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SYTHESIS, CHARACTERIZATION AND ANTIBACTERIAL PROPERTIES OF NICKEL (II) SCHIFF BASE COMPLEX DERIVED FROM BENZOIN AND O-AMINO BENZOIC ACID

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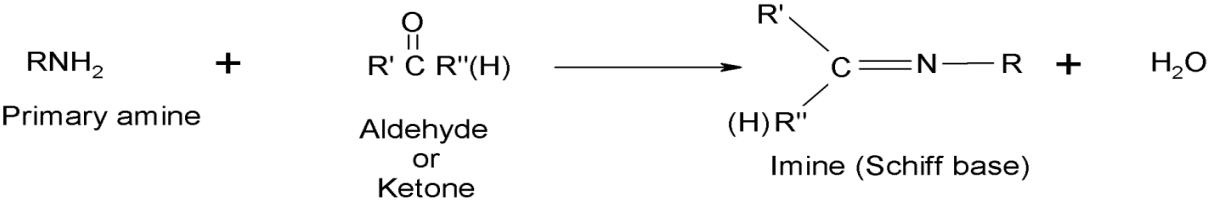
ABSTRACT
The complex of nickel (II) with Schiff base derived from benzoin and o-amino benzoic acid have been prepared and characterized by Infrared spectral analysis, conductance measurement, UV-visible spectral studies and elemental analysis. The melting point of the schiff base determined is 120°C. The decomposition temperature of nickel (II) complex is 155°C, while the molar conductance value is 10.7 ohm^{-1}cm^{2}mol^{-1}. Antimicrobial screening of the compounds were carried out in-vitro against Escherichia coli, Salmonella Typhi (Gram negative) and Staphylococcus aureus, Streptococcus spp. (Gram positive). The bioassay reveals a considerable activity of the Schiff base complex against the bacterial isolates.
Keywords: Schiff base, benzoin,, ligand, complex, bioassay.

INTRODUCTION

A Schiff base is nitrogen analog of an aldehyde or ketone in which the C=O group is replaced by C=N-R group. It is usually formed by condensation of an aldehyde or ketone with a primary amine,



where R may be an alkyl or an aryl group. Schiff base that contain aryl substituent are substantially more stable and more readily synthesized, while those which contain alkyl substituent are relatively unstable (Campbell *et al*., 1944).When an aldehyde or a ketone is condensed with primary amine, a Schiff base is produced, which is a compound containing azomethine group, *R – C = N –* (Holm *et al*., 1966; Hobday and Smith, 1972).

It has been known that a variety of metal ions on interaction with schiff bases yield chelates, for example; Holm *et al*., (1966) reported the synthesis and magnetic studies on schiff base complexes of copper (II). Transition metal Schiff base complexes have been found to play a vital role in medicine, biological systems and industries. The field of medicine has witnessed an increase in the number of complexes with therapeutic value, for example, cobalt (III) Schiff base complexes are potential antiviral agents, cis-dichlorodiamineplatinum (II) is an anti-cancer agent and copper (II) Schiff base complex is an anti tubercular agent (Lippard, 1994; Bleomink and Reedi, 1996). Preparation, physical characterization and antibacterial activity of Ni (II) Sciffbase complex was reported by Morad *et al* (2007).

Nair *et al* (2006), have studied the synthesis and antibacterial activity of some Schiff base complexes. The Schiff bases showed greater activity than their metal complexes.

Elzahany *et al (2008),* have synthesized some transition metal complexes with Schiff bases derived from 2-formylindole, salicylaldehyde and N-amino Rhodanine. The free ligands and their metal complexes were also screened for antimicrobial activities against *Bacillus cerens, Escherichia coli, Pseudomonas aeruginosa, Staphylococcus aureus and Candida albicans*. The results indicated that the ligands do not have any activity, where as their complexes showed more activity against the same organisms under identical experimental conditions..

The complex compound of a Schiff base derived from benzoin and o-amino benzoic acid with nickel (II) salt has been synthesised but not well characterized (Mahaptra *et al*., 1977) and the antimicrobial properties has not been reported. This paper reports the preparation, full characterization and antimicrobial properties of the nickel (II) Schiff base complex.

MATERIALS AND METHODS

All chemicals used were of analytical reagent grade (AR) and of the highest purity. All weighings were carried out on electrical meter balance model AB54. Melting point, decomposition temperature and coordinated water were determined on Gallenkamp melting point apparatus. IR spectral measurements were recorded using Fourier Transformed IR Genesis series in Nujol in the region of 4000-400cm^{-1}.

Electrical conductivity measurements were carried out using conductivity meter 4010. UV-visible spectral measurements were conducted on a pye Unicam UV-visible spectrophotometer. The antimicrobial activity was carried out using disc diffusion method.

Preparation of Benzoin

Into a 500cm^3 round bottom flask was added 65cm^3 of rectified spirit, 47.5cm^3 of benzaldehyde and a solution of 5g of Potassium cyanide in 50cm^3 of water. A condenser was then attached and the mixture refluxed on a steam bath for half an hour. The flask and its contents were cooled in an ice-bath to precipitate pale yellow crystals, which were suction filtered, washed with cold water, dried and then recrystallized from 40cm^3 of hot rectified spirit (Mahaptra *et al.,* 1977).

Preparation of the Schiff base Ligand

Into an ethanolic solution of the prepared benzoin (0.01mol) and 2-amino benzoic acid (0.01mol) in a 500cm^3 round bottom flask, 3.0g of anhydrous sodium acetate was added. The mixture was refluxed for an hour on a steam bath. To precipitate the grayish white product, the content was poured into an ice-cold water which was separated and recrystallized from rectified spirit, suction filtered, washed with water, and dried in a vacuum dessiccator (Mahaptra *et al.,* 1977).

Preparation of the Metal Complex

Ethanolic solutions of nickel II chloride (0.025mol) and schiff base (0.05mol) were mixed and the resulting mixture followed by drop wise addition of ammonia until the metal chelate separated, which were then suction filtered, washed with ethanol and ether and dried in vacuum dessicator. The crystals were recrystallized from rectified sprit and dried (Mahaptra *et al.,* 1977).

ANTI-BACTERIAL ASSAY

Preparation of sensitivity disc

Paper discs were made from Whatman No.1 filter paper using a paper puncher.and 50 discs each were placed in three screw-capped bottles and sterilized, by autoclaved at 121^{O}C for about 15minutes as demonstrated by Yusha'u, (2010) The bottles were then removed and allowed to cool at room temperature.

Preparation of Sub culture

2g of nutrient agar was dissolved in 60cm^3 of distilled water and then autoclaved at 121^{O}C for about 15minutes. It was removed and allowed to cool to room temperature. The media was poured into plates (petridishes). allowed to cool and solidify. The plates were inoculated singly with the organisms which are *E. coli* sp, *Salmonella* sp and *Staphylococcus aureus,* S*treptococcus* sp. Incubation was carried out at 37^{O}C for 24 hours as demonstrated by Yusha'u (2010).

Preparation of solution/serial dilution

The stock solution was prepared by dissolving 0.002g of the complex in 2cm^3 of DMSO to obtain concentration of 1000µg/cm^3. Two different concentrations were prepared from the stock solution 500µg/cm^3 and 250µg/cm^3. These were obtained by mixing stock solution (0.5cm^3) with 0.5cm^3 DMSO that is 0.5cm^3 DMSO was subsequently added to the stock after removal of 0.5cm^3. The solutions were introduced singly into each bottle containing 50 discs and allowed to stay for some time at room temperature to ensure maximum absorption of solution by the discs.

Inoculums' standardization and bioassay

The standardized inoculi of the bacterial isolates were swabbed onto the surface of nutrient agar in separate petri dishes. This was followed by placing the prepared discs of the complex and standard Ciprofloxacin (CPX) discs onto the surface of inoculated media. The plates were incubated at 37^{O}C for 18 to 24 hours after which zones of growth inhibition of each sample as observed as demonstrated by Yusha'u (2010).

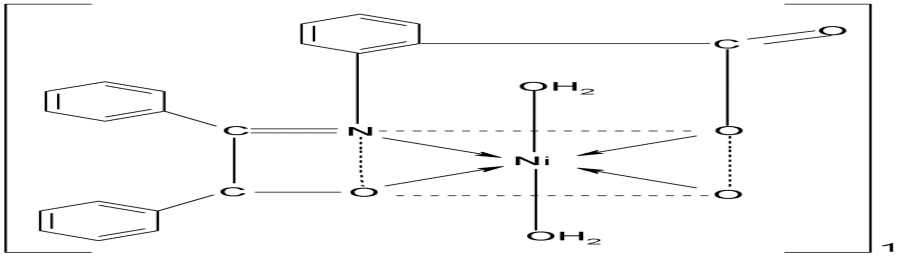
RESULTS AND DISCUSSION

The benzoin and Schiff base have been prepared as reported (Mahaptra, *et al*., 1977; Vogel, 1978), they are pale yellow and grayish white, respectively. The nickel (II) Schiff base complex have been prepared using the adopted synthetic procedure reported by Mahaptra, *et al*., (1977), the compound is pale green. The melting point temperatures of the benzoin and the schiff base are 137^{o}C and 120^{O}C, respectively, which are in agreement with the literature values (Vogel, 1978; Mahaptra, *et al*., 1977). The decomposition temperature of nickel (II) Schiff base complex is 155^{o}C, which present fairly stable complex compound. The percentage by weight (Table 1) of nickel in the complex is 12.90, and these revealed 1:1 metal to ligand ratio. The coordinated water analysis (Table 2) of the complex indicated three water molecules per molecular formula, which is in complete agreement with the report on cobalt (II) Schiff base complex (Mahaptra *et al*., 1977). The complex is insoluble in water and common organic solvents, but is readily soluble in dimethylsulphoxide (DMSO). The molar conductance value (Table 3) of nickel (II) Schiff base complex in 1 x 10^{-3}M DMSO solution was 10.7 ohm^{-1} cm^2 mol^{-1}, suggesting its non-ionic nature (Geary, 1971). The IR spectrum (Table 4) of the free schiff base shows bands of medium intensity at 1210, 1590, and 1670cm^{-1} which are for v(C-O), v(C=N) and v(C=O), respectively (Silverstein and Bassler 1967; Freedman, 1961; Nakamato, 1963). These three bands confirmed coordination of the Schiff base to the metal ion. The bands in the regions 511-558cm^{-1} and 470-478cm^{-1} are assigned to v(M—N) and v(M—O) stretching vibrations, respectively (Mahaptra *et al*., 1977), confirming coordination of the Schiff base to the metal ion.

The broad bands in the region 3350-3355cm^{-1} observed in the complex have been established to be hydrated due to the broad bands in the region 3350—3355cm^{-1} (Patel and Agwara, 1990).

The coordinated water analysis of the complex indicated three water molecules per molecular formula, which is in complete agreement with the report on cobalt (II) Schiff base complex (Mahaptra *et al*., 1977).

The spectrophotometric analysis (Table 4) revealed 1:1 (metal-ligand) stoichiometry in the complex compound, which is common feature in the first row transition metals (Holm and O'Connon, 1971; Mahaptra *et al*., 1977). Therefore, from the analyses carried out in this work, the proposed general molecular structure of nickel (II) Schiff base complex is as presented below;



NICKEL (II) SCHIFF BASE COMPLEX

The biocidal assay carried out on the ligand and the metal complex yields a rather considerable activity against the isolates used. The ligand showed no activity against Gram negative isolates (Table 5) buts was active against one of the gram positive isolate i.e. *Staphylococcus aureus*.., having a diameter of 11mm and 10mm for 1000µg/cm^3 and 500µg/cm^3 respectively (Table 6). Table 7 shows the sensitivity of the metal complex on *E.coli sp*. and *Salmonella* sp. (gram negative). There was a positive result for both isolates at 1000µg/cm^3 having a diameter of 12mm and 8mm respectively, but only *E.coli* sp. showed at 500µg/cm^3 and both did not show any activity at 250µg/cm^3. *Staphylococcus* showed sensitivity to the metal complex at all the concentrations having diameters of 12mm, 10mm and 8mm for 1000µg/cm^3, 500µg/cm^3, and 250µg/cm^3 respectively, whereas, *Streptococcus* showed no sensitivity. Ciprofloxacin and Gentamycin are control disc for Gram negative and Gram positive respectively.

Table 1: Percentage Composition by Weight of Metal in the Complex

.	Compound	%Metal Calculated	%Metal Observed
	$[NiL(H_2O)_3]$	13.23	12.90

Table 2: Percentage Composition by Weight of Water in the Complex

Compound	%Water Calculated	%Water Observed
$[NiL(H_2O)_3]$	12.17	12.12

Table 3: Conductivity Measurement of the Complex in DMSO (1 x 10^{-3}M)

Compound	Specific Conductance (k)	Molar Conductance (Ohm^{-1}cm^{2}mol^{-1})
$[NiL(H_2O)_3]$	$9.3x10^{-6}$	10.70

Table 4: Infrared Spectral Data of the Schiff Base (L) and the Complex

S/No.	Compound	v(C=N) (cm^{-1})	v(C-O) (cm^{-1})	v(O-H) (cm^{-1})	v(M-N) (cm^{-1})	v(M-O) (cm^{-1})
1	Schiff Base	1590	1210	-	-	-
2	$[NiL(H_2O)_3]$	1596	1279	3355	558	467

Table 5: Screening of Bacterial isolate (Gram negative)s on the liigand

Isolates/ Test organism	1000µg/cm^3 (mm)	500µg/cm^3 (mm)	250µg/cm^3 (mm)	CPX (mm)
E- coli sp.	_ _ _	_ _ _	_ _ _	40
Salmonella sp.	_ _ _	_ _ _	_ _ _	_ _ _

Table 6: Screening of Bacterial isolate (Gram positive)s on the liigand

Isolates/Test organism	1000µg/cm^3 (mm)	500µg/cm^3 (mm)	250µg/cm^3 (mm)	GENTAMYCIN (mm)
Staphylococcus	11	10	_ _ _	15
Streptococcus	_ _ _	_ _ _	_ _ _	_ _ _

Table 7: Screening of Bacterial isolate (Gram negative)s on the complex

Isolates/Test organism	1000µg/cm^3 (mm)	500µg/cm^3 (mm)	250µg/cm^3 (mm)	CPX (mm)
E- coli sp.	12	7	_ _ _	45
Salmonella sp.	8	_ _ _	_ _ _	_ _ _

Table 8: Screening of Bacterial isolate (Gram positive)s on the complex

Isolates/Test organism	1000µg/cm^3 (mm)	500µg/cm^3 (mm)	250µg/cm^3 (mm)	GENTAMYCIN (mm)
Staphylococcus	12	10	8	28
Streptococcus	_ _ _	_ _ _	_ _ _	_ _ _

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