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Studies on some Mixed Ligand Complexes with Schiff base and 2,2'- Bipyridine

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ABSTRACT

Some new mixed ligandmetal complexes of transition metals were synthesized from a schiff base which is obtained by the condensation of isoniazid and pnitrobenzaldehyde as primary ligand and 2,2'-bipyridine as a secondary ligand. The ligand and their metal complexes were studied using various spectroscopic methods namely molar conductance, magnetic susceptibility measurement, IR data and UV /vis spectroscopic techniques. The mixed ligand complexes were found to have formulae $[M(L1)(L2)]X_2$ where M = Ni (II) and Cu (II) . The resultant data expose that the complexes have a geometry demonstrative electrolytic nature. Its new compounds and their biological potency were chemically analyzed against Escherichia coli and Bacillus Cereus showing the enriched activity of complexes against the species as compared to the free ligand

Keywords : Ligandmetal, Pnitrobenzaldehyde, Spectroscopic Techniques

I. INTRODUCTION

The co-ordination chemistry of various ligandswith transition metal ions inhanced by using advanced techniques in the field of inorganic chemistry and pharmaceutical industries[1]. Schiff base played an important role as chelating ligandsin main group with transition metal ions and gives stability under suitable conditions as oxidative and reductive . Amine group in this complexes behave like marginal between hard and soft Lewis bases[2-4].

Schiff base obtained from amino and carbonyl group that co-ordinate with metal ions through azomethine nitrogen [5].(CN) linkage of this derivatives imperative role for biological activity like antifungal, antibacterial ,anticancer ,antimalarial[6-8]activities. The interesting and significant properties of schiff base complexes to reversibility bind oxygen[9] ,catalytic and photochromicproperties [10].

Mixed ligand complexes played a vital role in environment area as biological catalyst. Schiff base originated from heterocyclic compound as p-nitrobenzaldehyde and furan -2carbaldehyde .This complexes are applied in treatment and prevention for phthisis. The bypyridine ligands are widely used in formation of metal complexes regarding its strong redox stability.

Experimental

All these chemicals are purchase commercially from chemical laboratories. By using suitable apparatus measured its melting point and products characterized by likeing of spectroscopic data(UV-visible and FT-IR) and melting points with selected samples. The absorbance wavelength are determined by UV-visible.A quartz cuvette and ethyl alcohol used as a reference. its magnetic susceptibility determined by a balance at room temperature.

General Procedure for Synthesis of N-(4 nitrobenzylidene) isonicotinohydrazone Schiff base Ligand as the Primary Ligand, L1

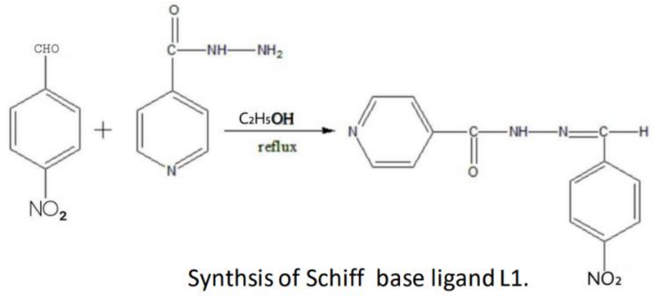
INH (1.37 g, 10.0 m mol) was mixed with absolute ethanol (15 mL) and the mixture brought to the boil, producing a slurry. Barely sufficient additional ethanol was then added to give a homogeneous solution at reflux. P-nitrobenzaldehyde(1.22 mL, 10 mmol) was added drop-wise over 5 minutes and washed with 5 mL of ethanol. The reaction mixture was refluxed for 4 hours then allowed to cool slowly and to stand overnight. Finally, it produces a white crystalline solid which was filtered off and dried.

Cloud computing and cloud storage have become hot topics in recent decades. Both are changing the way we live and greatly improving production efficiency in some areas. At present, due to limited storage resources and the requirement for convenient access, we prefer to store all types of data in cloud servers, which is also a good option for companies and organizations to avoid the overhead of deploying and maintaining equipment when data are stored locally. The cloud server provides an open and convenient storage platform for individuals and organizations, ut it also introduces security problems. For instance, a cloud system may be subjected to attacks from both malicious users and cloud providers. In these scenarios, it is important to ensure the security of the stored data in the cloud. In [1], several schemes were proposed to preserve the privacy of the outsourced data. The above schemes only considered security problems of a single data owner. However, in some applications, multiple data owners would like to securely share their data in a group manner. Therefore, a protocol that supports secure group data sharing under cloud computing is needed. A key agreement protocol is used to generate a common conference key for multiple participants to ensure the security of their later communications, and this protocol can be applied in cloud computing to support secure and efficient data sharing. Since it was introduced by Diffie-Hellman in their seminal paper [4], the key agreement protocol has become one of the fundamental cryptographic primitives. The basic version of the Diffie-Hellman protocol provides an efficient solution to the problem of creating a common secret key between two participants. In cryptography, a key agreement protocol is a protocol in which two or more parties can agree on a key in such a way that both influence the outcome. By employing the key agreement protocol, the conferees can securely send and receive messages from each other using the common conference key that they agree upon in advance. Specifically, a secure key agreement protocol ensures that the adversary cannot obtain the generated key by implementing malicious attacks, such as eavesdropping. Thus, the key agreement protocol can be widely used in interactive communication environments with high security requirements (e.g., remote board meetings, teleconferences, collaborative workspaces, radio frequency identification [5], cloud computing and so on).

The Diffie-Hellman key agreement [4] provides a way to generate keys. However, it does not provide an authentication service, which makes it vulnerable to man-in-the-middle attacks. This situation can be addressed by adding some forms of authentication mechanisms to the protocol, as proposed by Law et al. in [6]. In addition, the Diffie-Hellman key agreement can only support two participants. Subsequently, to solve the different key attacks from malicious conferees, who attempt to deliberately delay or destroy the conference, Yi proposed an identity-based fault-tolerant conference key agreement in [7]. Currently, many researches have been devoted to improving the security and communication efficiency of the key agreement protocol, which is covered in the literature [8] Note that in Chung and Bae's paper [12] and Lee et al.'s paper [13], block design is utilized in the design of an efficient load balance algorithm to maintain load balancing in a distributed system. Inspired by [12] and [13], we introduce the symmetric balanced incomplete block design (SBIBD) in designing the key agreement protocol to reduce the complexity of communication and computation. As far as we know, the work to design the key agreement protocol with respect to the SBIBD is novel and original.

Experimental procedure for synthesis of mixed ligand complexes

To the warm methanolicsolution (10 mL) of primary ligandL1 (0.255 g, 1 mmol), 10 mL warm methanolic solution (0.257 g, 1 mmol) of nitrate salt of metal Cu(II) and Ni(II) were added. After 30 minutes 5 mL warm methanolicsolution of 2,2′ - bipyridine (0.156 g, 1 mmol) was added drop-wise as a secondary ligand(L2) and the resulting mixture was refluxed for about 3-4 hours. The obtained precipitate was filtered, washed with methanol and dried under vacuum on anhydrous CaCl2



Synthsis of Schiff base ligand L1.

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Antimicrobial activity

The ligand(L1) and its mixed ligandcomplexes with (L2) were screened for in vitro antimicrobial activity in DMSO against gramnegative Escherichia coli and gram-positive Bacillus cereus strains by Kirby Bauer's disc diffusion technique. A uniform suspension of test organism of 24 hours old culture was prepared in a test tube containing the sterile saline solution. Sterile nutrient agar was then added in each of the Petri dishes. The dishes were related to ensuring the uniform mixing of the microorganism in the agar medium which was then allowed to solidify.

A Special type of filter paper discs are put in the various compound solution keep it on the labelled plates the DMSO are acts as solvent controller. A standard kanamycin compound are used for comparison . This sample put in the refrigerator for half an hour. the diffusion and incubated process maintain at 37ºC for 24 hours. A scale is used for measuring diameter of the inhabition zone around each dish and results recorded in millimeter. The experimental data of antibacterial activity of primary ligand (L1)mixed ligand complexes and graded drugs are shown in table.

Results and Discussion

By the reaction of Cu(II) and Ni(II) with ligand(L1)and 2,2′ - bipyridine(L2), complexes of the type $[M(bpy)(SB)]X_2$ were obtained. These complexes have a different colour, stable at room temperature, insoluble in common polar solvent but soluble in DMSO and DMF, do not have the sharp melting point but decompose above 523K. The measurement of molar conductivity at 10-3 M concentration carried out in DMSO at room temperature. The molar conductivity values show that the nitrate complexes were 1:2 electrolytes . The analytical and physical data (colour, melting point, molar conductivity and magnetic moment) of the complex are observed . For the Cu(II) and Ni(II) complexes the magnetic moments were 1.83 BM and 0.22 BM indicating paramagnetic and diamagnetic in nature.

Table 1 Analytical and physical properties data of L1 and its mixed ligand complexes with L2.

Symbol of Compounds	Complexes	M.P or De (Decomposition Temp) / °C	Color	Solubility DMSO & DMF	Molar conductance ohm^{-1} cm^{2}mol^{-1}	μ_{eff} in B.M
Ligand (L1)	$(C_{13}H_{10}N_4O_3)$	143	White	(+) ve	5	
[Cu(L1)(L2)] $(NO_3)_2$	$[Cu(C_{13}H_{10}N_4O_3)(C_{10}H_8N_2)](NO_3)_2$	275 (De)	Brown	(+) ve	156	1.83
[Ni(L1)(L2)] $(NO_3)_2$	$[Ni(C_{13}H_{10}O_3N_4)C_{10}H_8N_2)](NO_3)_2$	285 (De)	Yellow	(+) ve	149	Dia

Table 2 Key infrared bands (cm^{-1}) of L1 and its mixed ligand complexes with L2.

Symbol of Compound	Compound	ν (C=O)	ν (C=N)	ρ(Py) bending	ν (M-O)	ν (M- N)
Ligand (L1)	$(C_{13}H_{10}N_4O_3)$	1658.84	1598.69			
Ligand L2	$(C_{10}H_8N_2)$		1642 m	650		
$[Cu(L1)(L2)](NO_3)_2$	$[Cu(C_{13}H_{10}N_4O_3)(C_{10}H_8N_2)](NO_3)_2$	1636.35	1592.95	678.73	528.02	433.99
$[Ni(L1)(L2)](NO_3)_2$	$[Ni(C_{13}H_{,0}N_4O_3)(C_{10}H_8N_2)](NO_3)_2$	1628.82	1571.41	681.68	534.71	426.92

Table 3 UV- Visible spectrum of the Ligand L1 and its mixed ligand complexes with L2.

Symbol of Compound	Compound	λ in nm	Assignment
Ligand (L1)	$(C_{13}H_{10}N_4O_3)$	283 333	π-π* n-π*
$[Cu(L1)(L2)](NO_3)_2$	$[Cu(C_{13}H_{10}N_4O_3)(C_{10}H_8N_2)](NO_3)_2$	270 367	π -π* C.T
$[Ni(L1)(L2)](NO_3)_2$	$NiC_{13}H_{10}N_4O_3)(C_{10}H_8N_2)](NO_3)_2$	269 370	π -π* C.T

Table 4 Antibacterial screening results of Ligand L1 and its mixed ligand complexes with L2.

Compounds	Antibacterial Zone of Inhibition (in mm): Gram Negative *Escherichia coli*	Gram Positive *Bacillus cereus*
Kanamycin	32	35
Ligand (L1)	4	4
$[Cu(L1)(L2)](NO_3)_2$	20	22
$[Ni(L1)(L2)](NO_3)_2$	18	15

IR spectral studies

The IR spectrum of the primary ligand(L1) reveals characteristic bands at 1658.84.5 cm1 and 1598.69 cm-1 assigned to ν(C=O) and ν(C=N) .The band at 1658.84.5 cm-1 determinableto the ν(C=O) stretching vibration of the Schiff base ligandis shifted to another region 1628– 1637 cm-1 in the complexes of Cu(II) and Ni(II) reflecting coordination of the carbonyl oxygen to the metal ions .The presence of band at 528– 535 cm-1 in the IR spectra of complex is due to M–O stretching vibration.

The azomethine band at 1598.69 cm-1 of Schiff base was shifted to lower frequency range from 1571–1593 cm-1 in the spectra of the complexes, confirming the involving of the azomethine nitrogen atom in the coordination with the metal ion. In the IR spectra of this complex, the new bands which displayed in the 426– 434 cm-1 range are assigned to the ν(M–N) vibration . The strong sharp band observed at 1384 cm-1 in the complex can be assigned to uncoordinated nitrate ion . The band at 650 cm-1 is assigned to ν(C=N) of pyridine for secondary ligand, L2. This band is shifted to 671-682 cm-1 for mixed ligandcomplexes .All of these IR data confirm that ligandscoordinated in Cu(II) and Ni(II) metal complexes through their O and N atoms respectively.

UV- visible spectra

The UV-Visible spectra of the ligandL1 display two bands at 281 and 347 nm which are assigned to π- π* and n-π* transition respectively. The complexes reflect the charge transfer transitions which can assigned to charge transfer from the ligandto metal (LMCT) and vice -versa. The absorption bandsfor Complexes range from 367– 370 nm may be associated with L M → charge transfer and vice versa (M L →) . In the UV-region, the complexes reflect absorption band at 269–270 nm. which may be assigned to π π– * transition. All the spectral complexes reveals bands assigned to π π– * and M L → charge transfer. A square planar geometry prefer by the metals.

Antimicrobial screening result

The Schiff-base ligandL1 and its mixed ligandcomplexes with L2 noticed here were evaluated for antimicrobialactivity against Escherichia coli and Bacillus cereus. The values of zone inhibition were calculatedin mm. The antimicrobiaI data observed for ligandL1 and its mixed ligandcomplexes with L2 are tested.

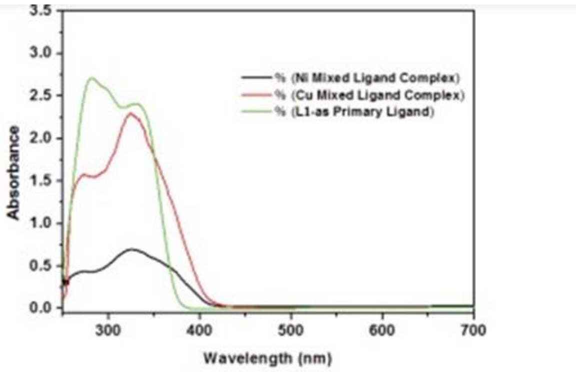

Figure 4 UV- Visible Spectrum of the L1 and its Mixed Ligand Complexes.

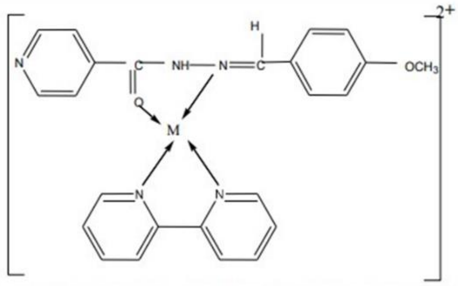

Figure 5 The proposed geometry of M- mixed ligand complexes for L1 and L2 (where, M= Cu(II) and Ni(II)).

The inhibitory zone data exhibits that the ligandL1, as well as its mixed ligand complexes with L2, reflect average antisepticaction. The anatomical area action of Schiff base ligandappear from the presence of imine group which imports in elucidating the mechanism of conversionreaction in biological arrangements.

Therefore antibacterial potency shown by this mixed ligand complexes with second ligand(L2) due to chelation of these metals with organic ligands enhancing its effect as a result. The DMSO control and not observed antimicrobial function against bacterial strains. But this processed sample found to be active.

Conclusions

The complexes as Schiff base ligand(L1) and copper (II) and Nickel(II) mixed ligand with

L2 were prepared and characterized by using different spectral and other techniques. Its IR spectral data shows Schiff base (L1) and bpy (L2) ligand coordinated with metal ions through Carbonyl Oxygen , Nitrogen of azomethine and pyridine. Similarly notice about their square planar geometry of the complexes. The various data indicating more antibacterial potency of mixed ligand complexes than Schiff base ligand(L1). Therefore, tested complexes found to be moderately active.

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