

SYNTHESIS, CHARACTERISATION AND BIOLOGICAL APPLICATIONS OF NICKEL (II) METAL COMPLEXES

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ABSTRACT

The Nickel (II) metal complexes have been prepared by treating nickel sulphate with different newly prepared ligands. The preparation of ligands with various aldehydes such as anisaldehyde, benzaldehyde, p-nitrobenzaldehyde, α -naphthaldehyde and p-methylbenzaldehyde. The present ligands and metal complexes have been characterised by elemental analysis and various physicochemical techniques such as Electronic absorption, Molar conductance, Magnetic susceptibility, Infrared spectral studies and Thermal analysis. The results of various physicochemical techniques discussed in detail and the possible structures are proposed to the reagents and complexes. The newly synthesized complexes have been screened for their antibacterial and antifungal effects against the number of gram positive and gram negative bacteria and different fungi.

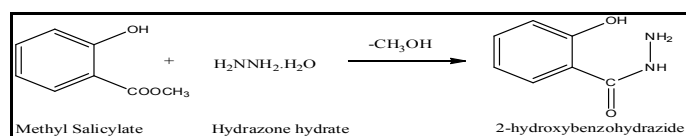
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INTRODUCTION

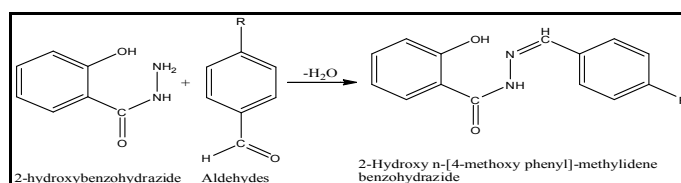
A remarkable effort has been devoted by organic chemists over the past 20 years to the Design, synthesis, characterization and application¹ of diverse chiral Schiff bases reagents are becoming increasingly important in the pharmaceutical, plastic and dye industries generally the chiral compounds has two enantiomers which elicit different properties and responses. The ligands containing donor atoms like N, O are important to prepare metal complexes which shows biological activity. The presence of functional group, usually hydroxyl, azomethine, sufficiently near the site of coordination in the hydrazone ligands, gives a five or six membered chelate ring on reaction with metal ion, which shows structural variations depending upon the type of substituent present on aromatic ring². It is well known that transition metal complexes are biologically active against pathogenic³⁻⁷ microorganisms.

Experimental

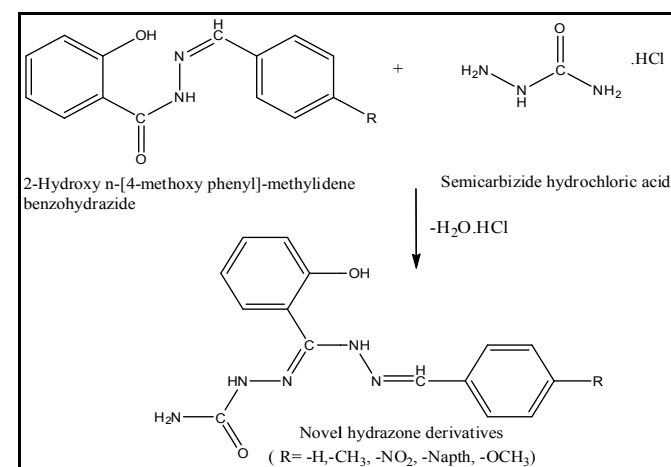
Step-I



Step-II



Step-III



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Step-IV

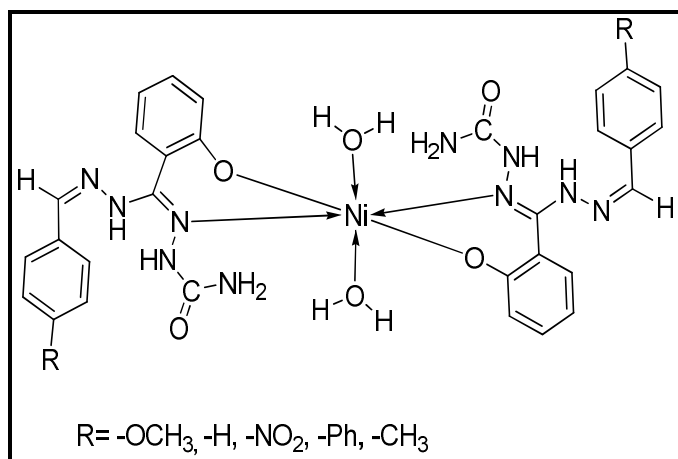


Fig 1 Proposed Structure of the Nickel Metal Complex

RESULTS AND DISCUSSION

All nickel metal complexes are brown and light green solids (Table-1), insoluble in water and shows varying solubility in common organic solvents. The nickel complexes are non-hygroscopic and thermally stable having decomposition temperature more than 250°C indicating a strong metal-ligand bonding. The value observed for molar conductance of all complexes fall between 0.0002 to 0.0008 mhos.cm²mol⁻¹ in 10⁻³ M DMSO solution indicate^{8,9} the nickel complexes are non-electrolytic in nature.

Table 1 Physical and analytical data of nickel metal complexes

Comp.	Molecular formula (Formula wt.)	Yield %	Decomposition Temperature in °C	μ_{eff} (B.M.)	Color	% Elemental analysis found (calculated)			
						Ni	C	H	N
N ₁	[C ₃₂ H ₃₂ N ₁₀ O ₆ Ni.2H ₂ O] (747.39)	75	>225	5.18	Brown	7.85 (7.84)	51.43 (51.44)	4.85 (4.86)	18.74 (18.76)
N ₂	[C ₃₀ H ₂₈ N ₁₀ O ₄ Ni.2H ₂ O] (687.34)	74	>225	4.95	Brown	8.54 (8.55)	52.42 (52.44)	4.69 (4.70)	20.38 (20.40)
N ₃	[C ₃₀ H ₂₆ N ₁₂ O ₈ Ni.2H ₂ O] (772.33)	72	>225	5.32	Light green	7.60 (7.59)	46.65 (46.67)	3.92 (3.91)	21.76 (21.77)
N ₄	[C ₃₈ H ₃₂ N ₁₀ O ₄ Ni.2H ₂ O] (787.45)	74	>225	4.98	Light green	7.43 (7.44)	57.81 (57.80)	4.85 (4.84)	17.74 (17.76)
N ₅	[C ₃₂ H ₃₂ N ₁₀ O ₄ Ni.2H ₂ O] (715.39)	71	>225	5.08	Light green	8.23 (8.24)	53.88 (53.90)	5.37 (5.36)	19.63 (19.62)

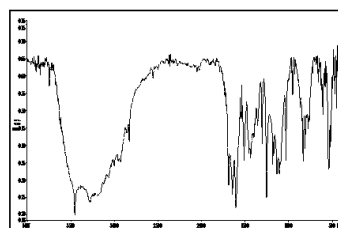
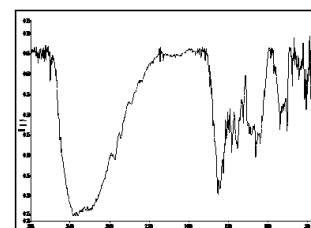
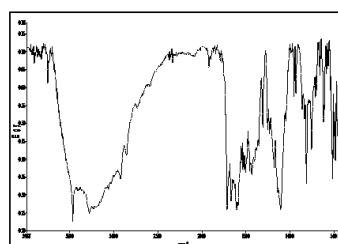
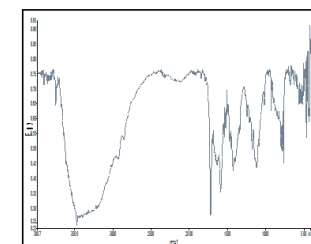
Table 2 IR Spectral Bands (cm⁻¹) of the Nickel Complexes

Inference	[C ₃₂ H ₃₂ N ₁₀ O ₆ Ni.2 H ₂ O]	[C ₃₀ H ₂₈ N ₁₀ O ₄ Ni.2 H ₂ O]	[C ₃₀ H ₂₆ N ₁₂ O ₈ Ni.2 H ₂ O]	[C ₃₈ H ₃₂ N ₁₀ O ₄ Ni.2 H ₂ O]	[C ₃₂ H ₃₂ N ₁₀ O ₄ Ni.2 H ₂ O]
v (O-H)	3496	3504	3512	3493	3494
v (C=N) azomethine	1590	1599	1587	1592	1595
v (N=C-O)	1511	1519	1516	1506	1521
v (Ni-O)	537	516	526	501	521
v (Ni-N)	416	417	419	428	433

Infrared Spectra

The IR spectrum of the free ligands show four bands at around 1607 cm⁻¹, 3196-3048 cm⁻¹, 3269 cm⁻¹, 1647 cm⁻¹ related to azomethine (-C=N), (-N-H), free (-O-H) stretching phenolic moiety and amide¹⁰ carbonyl (-C=O) stretching mode respectively. The absence of a weak broad band in 3196-3048 cm⁻¹ region, noted in the spectra of the nickel metal complexes indicates deprotonation of bonded (-NH) group during complex formation and bonded the oxygen of amide carbonyl to nickel metal ion. The v(C=N) band is shifted to lower wave number with respect to free ligand, indicating that the nitrogen of azomethine group is bonded to the nickel metal ion, which

was further confirmed by observation of the red shift in the v(N-N) stretching¹¹⁻¹³ frequency from 926 to 983 cm⁻¹ region. Bands present in the nickel metal complexes at 1607 cm⁻¹ and 1633 cm⁻¹ are related¹⁴ to the >C=N-N=C< bonding. The spectra of nickel metal complexes showed a new band at around 501-537 cm⁻¹ and 416-433 cm⁻¹ for v(Ni-O) and v(Ni-N)¹⁵ vibrations respectively.

Figure 2 IR of [C₃₂H₃₂N₁₀O₆Ni.2H₂O] complexFig 3 IR of [C₃₀H₂₈N₁₀O₄Ni.2H₂O] complexFig 4 IR of [C₃₀H₂₆N₁₂O₈Ni.2H₂O] complexFig 5 IR of [C₃₈H₃₂N₁₀O₄Ni.2H₂O] complex

Therefore oxygen and nitrogen from ligand form coordinate¹⁵ bond with central metal atom.

The presence of coordinated two water molecules is further confirmed by the band around 831-842 cm⁻¹ which is absent in the spectra. The presence of coordinated water reflected in the spectra with broad peak around 3500 cm⁻¹ is also confirmed and supported by TG/DT analysis of these complexes.

Magnetic Susceptibility Measurement

The effective magnetic moments were calculated after applying diamagnetic corrections for the ligand components using¹⁶ Pascal's constants. The room temperature μ_{eff} value for the nickel(II) complexes were found in the range 2.71-3.02 B.M., these magnetic susceptibilities of the complexes are consistent with octahedral geometry around the central Ni(II)

metal ion. The magnetic moments of the compounds investigated are in agreement with the findings of electronic absorption spectra.

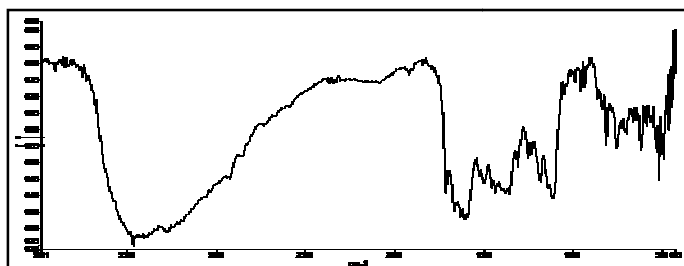


Fig 6 IR of [C₃₂H₃₂N₁₀O₄Ni.2H₂O] complex

Electronic Absorption Spectra

Electronic absorption spectral study is an important tool used¹⁷ to determine the geometry around a central metal atom. The precise interpretation of these electronic spectra can be achieved if the absorption spectra of corresponding ligands¹⁸ are very well accounted. The electronic spectra of nickel(II) metal complexes display three absorption d-d bands in the range of 11198-11494cm⁻¹, 14285-14947 cm⁻¹ and 27424-27855 cm⁻¹. The electronic spectra also exhibit a fourth high energy band around 29850-30120cm⁻¹, which can be assigned as a ligand to metal charge transfer transition.

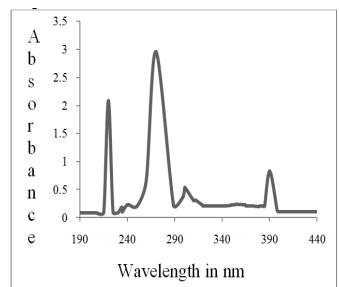


Fig 7 Ultraviolet spectrum of Ni Anisaldehyde

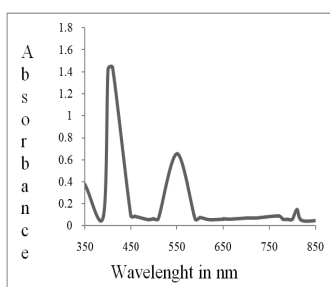


Fig 8 Visible spectrum of Ni anisaldehyde

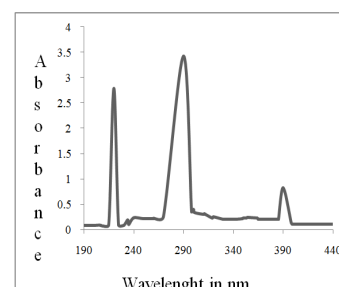


Fig 9 Ultraviolet spectrum of Ni benzaldehyde

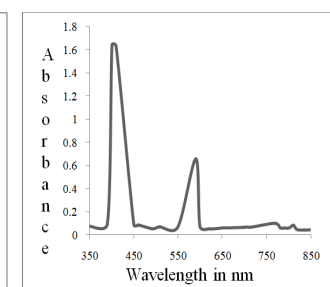


Fig 10 Visible spectrum of Ni benzaldehyde

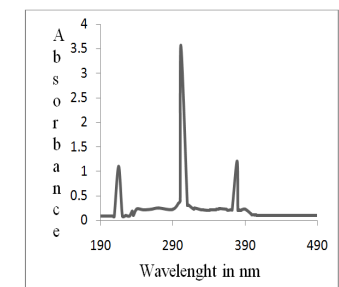


Fig 11 UV spect. of Ni Paranitrobenzaldehyde

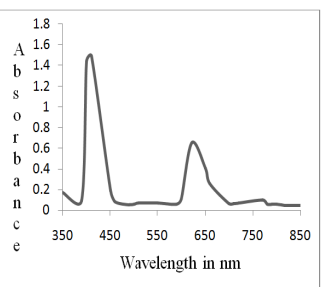


Fig 12 Visible spectrum of Ni paranitrobenzaldehyde

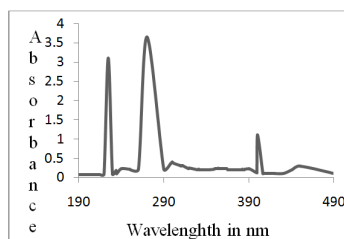


Fig 13 UV spectrum of Ni 1-naphthaldehyde

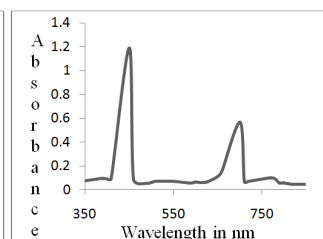


Fig 14 Visible spect. of Ni 1-naphthaldehyde

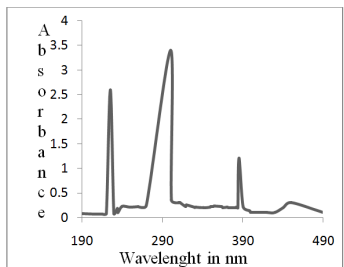


Fig 15 UV spect. of Ni 4-methylbenzaldehyde

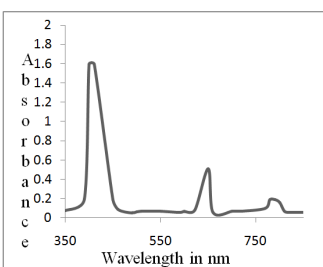


Fig 16 Vis. spect. of Ni 4-methylbenzaldehyde

Thermal measurements

In the initial step the Nickel complexes are dehydrated in the temperature range between 100°C to 180°C indicates that the complex is thermally stable at room temperature. The dehydrated product is stable up to 180°C above that temperature the second step starts; complex loses some moiety C₁₅H₁₅CHN₅ in the temperature range between 180°C to 360°C. The complex loses another moiety in the temperature range 360°C to 560°C which could be attributed due to loss of C₁₅H₁₅CHO₅N₅. Further decomposition occurs in the temperature range of 560°C to 680°C then the formation nickel oxide.

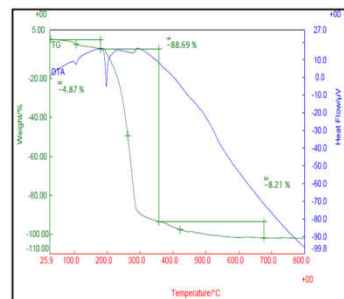


Figure 17 TG/DTA Thermogram of N₁

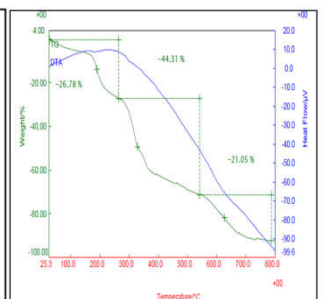


Figure 18 TG/DTA Thermogram of N₂

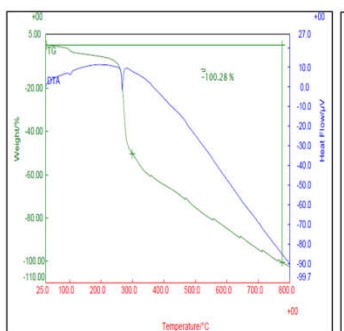


Figure 19 TG/DTA Thermogram of N₃

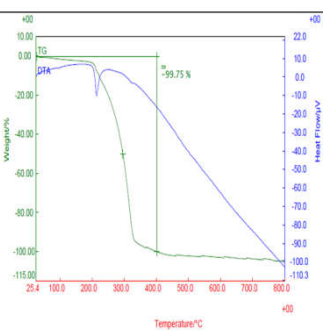
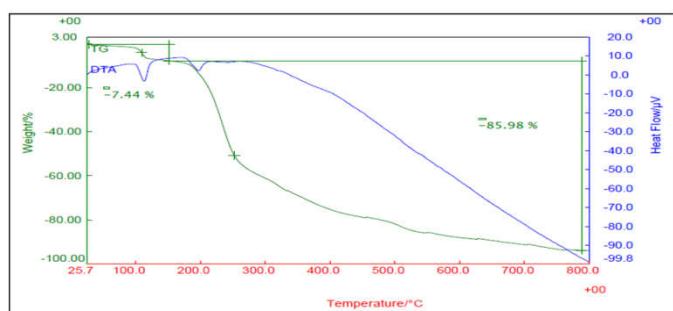


Figure 20 TG/DTA Thermogram of N₄

Fig 21 TG/DTA Thermogram of N₅

Biological Activity

Antibacterial activity done by agar well diffusion method. Muller Hinton agar and *Escherichia coli*, *Staphylococcus aureus* bacteria used for well diffusion method. The antifungal activity of the complexes against *Candida albicans* and *Aspergillus niger* pathogenic fungi was determined by Tube Dilution Method and Sabouraud broth used for antifungal activity.

Table 3 Antimicrobial Activity of the Ni(II) complexes

Complex	Antibacterial activity at 450 ppm (zone of inhibition in mm)		Antifungal activity at 350 ppm (% Inhibition)	
	<i>E. coli</i>	<i>S. aureus</i>	<i>C. albicans</i>	<i>A. niger</i>
N ₁	5	6	41	53
N ₂	5	7	40	51
N ₃	4	6	45	58
N ₄	7	4	47	59
N ₅	6	5	39	50
Na-MINAP	3	2	15	14
NiSO ₄ .7H ₂ O	3	3	31	32
Tetracycline	15	14	-	-
Amphotericin	-	-	97	98

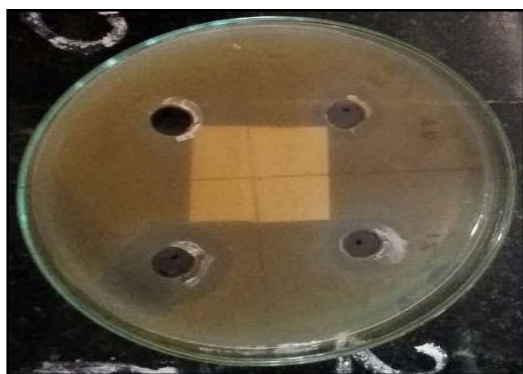


Figure 22 Antibacterial activity of Nickel complexes

CONCLUSION

Based on the above results the following conclusion may be drawn. The higher decomposition temperature and electrical conductance studies show the presence of strong metal-ligand bonding and non-electrolytic nature of the complexes, respectively. Room temperature magnetic studies are indicative of paramagnetic nature and an octahedral geometry of the Ni(II) complexes which is supported by the electronic spectra.

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The IR spectra show bonding of the metal through N- and O-donor atoms of the two ligands. Thermal analysis of endothermic peak confirms the presence of coordinated water molecules. The MIC complexes at which the culture does not show bacterial and fungal growth were found to be 450ppm and 350ppm respectively.

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