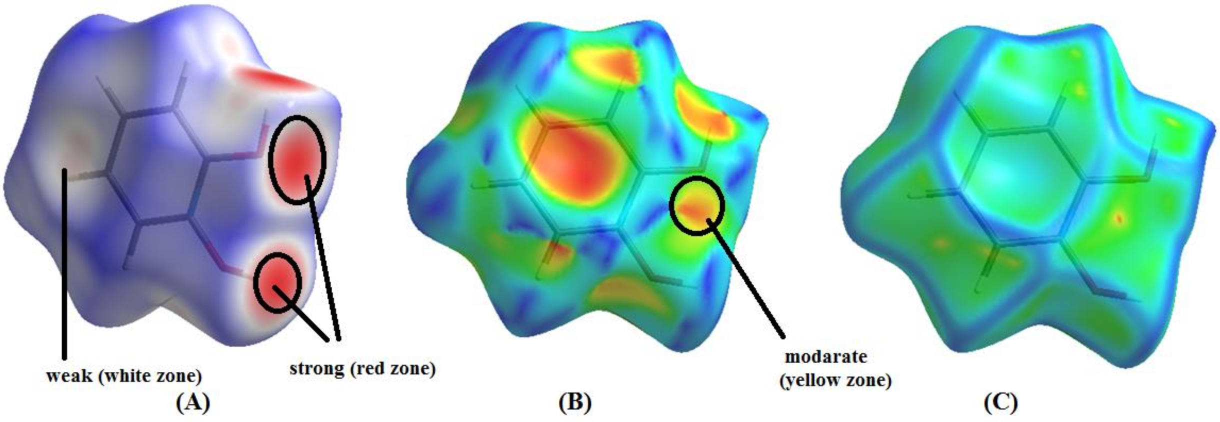


Figure 4: Hirshfeld surface for Catechol crystal compounds mapped with (A) denorm, (B) shape index and (C) curvedness.

The extent of intermolecular stacking interactions between these compounds can be derived from their 2D fingerprint plots (figure 5), indicates that intermolecular interactions especially O···C interactions in is found to be 6.5% and O···H is found to be 21.2% in these starting compound. Further intermolecular interaction such as C···H is found to be 23.6% and the major part of interaction such as H···H is found to be 47.4%.

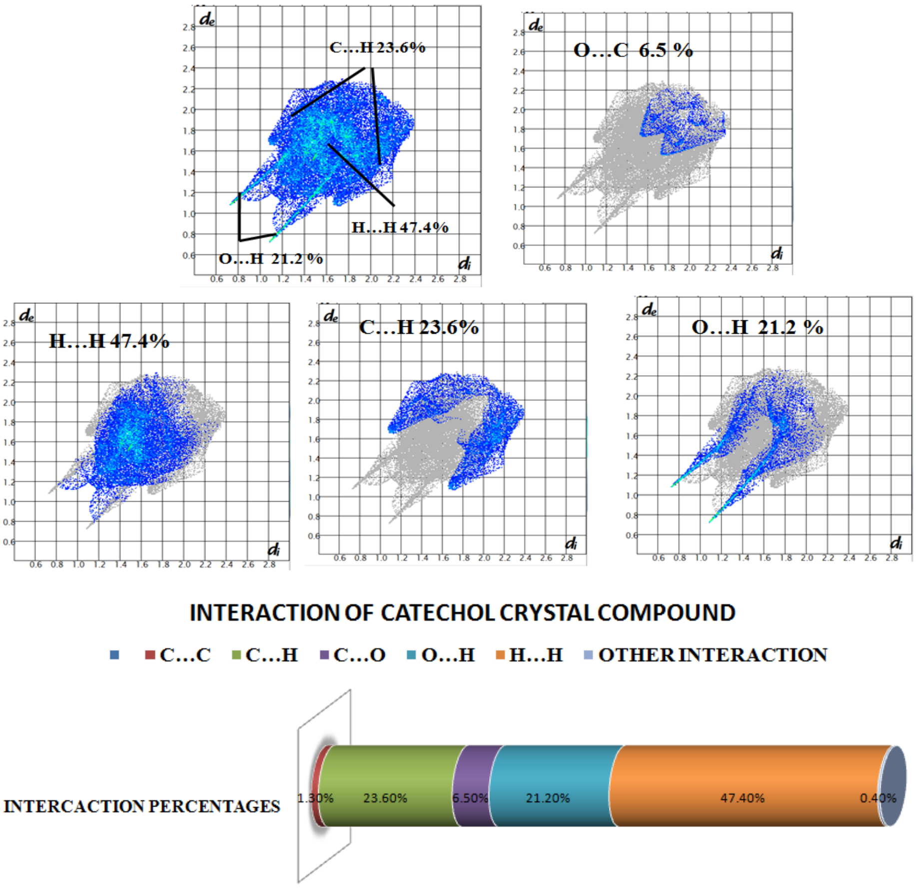
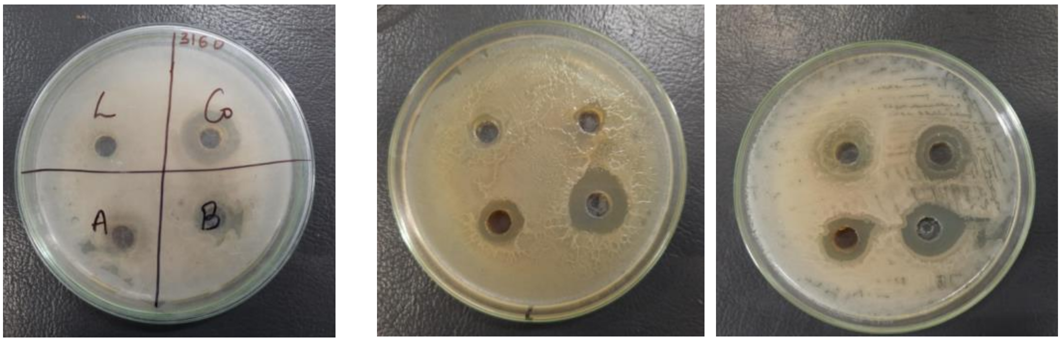


Figure 5: Diagrammatic representation of catechol crystal compounds 2D fingerprint map of distinct contributions

Antimicrobial Activity

For biological effectiveness against gram-positive staphylococcus aureus, bacillus subtills, and gram-negative E. coli species, the produced metal complexes were examined. Nutrient agar was made, autoclaved at 121 °C for 45 minutes, chilled, and then added to Petri dishes after antimicrobial activities were tested using a disc (diffusion methods). We compared the level of microbial growth suppression to that of the positive control[44-57]. To determine the activity index percent for any component, apply the equation below. The Agar Well diffusion method was utilised to test the antimicrobial activity. To evaluate the antibacterial efficacy against common pathogens such as E. Coli, S. aureus, and B. subtilis, cell free supernatant was employed. Moti Lal Nehru Engineering College Prayagraj provided the means to achieve these three cultures. (Figure 6)(Table 1)



Zone of inhibition (a) s. aureus (b) B. subtilis

Zone of inhibition (c) E. coli

Zone of inhibition (d) P. aeruginose

Figure 6: Representation of Hg (II) complexes compounds

Table 1- Antimicrobial result of schiff's base ligands synthesized compound are selective more active to words gram negative strain bacteria

Sample code	Staphylococcus aureus (3160)	Bacillus subtills (121)	p. aeruginose (1680)	E. coli (433)
L	NA	0.8 ± 2	NA	NA
Co-compounds	1.8 ± 2	0.8 ± 2	2.2 ± 2	NA
Hg_1- L (Cl)	Active	2.1± 2	2.4 ± 2	NA
Hg_2-L (Br)	Not considered	1.3± 2	1.8± 2	NA

CONCLUSION

In this study we have reported the synthesis of new schiff base derivatives and their Hg (II) complexes. The structural characterizations of synthesized compounds were made by using the IR, ^{1}H-NMR, UV and Antimicrobial activity.

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