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## Synthesis, Characterization and Nematicidal Activity of Bis-(2-Hydroxy-1-Naphthaldehyde)-O-Phenylenediamine and its $\text{Cu}^{2+}$ , $\text{Ni}^{2+}$ and $\text{Zn}^{2+}$ Ions

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Received: 24/08/2021 Accepted: 22/09/2021 Published online: 26/09/2021

### Abstract

Schiff base complexes derived from 2-hydroxy-1-naphthaldehyde and o-phenylenediamine and its  $\text{Ni}^{2+}$ ,  $\text{Cu}^{2+}$  and  $\text{Zn}^{2+}$  complexes have been synthesized. The ligand Bis(2-hydroxy-1-naphthaldehyde)-o-phenylenediamine (BHNOPDA) and its complexes were characterized by molar conductivity, magnetic susceptibility, melting point, solubility test and spectrophotometrically (IR and UV-Vis). The solubility results indicated that the ligand and metal complexes were soluble in methanol, ethanol acetone, diethylether, chloroform, dimethylsulphoxide, dimethylformaldehyde while the melting point of ligand and complexes was found to be between 154-268°C, indicating fairly stable compounds. The complexes had molar conductivity of 46.3-54.10 indicating an electrolytic property. The IR spectrum of BHNOPDA showed characteristic vibrations of C=N and OH groups at 1610.09 and 3471.91  $\text{cm}^{-1}$ , respectively. The spectra of  $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$  and  $\text{Zn}^{2+}$  complexes, the azomethine band  $\nu(\text{C}=\text{N})$  was shifted to lower wave number (1580.21–1606.47  $\text{cm}^{-1}$ ) whereas, the  $\nu(\text{OH})$  is shifted to lower frequency upon complex formation suggesting involvement of C=N and OH groups in coordination. UV-Vis spectrophotometer showed a complexation of the metal and ligand, demonstrating a tetrahedral geometry for both the ligand and the complexes. The magnetic susceptibility values indicated that  $\text{Cu}^{2+}$  and  $\text{Ni}^{2+}$  complexes are paramagnetic, while  $\text{Zn}^{2+}$  complex is diamagnetic. The ligand and its metal complexes were screened for nematicidal activities against a root knot nematode. Hatching and mortality test of the ligand and its metal complexes against *Meloidogyne incognita* showed an enhanced activity of the metal complexes as compared to the free ligand.

**Keywords:** Schiff base ligand, Complexes, Infra-red Nematicidal activity, *Meloidogyne incognita*

### Introduction

Nematicidal agents are chemicals used against nematodes with an intention of eradicating them as they either sacrifice the nematodes or by inhibiting their growth. Plant parasitic nematodes in the last few decades have been recognized as important limiting factors for crop production, particularly, in tropic and sub-Saharan regions. Several species of plant parasitic nematodes have been reported to serious losses in this high value crops. However non-specific of the symptoms often leads to nematodes problems being diagnosed as due to nutritional or soil factors [1]. This has led to a continuous search for new chemicals to achieve the effective control of nematodes [2]. Metals particularly the d-block metals and their complexes are not left out of this important quest because in their stable oxidation states, they form stable complexes that could be potential nematicidal agents [2].

The aspects of d-block metals have ability to form coordination compounds. Such compounds are formed between a metal ion and a molecule with one or more unshared electron pairs called a ligand or a Schiff base ligand [3]. A Schiff base is a 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. Synthesis of complexes of Schiff base having novel structural features and unusual physico-chemical properties have considerable importance in biological process and constitute an active area of research in modern coordination chemistry [4,5]. Studies in bioinorganic chemistry [1] have revealed that transition metals have an important place in medicinal chemistry. These metal complexes have been studied and known to show pharmacological properties such as antibiotics, antimicrobial activities, anti-inflammatory, ulcerogenic and treatment of neurological disorders [6, 7]. Studies [9,10] have shown that, the metal complexes have a higher biological activities compared to the free ligands because of chelation. This is because chelation enhances the movement of these complexes across the membrane. This increase movement is because chelation tends to reduce the polarity of metals [11, 12]. Metal complexes of Schiff base derived from sulfane thiadazole and salicylaldehyde exhibit increased insecticidal activities and hence destroy a large number of insects [13]. This work is aimed at the synthesis,

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characterization and nematocidal activity of the Schiff base ligand Bis(2-hydroxy-1-naphthaldehyde)-o-phenylenediamine (BHNOPDA), and complexes of some transition metals as follows:  $\text{Ni}^{2+}$ ,  $\text{Cu}^{2+}$  and  $\text{Zn}^{2+}$ . This is in promotion of the sustained interest in the use of the transition metal complexes in the effective control of nematodes world over.

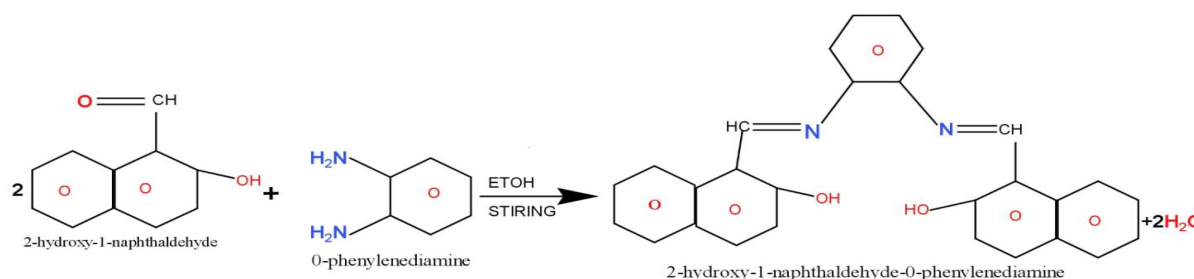
### Materials and methods

All reagents used are of analytical grade and were used without further purification. Melting/decomposition temperature was determined by Melting point SMP10 apparatus. Microwave Oven was used for the synthesis of the ligand and the metal complexes, IR spectra measurements were recorded using carry 630 FTIR spectrophotometer. Molar conductivity measurements was carried out using conductivity series 510 conductivity meters/pH meter, UV-Visible spectral measurements was done using shimadzu 1800UV-Visible Spectrophotometer, Magnetic

susceptibility measurements was determined by the Gouy balance, DHG-9053A dry Oven was used for drying of the synthesized compounds.

### Synthesis of Bis (2-hydroxy-1-naphthaldehyde)-o-phenylenediamine

The Schiff base Bis(2-hydroxy-1-naphthaldehyde)-o-phenylenediamine (BHNOPDA) was synthesized as reported by Rajmane [13] and as represented in scheme 1. Exactly 6.04g (2mmol) of 2-hydroxy-1-naphthaldehyde was mixed with 3.50g (1mmol) of o-phenylenediamine in 2:1 molar ratio in 25 mL ethanol with continues stirring until complete solubility. The mixture was then exposed to microwave irradiation at 120W power for 15 minutes. The solution was then concentrated under reduced pressure, which on cooling gave crystalline precipitates. The crystal were washed with ethanol and recrystallized in the same solvent, and dried in an oven at 45°C.



**Scheme 1: Synthesis of Bis(2-hydroxy-1-naphthaldehyde)-o-phenylenediamine**

### Synthesis of metal complexes

The metal complexes were synthesized using method adopted by Reiss *et al.* [14]. Exactly 2.0 g of Schiff base was mixed with 25 mL methanol containing 2.0 g of metal salt with continues stirring until complete solubility. The pH of the mixture was adjusted to between 7-8 by adding 1.0 M NaOH solution and then placed in the Micro-wave oven for 15 minutes for the complexation reaction to complete. The precipitated solution was removed and dried in an oven at 45°C, the crystals were collected and the yield was obtained and weighed.

### Nematicidal activity

The ligand and its metal complexes were tested for nematocidal activity against *Meloidogyne incognita* (root knot nematode) obtained from heavily infected tomato (*Lycopersicon esculentum*) which were collected from Baga farm in Pankshin Local Government Area of Plateau State. Egg masses were separated from the heavily infected tomato (*Lycopersicon esculentum*) plants and washed under running water. After cutting the roots into

smaller pieces, 1% of sodium hypochlorite was added, shaken and sieved to obtain mass of the eggs of the nematodes [15]. The compounds were tested for hatching and mortality of root knot nematode at three different concentrations alongside with control.

### Hatching test of nematodes

Exactly 20 egg masses were picked and placed in 100.0 mL of 2.5, 5, 10 ppm concentrations of the ligand and the metal complexes, alongside with control (distilled water) to monitor the hatching within 10 days. A regular time interval of 2, 4, 6, 8 and 10 days was employed in monitoring the hatched larvae of the nematodes, which the number of unhatched nematodes was observed and recorded within the time exposure. These were observed under an inverted binocular microscope in accordance with the method described elsewhere [3, 4, 9, 16].



### Mortality test of nematodes

Exactly eight (8) freshly hatched juveniles (J2) were taken into different concentrations of the ligand and its metal complexes. The number of unsacrificed nematodes was observed at the time interval of 2, 4 and 6 h respectively. For each treatment, a control was also formed in distilled water. After six (6) h of exposure, revived juveniles were counted and sacrificed nematodes were confirmed. Nematodes were considered sacrificed when they could not move.

## Results and Discussion

### Physical Properties of the Ligand and its metal Complexes

The reaction between 2-hydroxy-1-naphthaldehyde-o-phenylenediamine (BHNOPDA) and  $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$  and  $\text{Zn}^{2+}$  ions produce metal complexes which are crystalline and coloured, with high percentage yield, which ranged from 83.2-95.6 percent (Table 1). The molar conductivity of the complexes was found to be in the range of 46.30-54.10  $\text{Ohm}^{-1} \text{cm}^2 \text{mol}^{-1}$ , showing electrolytic nature of the complexes (Reisset *al.* 2017) (Table 2). The complexes were found to melt in the temperature range of 154.6-268.0  $^{\circ}\text{C}$  showing a fairly stable complex compounds. The complex compounds are insoluble in water but soluble in organic solvents (Table 3).

**Table 1: Some Physical Properties of the Schiff Base Ligand and its Metal Complexes**

Ligand/Complexes	Colour	Melting point ( $^{\circ}\text{C}$ )	%Yield	pH
BHNOPDA	Yellow	159.2 – 161.6	92.12	7.65
CuBHNOPDA	Pale green	154.6 – 158.1	91.01	7.15
NiBHNOPDA	Dark brown	210.2 – 45.7	83.2	7.80
ZnBHNOPDA	Redish – brown	264.0 – 268.0	95.6	7.45

**Table 2: Molar Conductivity Data of the Schiff base Ligand and its Metal Complexes in DMSO**

Ligand/Complexes	Concentration ( $\text{Moldm}^{-3}$ )	Specific Conductance ( $\text{ohm}^{-1} \text{cm}^{-1}$ )	Molar conductance ( $\text{ohm}^{-1} \text{cm}^2 \text{mol}^{-1}$ )	Remark
BHNOPDA	$1.0 \times 10^{-3}$	$26.40 \times 10^{-6}$	26.40	Non-electrolytic
CuBHNOPDA	$1.0 \times 10^{-3}$	$54.10 \times 10^{-6}$	54.10	electrolytic
NiBHNOPDA	$1.0 \times 10^{-3}$	$50.50 \times 10^{-6}$	50.50	electrolytic
ZnBHNOPDA	$1.0 \times 10^{-3}$	$46.30 \times 10^{-6}$	46.30	electrolytic

**Table 3: Solubility Data for the Schiff Base Ligand and its metal complexes in different solvents at room temperature**

Ligand/Complexes	Distilled Water	Methanol	Ethanol	Acetone	Ether	Chloroform	DMSO	DMF
BHNOPDA	NS	CS	CS	CS	CS	CS	CS	CS
CuBHNOPDA	NS	CS	CS	CS	CS	CS	CS	CS
NiBHNOPDA	NS	SS	SS	SS	SS	SS	CS	SS
ZnBHNOPDA	NS	SS	SS	SS	SS	SS	CS	SS

NS – Not Soluble SS – Slightly Soluble CS – Completely Soluble

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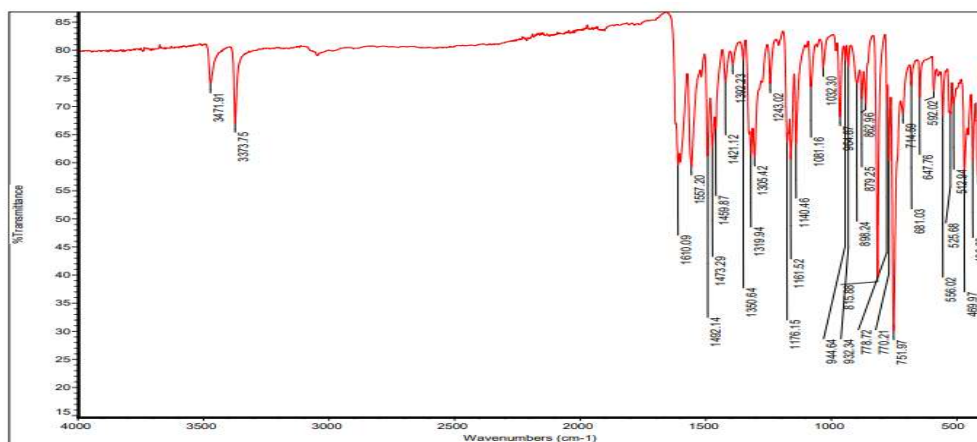
### IR Spectra of the ligand and its metal complexes

The relevant IR spectra assignment is shown on Table 4. The IR spectrum of the ligand was compared with that of the metal complexes so as to identify the coordination sites of the ligand. The IR spectra of the synthesized ligand and the complexes of Cu(II), Ni(II) and Zn(II) are recorded in the region of 500-4000 $\text{cm}^{-1}$ . A strong band was observed for the ligand around 1610.09  $\text{cm}^{-1}$ , characteristic of the azomethine (C=N) stretching vibration. In the metal chelates, the band characteristic of the azomethine group was shifted to around 1606.47  $\text{cm}^{-1}$  from 1610.09  $\text{cm}^{-1}$  in the ligand, suggesting coordination of the azomethine nitrogen atom to the metal ion [7, 9, 10]. The spectrum also showed broad bands in the region of 3373.75 and 3471.91  $\text{cm}^{-1}$  which may be due to  $\nu(\text{OH})$  and the broadening of the same was due to intermolecular H-bonding between OH groups [5, 17]. These bands were missing in the metal complexes

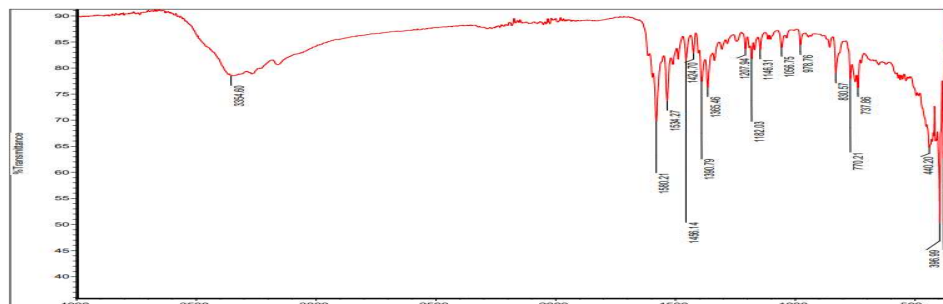
indicating that the OH character of the ligand has been lost upon complexation. The various absorption bands for the ligands and the complexes in the region 1176.15-1195.31  $\text{cm}^{-1}$  may be assigned to  $\nu(\text{C}-\text{C})$  aromatic structure [18]. Vibrations in the regions around 400.25-469.97  $\text{cm}^{-1}$  have been assigned to  $\nu(\text{M}-\text{O})$  and 380.89-396.99  $\text{cm}^{-1}$   $\nu(\text{M}-\text{N})$  bands. The bands may be due to the established coordination of the ligand to the respective metal ions in each complex compound. These agreed with the result obtained from previous literature values [2, 7, 9, 16]. Finally, the ether group,  $\nu(\text{C}-\text{O})$  has bands around 1207.94-1282.36  $\text{cm}^{-1}$  and has a shift to lower frequencies probably due to the conjugation with the bond and partly because of resonance effects and this also confirm the participation of the ether group or the aromatic ring in the coordination of the metal and the ligand.

**Table 4: Important Infrared band ( $\text{cm}^{-1}$ ) of the Ligand and its Metal Complexes**

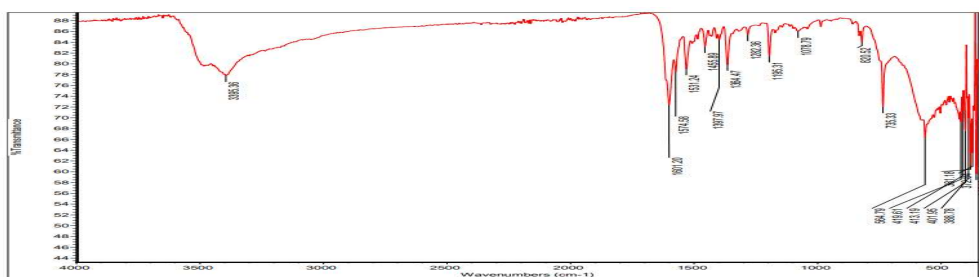
Ligand/Complexes	O - H	C - N	C - O	C - C	M - O	M - N
BHNOPDA	3471.91	1610.09	1243.02	1176.15	469.97	396.99
CuBHNOPDA	3354.60	1580.21	1207.94	1182.03	440.20	396.99
NiBHNOPDA	3395.36	1606.47	1282.36	1195.31	419.61	388.76
ZnBHNOPDA	3463.18	1601.20	-	-	400.25	380.89



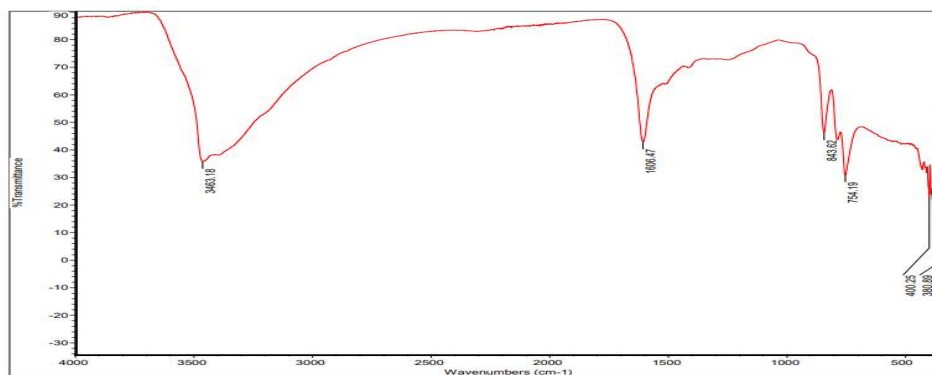
**Figure 1: FTIR Spectrum for BHNOPDA**



**Figure 2: FTIR Spectrum for CuBHNOPDA**



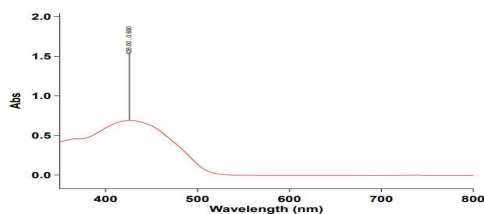
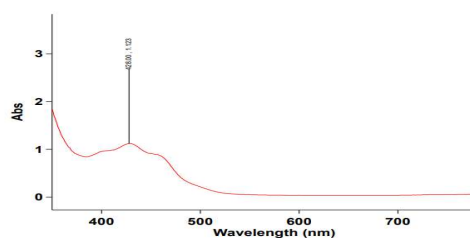
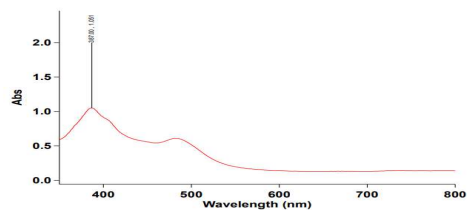
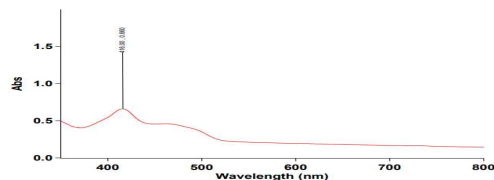
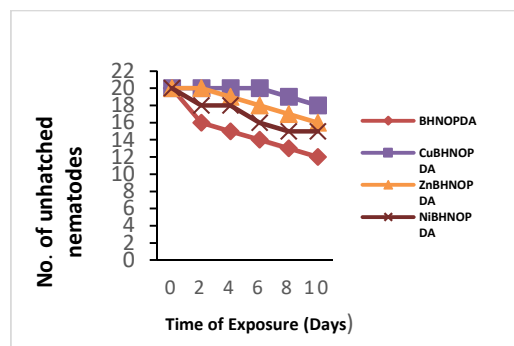
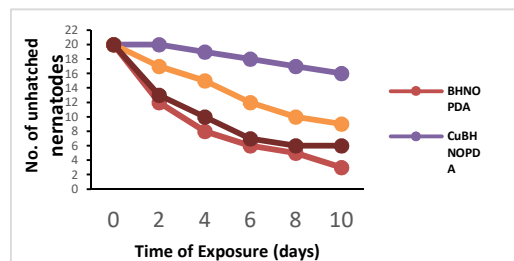
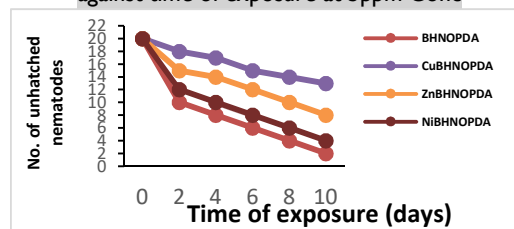
**Figure 3: FTIR Spectrum for NiBHNOPDA**



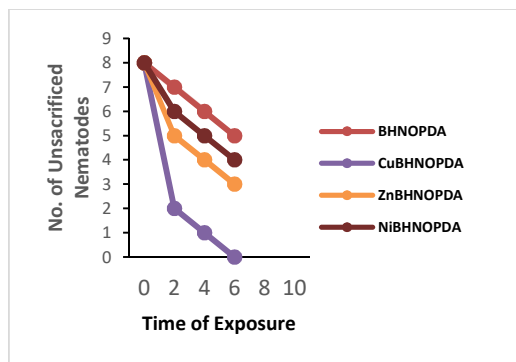
**Figure 4: FTIR Spectrum for ZnBHNOPDA**

**Table 5: Electronic and Magnetic Moment Data for the Schiff base Ligand and its Metal Complexes**

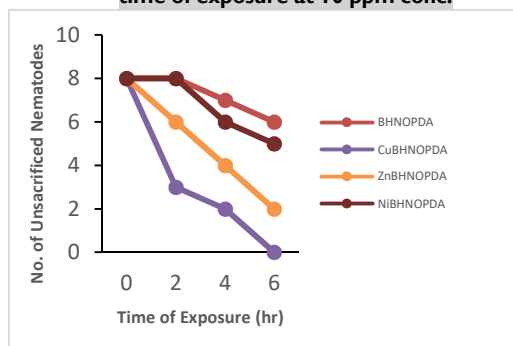
Ligand/Complexes	$\lambda_{\text{max}}$ (nm)	Assignment	Magnetic Moment (B.M)	$\mu_{\text{eff}}$
BHNOPDA	426.00	$\pi - \pi^*$	-	
CuBHNOPDA	420.00	$^2T_2 \longrightarrow ^2E_g$	2.10	
NiBHNOPDA	387.00	$^3A_{2g} (F) \longrightarrow ^3T_{2g} (P)$	3.05	
ZnBHNOPDA	416.00	C – T	-	

**Figure 5: UV-VIS Spectrum of BHNOPDA****Figure 6: UV-VIS Spectrum of CuBHNOPDA****Figure 7: UV-VIS Spectrum of NiBHNOPDA****Figure 8: UV-VIS Spectrum of ZnBHNOPDA****Figure 9: Plot of unhatched eggs of nematode against time of exposure at 10 ppm Conc.****Figure 10: Plot of unhatched eggs of nematode against time of exposure at 5 ppm Conc****Figure 11: A plot of unhatched eggs of nematode against time of exposure at 2.5 ppm Conc**

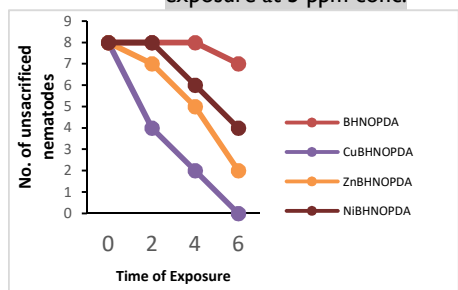




**Figure 12: A plot of un-sacrificed nematodes against time of exposure at 10 ppm conc.**



**Figure 13: Plot of unhatched nematodes against time of exposure at 5 ppm conc.**



**Figure 14: Plot of unsacrificed nematodes against time of exposure at 2.5 ppm conc.**

### Electronic spectra and magnetic moment of the ligand and its metal complexes

A useful tool for the evaluation of results provided by other methods of structural investigation is the electronic spectra also known as the ultra-violet visible spectra. It is used to designate or assign the stereochemistry of metal ions in a complex as shown by the positions and numbers of  $d-d$  transition peaks. The ligand and the metal complexes were absorbed in the visible region due to charge transfer processes from the energy in the visible light and hence, provide evidence that they are all coloured compounds [17]. The structure of the complexes was elucidated by the absorption bands they exhibited. The electronic spectra are represented in Table 5. The most useful transitions for analysis are the intense  $\pi \rightarrow \pi^*$  transitions and the weaker, but lower energy,  $n \rightarrow \pi^*$  transitions. The electronic spectrum of the ligand BHNOPDA showed a band at 426.00 nm due to the  $\pi \rightarrow \pi^*$  transition of the chromophore ( $-C=N-$ ), suggestive of a tetrahedral geometry [7, 19].

The UV-visible absorption spectra of all the metal complexes showed similarities, which indicates similarity in their structures and generally showed the characteristic bands of the free ligand with some changes both in frequencies and intensities. Upon complexation, the absorption bands of the complexes are to some extent shifted to shorter wavelength (Blue shift) compared to those of the ligand. These modifications of the shifts and intensity of the absorption bands indicates the coordination of the ligand to the metal ion. In the UV-region, Cu(II) complex showed absorption band at 400.00nm, which may be assigned to  $^2T_2-^2E$  transitions, and the magnetic moment of Cu(II) complex was 2.10 B.M. Both the electronic spectra and the magnetic values suggested a tetrahedral geometry for Cu(II) complex [19, 20]. The electronic absorption of Ni(II) complex showed one band at 387.0 nm. This band is assigned to a  $^3A_2g(F) \rightarrow ^3T_1g(P)$  transition [20 - 22] and the magnetic moment of Ni(II) complex was 3.05 B.M. These results suggested a tetrahedral geometry for Ni(II) complex. The electronic absorption of Zn(II) complex showed one band at 416.0 nm for charge transfer, which is assigned as due to  $^2E_g-T_2g$  transition, possibly in a tetrahedral environment [22]. The magnetic moment values of Cu(II) and Ni(II) complexes also indicated their paramagnetic nature, however, the electronic spectrum of Zn(II) complex does not have  $d-d$  transition but presents one band at 416.00 nm, may be attributed to a ligand to metal transfer [18, 19]. The Zn(II) complex was found to be diamagnetic. On the basis of the above observations and spectral data, it is suggested that all the metal complexes shows tetrahedral geometry structures as presented as Scheme 2.





### Nematicidal assay

The synthesized ligand and its metal complexes were tested for hatching inhibition and mortality of root knot nematodes (*M. incognita*) at three different concentrations of the synthesized compounds, which includes; 10, 5 and 2.5 ppm.

### Hatching of root knot nematode

The hatching values of root knot nematode in the synthesized compounds at three different concentrations each and at different intervals of exposure were studied and presented in Figures 1 to 3. This analysis was carried out for 10 days and the observation were made every after 2 days. In each of the treatment, exactly 20 egg masses were placed and their hatching were observed and recorded. Based on the trends of unhatched nematodes that follows in all the compounds, it was observed that, the number of unhatched nematodes was found to decrease as the time of exposure increased for both the ligand and all the metal complexes. And that for the ligand was found to be more effective than the metal complexes. In case of the metal complexes, Zn(II) and Ni(II) metal complexes showed a steady decrease in the number of unhatched as the time of exposure increased. However, Cu(II) complex was found to be extremely ineffective from the other metal complexes, having the highest number of unhatched nematodes. And the result is consistent with other findings [2, 17, 24]. Also, it was found that at lower concentrations hatching inhibition was less, whereas at higher concentrations, the egg hatching inhibition was high. These results indicate that the incorporation of metal ions in chelation can improve the nematicidal activity of the parent organic compounds.

### Mortality of root knot nematode

The mortality values of root knot nematode of the synthesized compounds at three different concentrations and at different interval of exposure were recorded and presented in Figures 4 to 6. This test was done using the synthesized Schiff base ligand and its metal complexes at three (3) different concentrations each, and the analysis was completed within 6hrs in which the observations were made every after 2hrs. In each of the treatment, exactly eight (8) second juveniles were pick and placed in each of compounds and their mortality was been observed and recorded. From the results of the analysis, it was generally observed that the number of unsacrificed nematodes decreases as the time of exposure increased for both the ligand

and all the metal complexes. And the result for the ligand was found to be less effective than the metal complexes. And for the metal complexes, Cu<sup>2+</sup> complex possessed the highest activity than the others. It was also observed that the number of unsacrificed nematodes was higher at lower concentrations, but lower at higher concentrations. This implies that mortality rate of nematodes increases with increase in concentration and decrease with decrease in concentration. This greater activity of the metal complexes might be due to azomethine linkage and heteroatoms present in these compounds [20, 25].

### Conclusion

The Schiff base Bis(2-hydroxy-1-naphthaldehyde)-o-Phenylenediamine and its corresponding metal (II) complexes of Cu<sup>2+</sup>, Ni<sup>2+</sup> and Zn<sup>2+</sup>, were synthesized and characterized successfully. The low molar conductance value indicates that the complexes are non-electrolytic in nature. Based on the physiochemical and spectral studies, a tetrahedral geometry has been assigned for all the complexes. The Schiff base and its metal complexes were insoluble in water, but soluble in most organic solvents. In the present studies, all the synthesized compounds had shown nematicidal activity against root knot nematode (*M. incognita*), by inhibiting egg hatch and causing second stage juveniles mortality and the nematicidal activity of these compounds was found to be dose and time dependent. Egg masses of the nematodes were obtained from the roots of heavily infected tomato (*L. esculentum*) for studies. The hatching analysis was carried out within 10days and the observation were made every after two days interval until a complete hatch rate was observed. And for mortality, the analysis was carried out within 6hrs, and the observation was done at an interval of 2 h until a complete mortality was observed. All the compounds showed complete hatch inhibition and mortality at concentration above 10 ppm, whereas the compounds at low concentrations showed less hatch inhibition and mortality. The results indicated that egg hatch inhibition and mortality increased with increase in concentration of the compound, but decreased as the time of exposure increases.

### Declaration of conflicting interests

The authors declared no potential conflicts of interest

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#### Cite this article

Iorungwa M.S., Iornumbe E.N., Wangka G.K., Timi S. and Iorungwa P.D. (2021). Synthesis, Characterization and Nematicidal Activity of Bis-(2-Hydroxy-1-Naphthaldehyde)-O-Phenylenediamine and its  $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$  and  $\text{Zn}^{2+}$  Ions. *FUAM Journal of Pure and Applied Science*, 1(2):29-38

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