Original Article

TEMPLATE SYNTHESIS OF NI(II) MACROCYCLIC COMPLEXES: CHARACTERIZATION AND BIOLOGICAL ACTIVITIES

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Abstract

T Ni(II) complexes of Schiff's base macrocyclic ligands i.e. 2,3,9,10-tetraphenyl-1,4,8,11- tetraazacyclotetradeca-1,3,8,10-tetraene (BDP), 2,4,10,12-tetramethyl-1,5,9,13 tetraazacyclohexadeca-1,4,9,12-tetraene (ADP) and 2,3,9,10-tetramethyl-1,4,8,11 tetraazatetradeca-1,3,8,10-tetraene (DDP) have been synthesized. All these synthesized complexes have been characterized by elemental analysis, molar conductance, magnetic susceptibility measurement, IR and UV-Visible spectral studies. On the basis of elemental analysis, the complexes have been found to have the general composition Ni(Lig)X2 where Lig = BDP, ADP, DDP and X = Cl-, SO42- and NCS-. Molar conductance of complexes has been measured in DMSO solvent and the obtained values indicate non-electrolytic nature of all complexes i.e. counter ions are present inside the coordination sphere. Magnetic moments and electronic spectral data suggested six coordinated octahedral geometry for the Ni(II) complexes. The ligand field parameters have been calculated for metal complexes by using various energy level diagrams. In vitro synthesized complexes have been examined against the growth of the some plant pathogenic fungi in order to assess their microbial properties.

Keywords: Macrocyclic, Schiff base ligands, Template synthesis, Ni(II) complexes, Spectral characterization, Microbial properties

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INTRODUCTION

Schiff base ligands are preferable ligands because they are simply produced via condensation reaction from carbonyl compounds and primary amines, such as aldehydes or ketones [1-3]. German scientist and Nobel Prize recipient Hugo Schiff, first developed Schiff bases in 1864 [4]. Schiff bases are important class of chemicals because of their potential to generate complexes with transition metal ions [5-6]. Schiff base ligands are simple to prepare and also form complexes with most of the metal ions and these metal ions coordinate with nitrogen donor atom of azomethine (-C=N) functional group [7-8]. The azomethine (C=N) moiety, plays a key role in bioactivities because nitrogen donor atom of this group offers a binding site for various metal ions to be coupled with diverse biomolecules such as amino acids and proteins for antimicrobial actions in biological systems [9-11]. As a result, antibacterial, antifungal, antitumor and anticancer properties have been reported for various kind of Schiff bases and their metal complexes [12-14]. Macrocyclic ligands are multidentate in nature as have high number of donor atoms bonded to a cyclic hydrocarbon framework [15]. Macrocyclic ligands probably contain minimum three donor atoms and ligand has at least nine atoms in thering. Coordinative interactions with donor atoms hold the coordinated metal ion within the ring cavity [16]. Macrocyclic metal complexes are also prepared by following the template method in which the ligand reactants (carbonyl and primary amine) and metal salt are condensed in a molar ratio 2:2:1 for reactants and metal salt, respectively [17]. Their mode of coordination is very important for understanding the molecular processes in biochemistry, catalysis, materials science, activation, encapsulation, transport, hydrometallurgy, separation phenomena and environmental chemistry., So macrocyclic metal complexes have been received significant and growing interest in field of coordination and pharmaceutical chemistry [18]. Schiff based macrocyclic complexes are a prominent class of molecules that have received a lot of attention due th their wide applications [19]. Ligands and complexes of this type are used as sensors, catalysts, DNA binding/cleavage agents, anticancer agents and models of metalloenzyme reaction centers, dioxygen transport and photosynthesis and as a catalyst for RNA cleavage etc [20-22]. In this work, we reported the template synthesis, characterization and microbial study of Ni(II) complexes of macrocyclic Schiff base ligands (2,3,9,10-tetraphenyl-1,4,8,11-

tetraazacyclotetradeca-1,3,8,10-tetraene (BDP), 2,4,10,12-tetramethyl-1,5,9,13-tetraazacyclohexadeca-1,4,9,12-tetraene (ADP) and 2,3,9,10-tetramethyl-1,4,8,11-tetraazatetradeca-1,3,8,10-tetraene (DDP)).

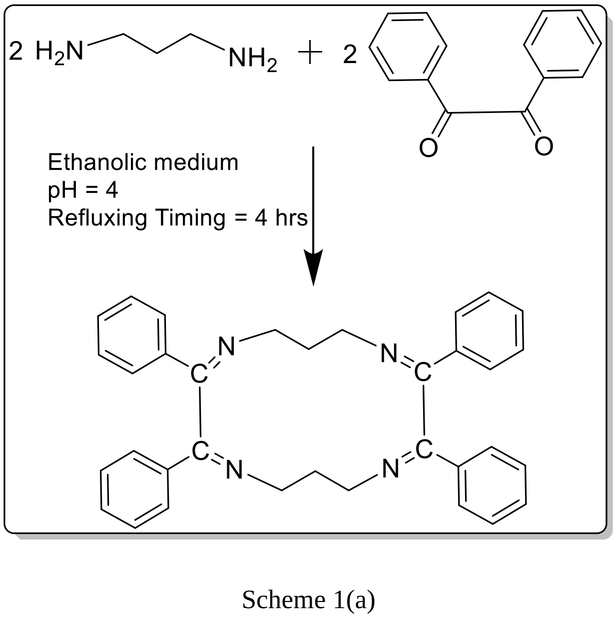
EXPERIMENTAL SECTION

All chemicals used in this research work have been bought from sigma-aldrich and of high purity, analytical (AR) grade. Different metal salts of Ni(II) have been purchased from E. Merck and used as received without following any purification process. The spectroscopic grade solvents were purchased from SRL and used without further purification. The UV-Visible spectra of Ni(II) complexes have been recorded on a Shimadzu UV mini-1240 spectrophotometer by using DMSO as solvent. The IR spectra (in KBr discs) have been done on IR BX-II spectrophotometer instrument (wave number range from 4000 to 400 cm−1). Elemental analysis data of complexes has been received by using Carlo-Erba 1106 elemental analyzer. Molar conductance value of complexes has been determined by using an ELICO (CM82T) conductivity bridge and as a solvent DMSO was used. Magnetic moment was measured at room temperature (RT) on a Gouy balance using CuSO4·5H2O as the calibrant. Microbial property (antifungal activity) of synthesized complexes has been done by following the well diffusion method.

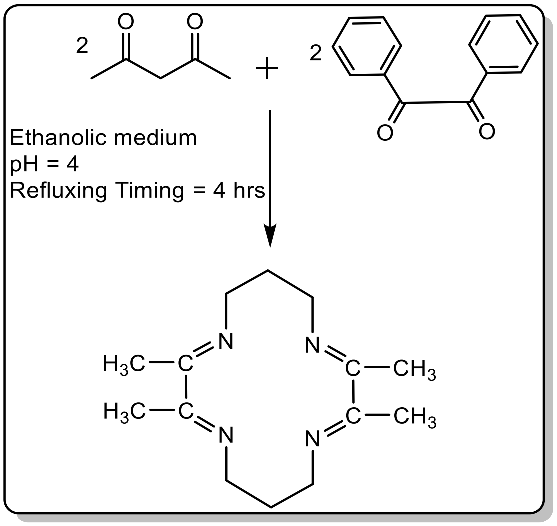
PREPARATION OF MACROCYCLIC LIGANDS

(BDP), (ADP) and (DDP)

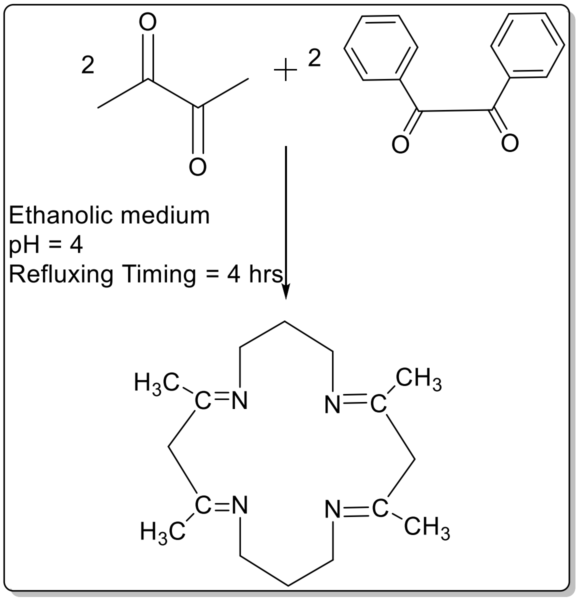
BDP was synthesized (Scheme 1(a)) by following the condensation method. In this method, an ethanolic solution of 1,3-diaminopropane (0.5 mol) and benzil (0.5 mol) was taken in a round bottom flask (RB) and 3 mL conc. HCl was also added to the round bottom flask. The reaction mixture was refluxed at 800C with continuous stirring for about 4 hrs. The resulting content has been kept overnight. The obtained product was filtered off, washed with ethanol and dried over P4O10. Melting point:1800C, colour: pale yellow. Same procedure was followed for the synthesis of ligand ADP (Scheme 1(b)) by taking ethanolic solutions of 1,3-diaminopropane (0.5 mol) and acetylacetone (0.5 mol) in the presence of 3 mL concentrated acetic acid. Prepared reaction mixture was refluxed for about 4 hrs with continuous stirring. The resulting mixture was cooled in ice bath and btained solid product was filtered off, washed with cold ethanol and dried over P4O10. Melting point: 1810C, colour: white. DDP (Scheme 1(c)) was synthesized by refluxing the ethanolic solutions of 1,3-diaminopropane (0.5 mol) and diacetyl (0.5 mol) in the same manner as described for BDP and ADP and resulting content was kept at low temperature (00C) for about 4 hrs. Obtained solid ligand was filtered off, washed and dried. Melting point: 1760C, colour: off white. Synthesis procedure of all three ligands has been presented in scheme 1.



Scheme 1(a)



Scheme 1(b)



Scheme 1(c)

Scheme 1: Synthesis of three macrocyclic ligands: (a) BDP, (b) ADP, (c) DDP

Preparation of Ni(II) Complexes With BDP, ADP and DDP Ligands

The complexes of BDP, ADP and DDP ligands were synthesized by template method. After synthesis of all the three ligands, yield found was low. With this obtained yield of ligands, it was not possible to synthesise its metal complexes. So, for the synthesis of Ni(II) complexes of these ligands, template synthesis method has been followed. In this method, all precursors i.e. corbonyl compound, primary amine and metal salts have been refluxed together. A hot ethanolic solution of benzil/acetylacetone/diacetyl (0.01 mol) was added to an ethanolic solution of 1,3- diaminopropane (0.01 mol) in 2:2 ratio and the resulting solution was refluxed for 1 hour at temperature 800C. Ethanolic solution of NiX2 was then added to the above reaction content and refluxing continued for about 20-26 hrs. After cooling the reaction product (present in round bottom flask) a variety of green coloured complexes have been separated out by following the ordinary filteration process. Obtained metal complexes have been washed with cold ethanol and also dried under vacuum over P4O10.

RESULTS AND DISCUSSION

On the basis of elemental analysis (Table 1) all the complexes have the composition Ni(Lig)X2. Molar conductance value (12.08-14.49) (Ω-1cm-1mol-1) of the complexes indicates that the complexes are non-electrolytic in nature which shows that the counter ions are coordinated to the Ni(II) metal ion. Thus on the basis of molar conductance values, complexes may be formulated as [Ni(Lig)X2]. IR spectral data shows that nitrogen donor atoms present in the macrocyclic ligand cavity are coordinated to Ni(II) metal ion and ligand behaves in a tetradentate manner. Electronic or UV-Visible spectral study suggests six coordinated octahedral geometry of all synthesized complexes. According to calculated ligand field parameters, covalent nature has been found among ligand and Ni(II) metal bonding.

Table-I Elemental Analysis and Molar Conductance Data of Ni(Ii) Complexes

Complexes	**Molecular Formula Molecular weight (g/mol)**	**Molar conductance □□$^{-1}$ cm^{-1} mol^{-1})**	**Colour (yield %)**	**Elemental analysis (%) Calculated (Found)**			
				C	**H**	**N**	**Ni**
[Ni(BDP)Cl$_2$]	$C_{36}H_{36}Cl_2N_4Ni$ (654.30)	12.08	Green (52)	66.08 (66.02)	5.55 (5.50)	8.56 (8.52)	8.97 (8.95)
[Ni(BDP)SO$_4$]	$C_{36}H_{36}N_4NiO_8S_2$ (775.52)	14.49	Mehndi (58.4)	55.75 (55.70)	4.68 (4.62)	7.22 (7.20)	7.57 (7.53)
[Ni(BDP)(NCS)$_2$]	$C_{38}H_{36}N_6NiS_2$ (699.56)	14.11	Dark green (51.4)	65.24 (65.21)	5.19 (5.16)	12.01 (12.00)	8.39 (8.34)
[Ni(ADP)Cl$_2$]	$C_{24}H_{44}Cl_2N_4Ni$ (516.23)	13.78	Green (60.02)	55.62 (55.58)	8.56 (8.52)	10.81 (10.70)	11.33 (11.31)
[Ni(ADP)SO$_4$]	$C_{24}H_{44}N_4NiO_4S_2$ (543.39)	543.39	Parrot green (55.08)	53.05 (53.00)	8.16 (8.14)	10.31 (10.27)	10.80 (10.76)
[Ni(ADP)(NCS)$_2$]	$C_{26}H_{44}N_6NiS_2$ (563.49)	12.50	Sea Green (52.12)	55.42 (55.41)	7.87(7. 84)	14.91 (14.88)	10.42 (10.42)
[Ni(DDP)Cl$_2$]	$C_{30}H_{56}Cl_2N_4Ni$ (600.32)	11.79	Dark green (55.06)	59.82 (59.80)	9.37 (9.35)	9.30 (9.26)	9.74 (9.70)
[Ni(DDP)SO$_4$]	$C_{30}H_{56}N_4NiO_4S_2$ (627.55)	13.25	Dark green (61.40)	57.42 (57.41)	8.99 (8.90)	8.93 (8.91)	9.35 (9.32)
[Ni(DDPY)(NCS)$_2$]	$C_{32}H5_6N_6NiS_2$ (647.65)	12.39	Mehndi (56.06)	59.34 (59.32)	8.72 (8.70)	12.98 (12.92)	9.06 (9.01)

C = carbon, H = hydrogen, N = nitrogen

IR Spectra

IR Spectra of all the complexes (Table 2) of BDP, ADP and DDP ligands, the absence of absorption around 3400 cm-1 shows that the free amino groups are not there (Fig. 1). No band is observed around 1700-1800 cm-1 which indicates elimination of water molecule in the ligands. The characteristic bands due to chelated ligand appear at 1500-1700 cm-1. The -C-CH3 and -CH2- groups present in the ligands are indicated by the bands at 1375 and 1425-1435 cm-1 respectively. The shifting of the vC=N band (1620 cm-1) towards the lower side (in the range of 1618-1579 cm-1) in the complexes indicates that coordination takes place through the nitrogen of the vC=N group, thus,on the basis of IR data all three ligands behave in tetradentate manner [23-24]. The free sulphate ion belongs to high symmetry point v1, and v3¬ and v4¬ appeared at positions 1137 and 618 cm-1 If the ion is coordinated to a metal, the symmetry gets lowered and splitting of the degenerate modes occur together with the appearance of new bands in the IR spectra corresponding to raman active bands in the free ion. For unidentate sulphate, the symmetry is lowered to v1¬ and v2¬ appear with medium intensity and v3¬ and v4¬ each split into three bands. The spectrum of sulphato complexes show a splitting of the v3 band in the range of 1104-1100 cm-1 and 1063-1058 cm-1 which indicates unidentate nature of sulphato group, while the spectra of nickel complexes with thiocynate group shows a band in the range of 2072-2061 cm-1, coressponding to C-N stretching and coordination of metal ion via nitrogen donor atom of thiocynato ion

[25-26]. Appearance of single sharp band in the range of 477-470 cm-1 also confirms the metal nitrogen coordination (N-Ni) in thiocynato complex [27].

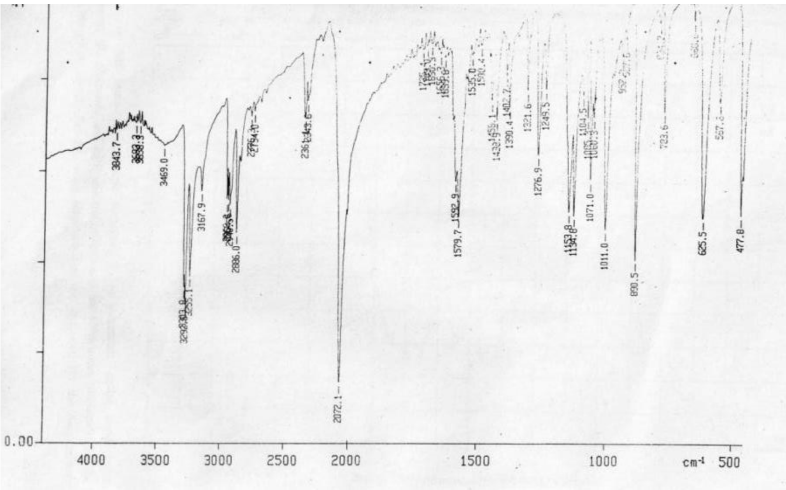


Fig. 1: IR spectrum of complex [Ni(BDP)Cl2]

Table-2: Characterstic Ir Spectral Bands of Ni(Ii) Complexes of Macrocyclic Ligands

S. No.	Compound	v(C=N) (cm^{-1})	N-Ni (cm^{-1})	Bands due to anions (cm^{-1})
1	[Ni(BDP)Cl$_2$]	1579	472	342 (supported to Ni-Cl bonding)
2	[Ni(BDP)SO$_4$]	1614	468	Splitting of v_3 into 1104-1063 (supported for unidentate nature of sulphate group)
3	[Ni(BDP)(NCS)$_2$]	1615	477	2072 (supported to coordination of Ni to N donor atom of thiocyanate group)
4	[Ni(ADP)Cl$_2$]	1618	471	345 (supported to Ni-Cl bonding)
5	[Ni(ADP)SO$_4$]	1611	468	Splitting of v_3 into 1102-1058 (supported for unidentate nature of sulphate group)
6	[Ni(ADP)(NCS)$_2$]	1603	471	2061 (supported to coordination of Ni to N donor atom of thiocyanate group)
7	[Ni(DDP)Cl$_2$]	1613	449	347 (supported to Ni-Cl bonding)
8	[Ni(DDP)SO$_4$]	1602	470	Splitting of v_3 into1100-1060 (supported for unidentate nature of sulphate group)
9	[Ni(DDPY)(NCS)$_2$]			2065 (supported to coordination of Ni to N donor atom of thiocyanate group)

Magnetic Moment

Magnetic moment of complexes has been recorded at room temperature. The value of magnetic moment was found in the range of 2.94-3.02. Theoretical values are also in good support of experimental values. The observed values along with the electronic spectral data correspond to the octahedral geometry of Ni(II) complexes of macrocyclic ligands [28].

Electronic Spectra

Nitrogen donor atoms containing macrocyclic ligand shows high-intensity absorption bands in its UV-visible spectra. These bands contribute for pi-pi* and antibonding (n)-pi* electronic transition for the imine group [29]. The UV-Vis spectral data has been illustrated in Table 3 and Fig 2. In spectra of complexes, bands are divided basically into two specific groups group I and II. Group I corresponds to the presence of ligand transitions spectral band, which appeared only in UV region. Group II spectral bands will be present in visible region which is for d-d transitions [30]. Transition bands i.e. v1, v2 and v3 appear in the range of 10065-10526, 15792-16642 and 24284-25204 respectively. These bands are responsible for the transitions 3A2g(F)-3T2g(F) v1, 3A2g(F)-3T1g(F) v2 and 2A2g(F)-3T1g(F) v3. So, examination of these transitions in the complexes' spectra recommend the six coordinated octahedral geometry for all the synthesized Ni(II) complexes of macrocyclic ligands (BDP), (ADP) and (DDP). An investigation of these bands indicates that the complexes have octahedral geometry and might possess D4h symmetry [31]. The position and intensity of transition bands which are related to charge transfer and d-d transition, depends on the position of nickel metal ion in the periodic table.

Table-3: Magnetic Moment and Electronic Spectral Bands of Ni(Ii) Complexes

S. No.	**Compound**	**μ_{eff} B.M.**	**UV spectral bands**			**Suggested structure**
			v_1(cm^{-1})	**v_2(cm^{-1})**	**v_3(cm^{-1})**	
1	[Ni(BDP)Cl_2]	2.98	10125	16541	25201	Octahedral
2	[Ni(BDP)SO_4]	2.96	10065	16065	24513	Octahedral
3	[Ni(BDP)$(NCS)_2$]	3.02	10238	15792	24284	Octahedral
4	[Ni(ADP)Cl_2]	2.94	10304	15987	24809	Octahedral
5	[Ni(ADP)SO_4]	2.97	10318	16642	24867	Octahedral
6	[Ni(ADP)$(NCS)_2$]	2.98	10515	16554	25204	Octahedral
7	[Ni(DDP)Cl_2]	2.96	10526	16257	24876	Octahedral
8	[Ni(DDP)SO_4]	2.94	10225	16246	24026	Octahedral
9	[Ni(DDPY)$(NCS)_2$]	2.99	10178	15739	25045	Octahedral

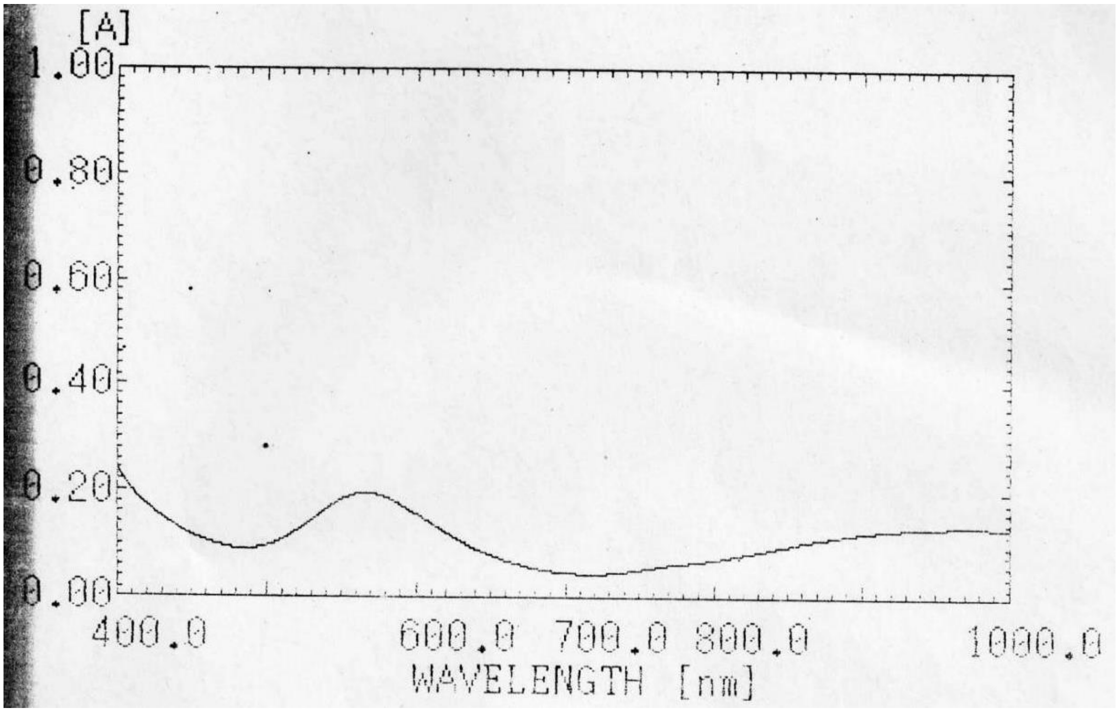


Fig.2. UV-Vis spectrum of [Ni(BDP)SO4]

Ligand Field Parameters

Various ligand field parameters Dq, B and β have been calculated and given in Table-4. Dq values have been evaluated by using Orgel energy level diagram. Nephelauxetic parameters β, has been calculated by using the equation:

$$\beta = \frac{B(\text{Complex})}{B(\text{free ion})}$$

where, B(free ion) is known as Racah parameter and its value is 1120 cm-1 for the Ni(II) metal ion. The other ligand field parameters like ligand field splitting energy Dq, Racah parameter B and ligand field stabilization energy (LFSE) have been calculated for the complexes [32]. The value of LFSE depends on the number of electrons in the d-orbitals of the metal ion, ligand field splitting energy and distribution of electrons between the t2g and eg levels. The complexes in the present study showed lower value of B than that of the free ion which indicates the orbital overlapping and delocalization of the d orbital. The value of β also is less than one, which suggested covalent nature of metal-ligand bond. In other words we can also say that the lower value of β indicates appreciable covalent character of metal-ligand bond.

Table-4: Calculated Ligand Field Parameter Data of Ni(Ii) Complexes

S. No.	**Compound**	**Ligand field parameters**			
		Dq	**B'**	**β**	**LFSE (Kjmol^{-1})**
1	[Ni(BDP)Cl$_2$]	1012	757.8	0.67	145.26
2	[Ni(BDP)(NO$_3$)$_2$]	1006	692.2	0.61	144.40
3	[Ni(BDP)(NCS)$_2$]	1023	624.1	0.55	146.84
4	[Ni(ADP)Cl$_2$]	1030	658.93	0.58	147.84
5	[Ni(ADP)SO$_4$]	1031	703.66	0.62	147.99
6	[Ni(ADP)(NCS)$_2$]	1051	680.86	0.60	150.86
7	[Ni(DDP)Cl$_2$]	1052	796.25	0.71	151.00
8	[Ni(DDP)(NO$_3$)$_2$]	1022	639.8	0.57	146.69
9	[Ni(DDPY)(NCS)$_2$]	1017	683.33	0.61	145.98

Biological Study

Well diffusion method has been employed for the examination of antifungal activity of all synthesized complexes [33]. A. niger and P. glomerata fungi species have been used to explore the inhibition zone for the tested compound at a concentration 1000 ppm. In this experimental work, Sabaround Dextrose Agar (SDA) was used as media, DMSO as control and fluconazole as standard drug. standard drug has been used to compare the activity zone of tested compounds. The antifungal screening data is presented in graphical form Fig. 4. From this figure it is resulted out that sulphate and thiocynate complexes of all ligands exhibit higher activity campared to the chloro complexes. This difference in activity may be due to the presence of extra hetero atoms (nitogen, sulphur and oxygen) [34]. Activity functioning of metal complexes can be explained on the basis of chelation theory. The chelation reduces the polarity of the metal ion mainly because of the partial sharing of its positive charge with the donor groups and possible p-electron delocalization within the whole chelation ring. The chelation ring increases the lipophilic nature of the central atom, which subsequently favours its permeation through the lipid layer of the cell memberane. The enhanced activity of the complexes can also be explained on the basis of their high solubility of the compounds, size of the metal ion and the presence of bulkier organic moieties. The mode of action may involve the formation of a hydrogen bond through the azomethine nitrogen atom with the active centers of the cell constituents, resulting in interference with the normal cell process [35]. The variation in the effectiveness of different compounds against different organisms depends either on the impermeability of the cells of the microbes or difference in ribosomes of microbial cells. It has also been proposed that concentration plays a vital role in increasing the degree of inhibition as the concentration increases, the activity increases, respectively.

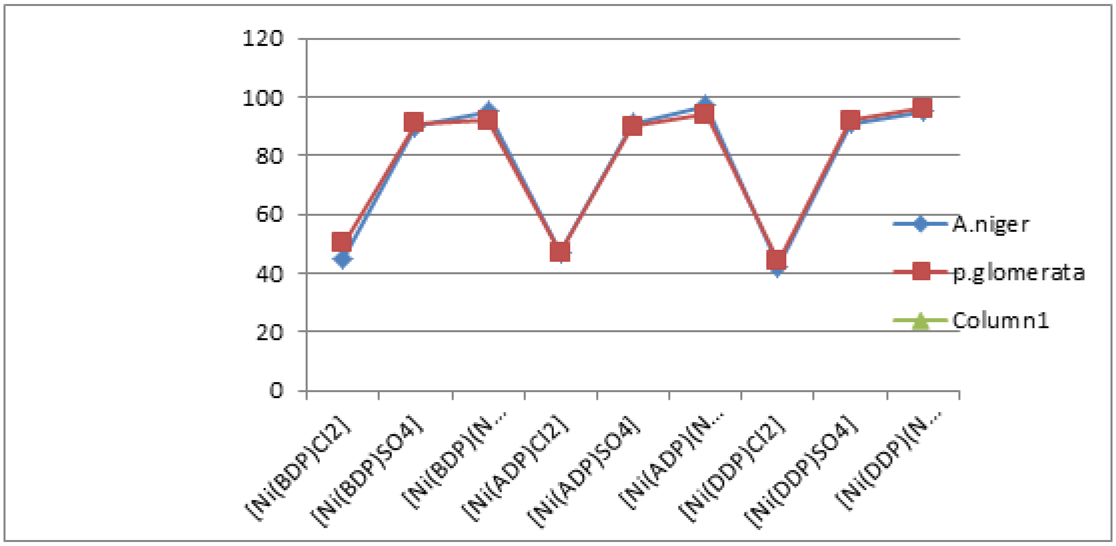
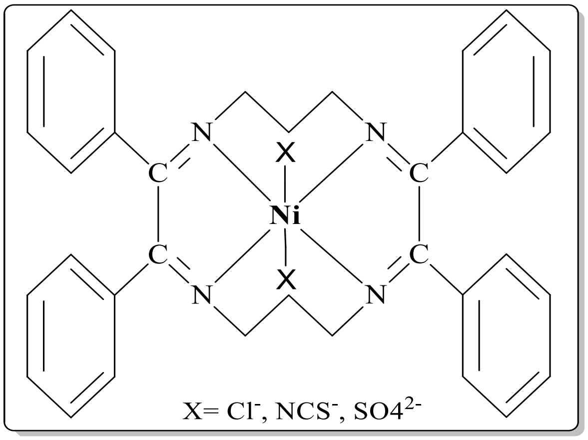


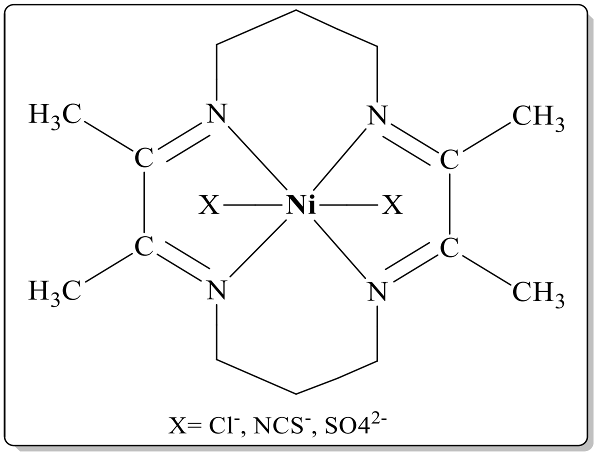
Fig. 4: Representation of antifungal data of all Ni(II) complexes in graphical line mode at concentration 1000 ppm

CONCLUSION

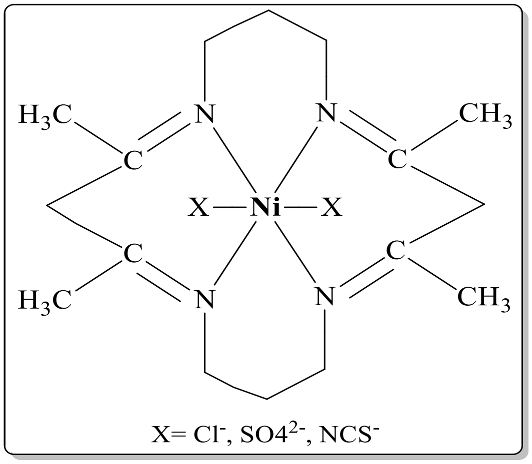
Thus, on the basis of magnetic moment, molar conductance, IR and electronic spectral studies and the subsequent discussion for the complexes given above, six coordinated octahedral geometry may be proposed for the Ni(II) complexes of all macrocyclic ligands.



(a) [Ni(BDP)X2]



(b) [Ni(ADP)X2]



(c) [Ni(DDP)X2]

Fig. 5: Proposed chemical structures of metal complexes

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