

Synthesis, Characterization and Applications of the Synthesized Copper(II) Complexes Containing Schiff Base Ligands in Antimicrobial Activities and Catalytic Reduction of Nitroaromatic Compound

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Abstract - This article describes the preparations of a series of Schiff base ligands [L1], [L2], and [L3] and their corresponding copper(II) complexes; [C1], [C2] and [C3]. The synthesized ligands and their copper(II) complexes were characterized using Fourier Transform Infrared (FTIR), Nuclear Magnetic Resonance (NMR) and Ultraviolet-visible (UV-Vis) spectroscopy. The ability of the compounds to inhibit bacteria species were tested against *E. Coli*, *Staphylococcus* and *Streptococcus* bacterial. The results revealed that all the compounds were active against bacteria species. The inhibition rate is in order L3>L2>L1 for ligands and C3>C2>C1 for complexes. The catalytic activities of the synthesized copper(II) complexes were evaluated in the reduction of aromatic compounds in the presence of sodium borohydride which acts as reducing agent. The progress of reaction was monitored using UV-visible spectroscopy and the percentage conversion was determined from the spectroscopy data. The results showed that [C3] has the highest catalytic activities with 97.5% conversion, followed by [C2] complex with 95.2% conversion, then [C1] complex with 90.8% conversion. The optimization of the catalyst dose shows that 1.0 mg of the catalyst dose was the most optimized amount with the highest conversion of 94.6% than other dose of 0.5 mg (92.4%) and 1.5 mg (91.4%). Recyclability and reproducibility tests of copper(II) complexes confirmed that all the three complexes were active, efficient and possess excellent reproducibility with consistent catalytic performances and could be re-used again without significant decrease in the catalytic activities.

Keywords: Schiff Base Ligands; Copper(II) Complex; Antibacterial studies, Catalysis, Spectroscopic analysis.

I. INTRODUCTION

Schiff bases are compounds derived from carbonyl compounds in which the functional group of the carbonyl compounds is substituted by an imine or azomethine group [1]. Typically, Schiff bases are prepared through condensation reaction of amine and an aldehyde in presence of organic

solvents such as, tetrahydrofuran (THF), methanol and 1, 2-dichloroethane [2]. Figure 1 shows the preparations of Schiff base (a) without refluxing the solvent and (b) with refluxing the solvent.

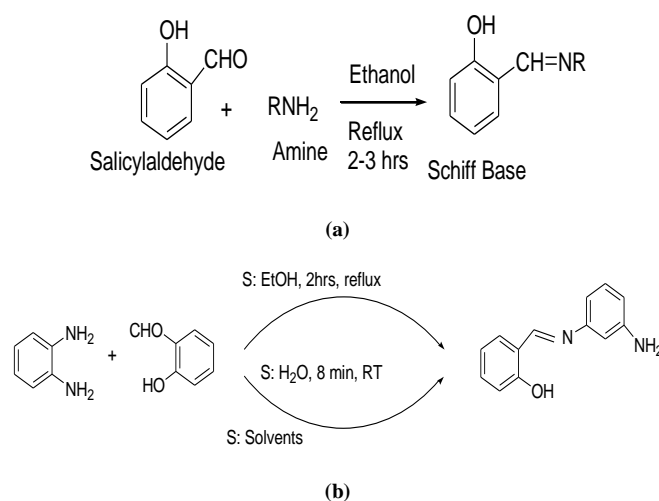


Figure Error! No text of specified style in document.: Preparations of Schiff base ligands via the reaction of salicylaldehyde and a primary amine (a) without refluxing the solvent and (b) with refluxing the solvent

Schiff bases are among the most commonly used ligands due to their sigma donations and pi acceptor attributes and their reactivity relationship as well as the bonding mode features of Schiff base ligands with metal complexes derived great influence in coordination's chemistry [3]. They coordinates easily with diverse range of transition metal ions, resulting in stable and deeply coloured metal complexes with impressive properties [4]. The properties associated with the Schiff base ligands, such as catalytic, magnetic, fluorescent as well as electrochemical, make the transition metal complexes of Schiff-base ligands extremely interesting [4]. Many metal complexes containing Schiff base ligands such as palladium, gold, and silver complex have been reported but they are expensive and the synthetic route are complicated. Copper complexes is a promising alternative to chemist owing to its abundance and preparatory parts are environmental friendly [5]. Copper metals can form complexes with neutral or anionic ligands and possess multiple oxidation states essential for their

reactivity. Such coordination complexes serve as catalysts for substrates activation and reaction accelerations and contributes to the formations or cleavage of a number of chemical bonds. The behaviour and selectiveness of these complexes are closely related to ligands design (Dursch). Reactions involving catalyst and transition metal, most essential factors in determining the role of catalyst is the accessibility of catalyst, less expensive metals such as copper have gained attention in the development of catalytic system [6]. The effect of ligands on the structure and reactivity of transition metal complexes is an important area of research in coordination and organometallic chemistry as well as in catalysis [7].

II. METHODOLOGY

2.1 Synthesis of N'-salicylidene-2-aminophenol (L1)

Salicylaldehyde (1.07 mL, 10.00 mmol) was refluxed with 2-aminophenol (1.10 g, 10.00 mmol) in ethanol for 3-4 hours forming the precipitate. Filtration of the precipitated product was done under suction, washed with ethanol and recrystallized from ethanol. The product obtained was dried over silica gel in a desiccator [8].

2.2 Synthesis of N'-salicylidene-2-aminothiazole (L2)

2-aminothiazole (5.22 g, 24.88 mmol) was weighed accurately, mixed with methanol (10 mL) and transferred with constant stirring to potassium hydroxide in methanol. The mixture was stirred and filtered. The filtrate (3.03 mL, 24.88 mmol) was measured and mixed with salicylaldehyde (1.07 mL) and methanol (20 mL) was dropwise added with constant stirring. The mixture was later evaporated at reduced pressure and kept at ambient temperature for 2 hours. Cold alcohol was used to washed the orange precipitate obtained and filtered by suction, the product (92.6%) was washed with diethyl ether and dried [8].

2.3 Synthesis of N,N'-bis(salicylidene)-o-phenylenediamine (L3)

The Schiff base was prepared by mixing 50 mL hot ethanolic solution of ortho-phenylenediamine (1.08 g, 0.01 mol) with 50 mL hot ethanolic solution of salicylaldehyde (2.44 g, 0.02 mol). The obtained mixture was refluxed with stirring for 2-3 hours. The precipitate formed was collected by filtration via Buchner funnel, recrystallized from ethanol and the product was dried at ambient temperature over anhydrous calcium chloride [9].

2.4 Synthesis of N'-salicylidene-2-aminophenol copper(II) acetate (C1)

A copper acetate monohydrate $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$, (0.123 g, 0.617 mmol) was completely dissolved in ethanol (10 mL)

and ethanolic solution of the synthesized ligand L1 (0.30 g, 1.23 mmol) was added drop-wise with vigorous stirring. A green precipitated of the product was form which was suction filtered, washed with ethanol and dried in a desiccator [8].

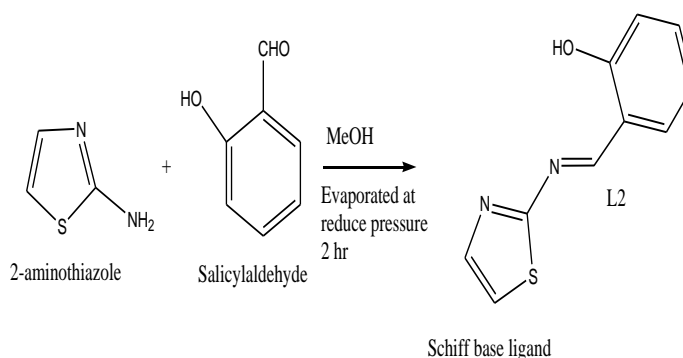
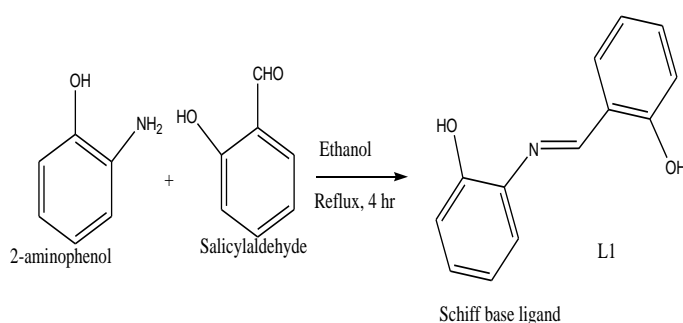
2.5 Synthesis of N'-salicylidene-2-aminothiazole copper(II) acetate (C2)

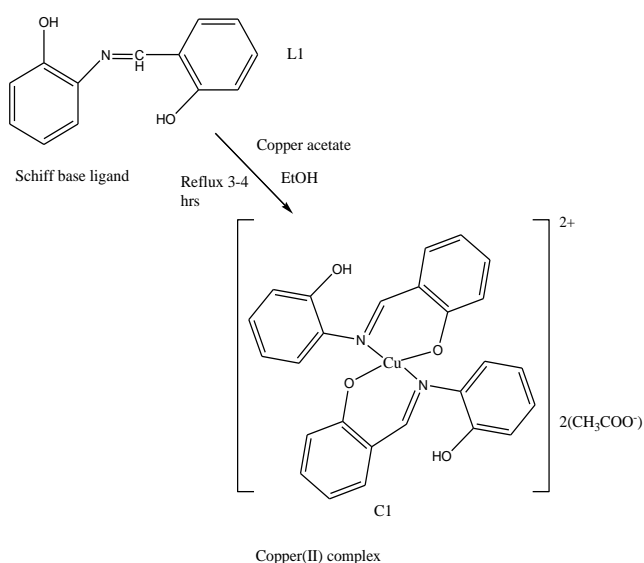
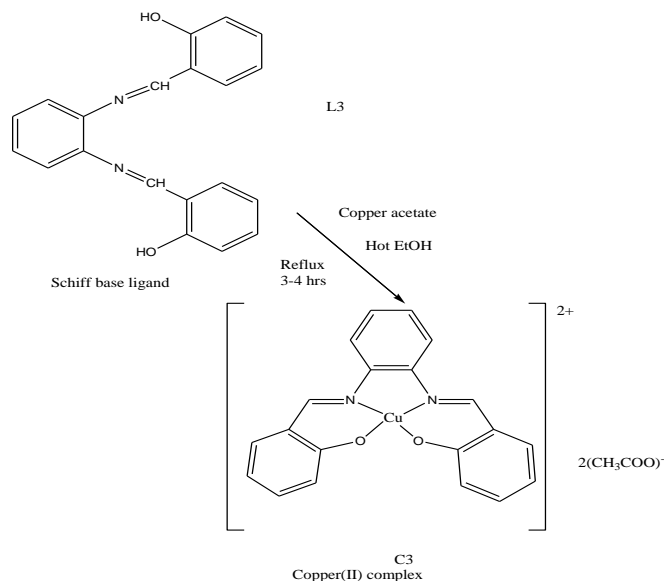
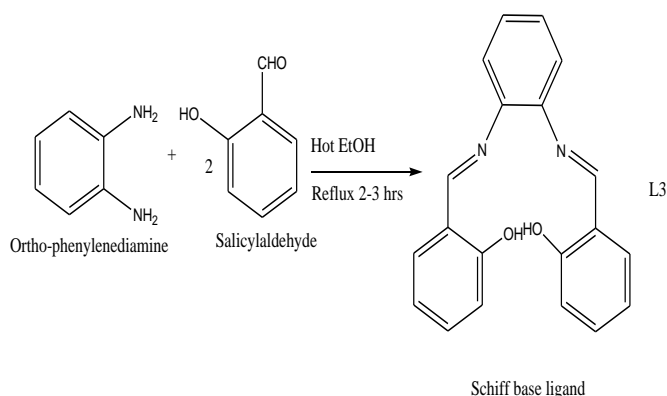
Hot solution of the copper acetate monohydrate (60 °C, 0.001 mmol) was mixed with the hot solution of Schiff base ligand (75 °C, 0.629 g, 0.002 mmol) in 25 mL methanol. The mixture was refluxed for 3-4 hours with constant stirring to form the precipitate of the complex. The precipitate was filtered and the product was purified by washing with methanol and dry [10].

2.6 Synthesis of N,N'-bis(salicylidene)-o-phenylenediamine copper(II) acetate (C3)

The synthesis of the metal complex was carried out by mixing 50 mL hot ethanolic solution of Schiff base ligand (3.16 g, 0.01 mol) to the hot ethanolic solution (50 mL) of copper acetate monohydrate (1.71 g, 0.01 mol). The pH was adjusted by adding few drops of dilute ammonium hydroxide solution which lead to the precipitate of the solid chelate. The mixture was refluxed for 2-3 hours with constant stirring which lead to the formation of precipitate which was washed until the colour turned to clear as product [9].

Reactions Scheme for the Synthesis





III. RESULTS AND DISCUSSIONS

3.1 FTIR Analysis

In infrared spectra of ligand L1, the 1614 cm^{-1} stretching mode allocated to azomethine group ($\text{C}=\text{N}$) in the Schiff base ligand indicates that the 2-aminophenol free amino group (NH_2) was converted to the azomethine group. The phenolic stretching mode appeared at 1228 cm^{-1} band, indicating the presence of phenolic oxygen group [11]. The appearance of band allocated to hydroxyl group (OH) in free Schiff base ligand at 3445 cm^{-1} stretching mode indicate that the hydroxyl group was presence in the Schiff base ligand [9].

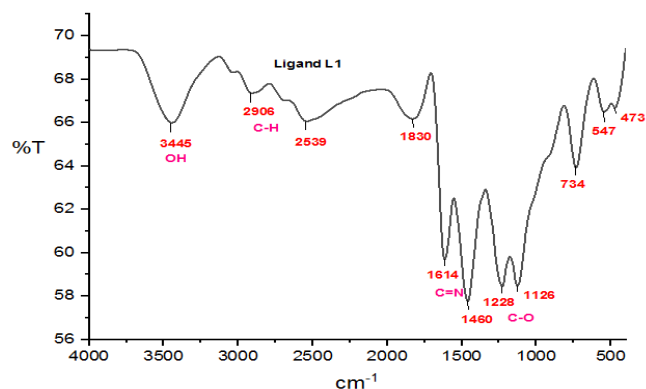


Figure 1: Shows the infrared spectrum of Schiff base ligand L1

Similarly, the band at 3445 cm^{-1} allocated to hydroxyl group (OH) in Schiff base ligand L1 was shifted to lower absorption band of 3430 cm^{-1} in the copper(II) complex suggesting that it was coordinated to the metal atom. The azomethine band was shifted from 1614 cm^{-1} to the lower absorption band of 1589 cm^{-1} from ligand to the complex suggesting that the azomethine group ($\text{C}=\text{N}$) was coordinated to the metal atom via azomethine nitrogen [10]. The phenolic band at 1228 cm^{-1} in free Schiff base ligand shifted to lower

band at 1153 cm^{-1} in the complex suggesting the coordination via phenolic oxygen. Thus, the ligand is bidentate. The ligand's mode of coordination was further confirmed by the appearance of two new bands at 743 cm^{-1} and 534 cm^{-1} in the spectra of the complex which was allocated to (Cu-N) and (Cu-O) respectively [11].

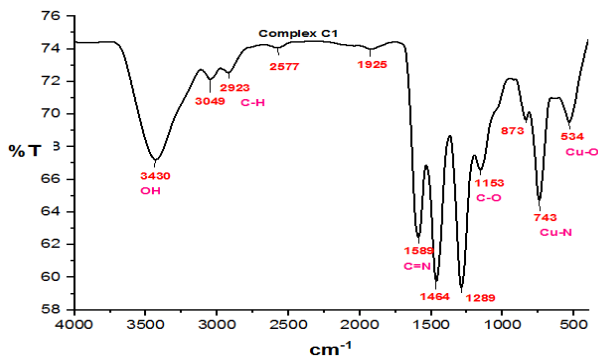


Figure 2: Shows the infrared spectrum of C1 complex

Table 1: FTIR data of ligand and complex

Functional groups	Freq. cm^{-1}	Freq. cm^{-1}
OH	3445	3430
C-H	2906	2923
C=N	1614	1589
C-O	1228	1153
Cu-N	-----	743
Cu-O	-----	534

3.2 $^1\text{H-NMR}$ Analysis

In proton NMR spectra of ligand L1, the signals of hydroxyl proton (OH) appeared as singlet at a chemical shift of $\delta 12.28\text{ ppm}$. The signal of azomethine proton (C=N) was present as singlet at $\delta 8.71\text{ ppm}$. The protons of the phenol were observed as multiplet at the signal from $\delta 7.47\text{--}7.43\text{ ppm}$. The aromatic protons was appeared as multiplet from $\delta 7.19\text{--}5.79\text{ ppm}$ in the Schiff base ligand L1 [12]. The appearance of extra peak at $\delta 1.58\text{ ppm}$ in the spectrum of ligand L1 was due to the presence of impurities [13].

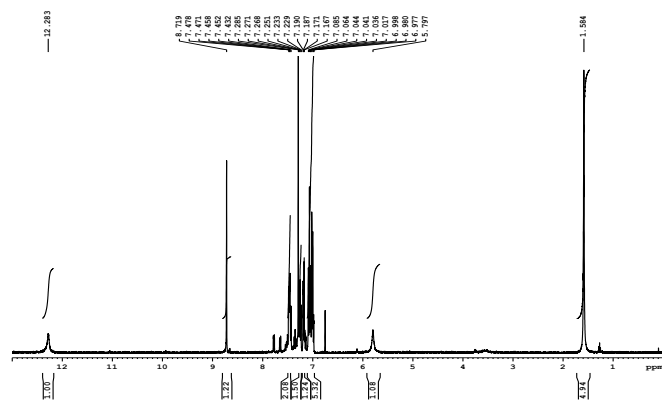


Figure 3: Shows the proton NMR of copper(II) complex

Similarly, the proton NMR of copper(II) complex C1, shows the strong involvement of C-O group in chelation through deprotonation of OH group in the Schiff base ligand which was indicated by singlet at $\delta 12.28\text{ ppm}$ in the free Schiff base ligand. The azomethine proton was shifted from $\delta 8.71\text{--}\delta 9.53\text{ ppm}$ from the ligand to the copper(II) complex suggesting the coordination of azomethine via nitrogen atom [11]. A shift of aromatic proton from $\delta 7.19\text{--}\delta 6.71\text{ ppm}$ from ligand to the copper(II) complex confirmed the complexation. The appearance of extra peak at $\delta 3.45\text{--}1.28\text{ ppm}$ in the spectrum of complex C1 was due to the presence of impurities [12].

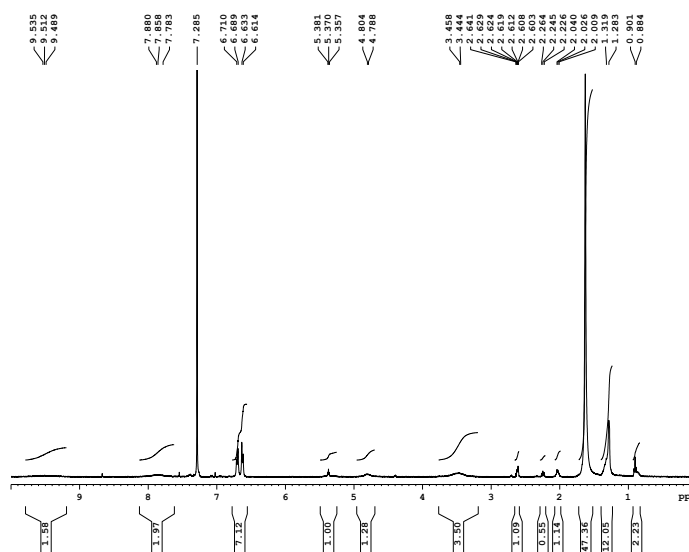


Figure 3: Shows the proton NMR of Schiff base ligand

Table 2: $^1\text{H-NMR}$ data for Schiff base ligand and complex

Assignments	Chemical shift (ppm)	
	L1	C1
OH	12.28 (s)	-----
N=CH	8.71 (s)	9.53-9.48 (m)
Phenolic-H	7.47-7.43 (m)	7.88-7.78 (m)
Aromatic-H	7.19-5.79 (m)	6.71-4.78 (m)

3.3 UV-Visible Analysis

The UV-visible spectrum of the Schiff base ligand L1, in methanol solution was recorded between $200\text{--}800\text{ nm}$. The weak bands in UV-visible region at 253 nm and 326 nm was allocated to $\pi\text{-}\pi^*$ transition of the aromatic ring and salicylidene ring and strong band at 433 nm was allocated to $n\text{-}\pi^*$ transition of the azomethine group. This value was found to agree with the literature [14]. Similarly, the UV-visible absorption band at 243 nm and 319 nm exhibited by the copper(II) complex C1, was allocated to $\pi\text{-}\pi^*$ transition suggesting that the ligand has coordinated to the metal atom via azomethine nitrogen in the copper(II) complex. The $n\text{-}\pi^*$ transition undergoes blue shift suggesting the lone electrons

pair of oxygen and nitrogen are link to metal ion. The $\pi-\pi^*$ transition of C=O group and organic moieties of copper(II) complex was found at 243. This has further undergone red shift with an additional wavelength to the absorption at 423 nm which attributed to $n-\pi^*$ of the phenol and C=N group. Figure 4.5 shows the UV-visible spectra of Schiff base ligand L1 and its copper(II) complex C1 [14].

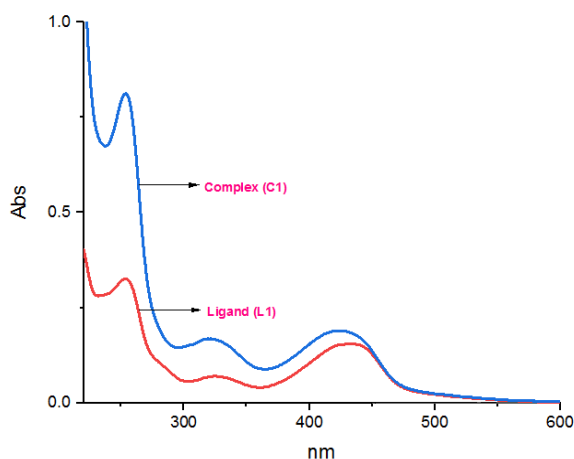


Figure 4: Uv-visible data of ligand and complex

Table 3: UV-Visible data for Schiff base ligand and copper complex

Comps	Wavelength (nm)	Assignment
Ligand	253, 326	$\pi-\pi^*$
	433	$n-\pi^*$
Complex	243, 319	$\pi-\pi^*$
	423	$n-\pi^*$

IV. CATALYTIC REDUCTION

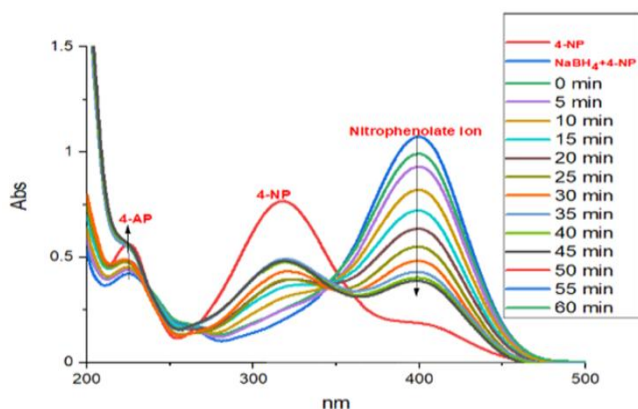


Figure 5: Catalytic activity of aromatic arene (4-NP)

Table 3 portray the catalytic performance of copper(II) complexes at different amount of catalyst used. C3 has the highest performance compared to C2 and C1, this could be due to the high stability of the C3 and also because C3 is tetradentate while C2 and C1 are bidentate.

Table 4: Percentage conversion of catalytic activity

Catalyst	Dose (mg)	% Conversions
C1	0.5	76.4%
	1	90.8%
	1.5	88.6%
C2	0.5	94.3%
	1	95.2%
	1.5	91.4%
C3	0.5	92.7%
	1	97.5%
	1.5	95.6%

4.1 Reaction rate for 4-Nitrophenol reduction

The plot of $\ln(A_t/A_0)$ versus time for 4-NP catalytic reductions gave strong linear corrections suggesting that first-order kinetics accompanied the reduction of 4-NP in the existence of NaBH_4 catalyst by copper complexes [14]. The equation rate can be written as $Kt = \ln(A_0/A_t)$, where K is the first-order kinetic rate constant, A_0 is the initial 4-NP concentration, and A_t is the final t-time concentration. The observed values of the first-order rate constant (k) and correlation coefficient (R^2) for copper(II) complex catalyst was $R^2 = 0.8533$, $R^2 = 0.9488$ and $R^2 = 0.9986$ for C1, C2 and C3 respectively (Figure 4.20). From the graph, it can depicted that C3 provides better rate of reaction than the corresponding C2 and C1. The higher rate of catalytic reduction of 4-NP is due to the exchange of electrons or the rate of transfer of electrons to the surface of the catalyst, which can be triggered by the diffusion of 4-NP to the surface of the metal and the diffusion of 4-NP away from the surface [14].

Plot of $\ln A_0/A_t$ vs Time (min) for 4-NP reductions catalyst by C1, C2 & C3

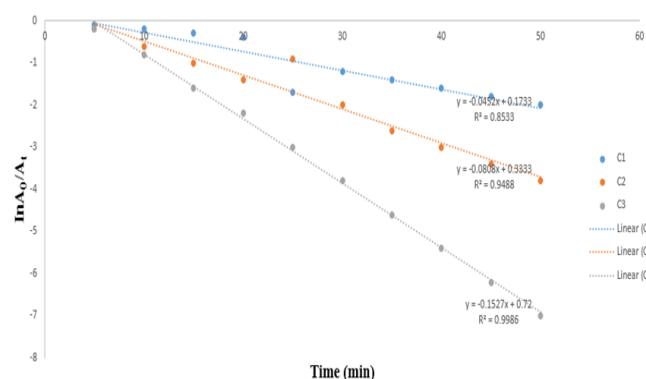


Figure 6: Rate of reaction for 4-nitrophenol reduction

4.2 Mechanism for the catalytic reduction

A pathway for the catalytic reduction of 4-nitrophenol to 4-aminophenol using copper(II) complex catalyst was formulated. The incorporation of sodium borohydride to the reaction medium (A) allows 4-nitrophenol to become

deprotonated, resulting in the formation of intermediate (4-nitrophenolate ion). As the required amount of copper(II) complex catalyst was introduced in to the reaction medium (B), the catalytic reduction immediately started resulting in the liberation of hydrogen gas (C) which conveyed the electrons from the donor sodium borohydride to the acceptor 4-nitrophenol after the adsorption of both to the surface of the catalyst (D). Once all the oxygen in the system has reacted, the reduction reaction of 4-nitrophenol begins and the evolution of small hydrogen gas bubbles covering the catalyst particles remains well distributed (E) in the reaction medium which creates a suitable environment for the catalytic reduction to occur. Finally, desorption can take place where water molecules are removed leading to the formation of product (F) [13].

Table 5: Optimization of catalyst at different dose

Catalyst	Dose (mg)	Cycle	% Conversion
C3	0.5	First cycle	92.4
	1	Second cycle	94.6
	1.5	Third cycle	91.4

4.4 Purification and characterization of products

In order to examine the successful conversions of 4-nitrophenol to 4-aminophenol, the product (4-AP) must be purified and characterized. Purification of the products was done by silica gel column chromatography and ¹H NMR spectroscopy was adopted for the characterization of the product. As shown in Figure 14 and 15 for comparison which shows the spectrum of the product that obtained after the successful conversions of 4-nitrophenol to 4-aminophenol. The appearance of signal at δ6.75 ppm and δ5.36 ppm was due to the aromatic and amino proton respectively. The remaining two peaks at δ2.63 ppm and δ2.61 ppm correspond to the solvent and residual water respectively [12].

4.5 Isolation of the products

UV-vis spectroscopy was adopted to monitor the progress of the conversion reaction. When the conversion was achieved, the products were isolated by diluting with diethyl ether (30 mL) and separated using liquid-liquids extraction. The extraction steps were repeated three times, and anhydrous sodium sulphate (Na₂SO₄) was combined with diethyl ether fractions containing the product. Diethyl ether was then evaporated via a rotary evaporator [12]. The purification of 4-aminophenol by chromatography using silica gel column was carried out by eluting the sample with a solvent consisting of hexane and ethyl acetate in a ratio of 1:2 together with the 30 mL 4-aminophenol standard, each eluent was introduced onto a thin layer chromatography (TLC) plate. The 4-aminophenol fraction was mixed, and solvents evaporated using a rotary evaporator. The result was characterized by proton NMR and UV-visible spectroscopy [17].

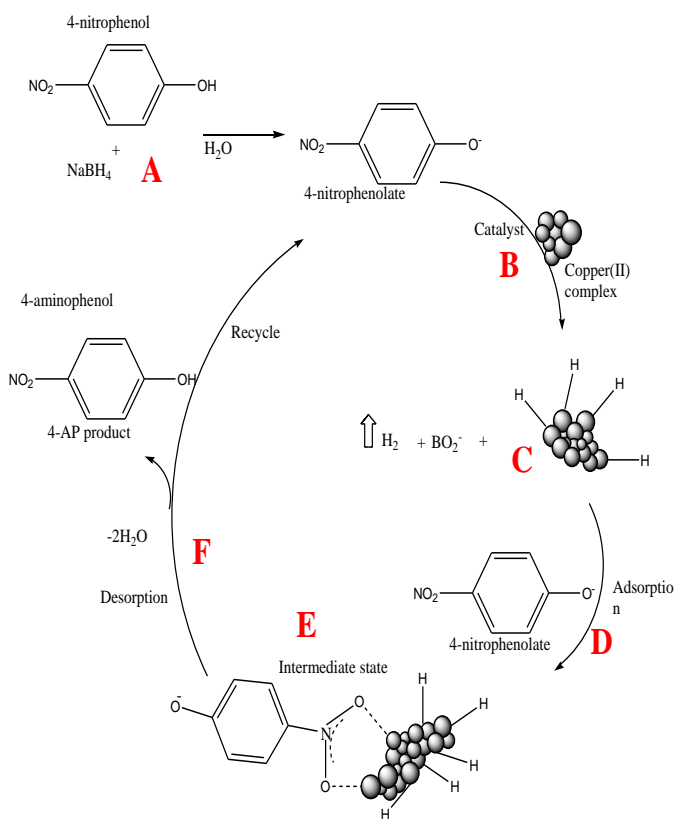


Figure 7: Mechanism for the catalytic reduction

4.3 Optimization of the amount of copper (II) catalyst

Under the same conditions as the model reactions, the catalytic experiments were carried out in triplicate, except that the amount of catalyst was changed (0.5 mg, 1.0 mg, and 1.5 mg) and the percentage conversions for each catalyst amount was calculated. 1mg of the catalyst used was found to be the optimized amount because of the higher percentages conversion when compared with the other amount. The result obtained agreed with the literature [11] shows the optimization of the catalyst at different dose.

V. ANTIBACTERIAL STUDIES

The antibacterial activity tests were conducted according to standard procedures using three different bacterial strains [15]. The results were recorded as shown in Table 6. The ability of the Schiff base ligands and their copper (II) complexes to inhibit the growth of the bacteria was compared to that of the known standard antibacterial drug (Gentamicin) as a control. The Schiff base ligand and their copper (II) complexes demonstrated abilities to inhibit bacterial growth. The results show that L1 has less inhibition ability than L2 and L3 across all bacterial species tested. The rate of inhibitions

are in the order $L3 > L2 > L1$. Similarly, C3 has the highest inhibition potential against bacterial species [15].

Table 6: Antibacterial activities of Schiff base ligand and copper complex

Test organism: E. Coli					
Samples	Conc. (mm)	First Run R1	Second Run R2	R1+R2/2	Control
L1	25	7.6	7.4	7.5	11.5
C1	50	8.6	8.3	8.5	
Test Organism: Staphylococcus Aureus					
L2	25	11.5	11.5	11.4	11
C2	50	10.7	10.5	10.6	
Test Organism: Streptococcus					
L3	25	11.2	11.5	11.4	11
C3	50	10.5	10.7	10.6	

VI. CONCLUSIONS

The preparations of a series of Schiff base ligands [L1], [L2], and [L3] and their corresponding copper(II) complexes; [C1], [C2] and [C3]. The synthesized ligands and their copper(II) complexes were characterized using Fourier Transform Infrared (FTIR), Nuclear Magnetic Resonance (NMR) and Ultraviolet-visible (UV-Vis) spectroscopy. The ability of the compounds to inhibit bacteria species were tested against E. Coli, Staphylococcus and Streptococcus bacterial. The results revealed that all the compounds were active against bacteria species. The inhibition rate is in order $L3 > L2 > L1$ for ligands and $C3 > C2 > C1$ for complexes. The catalytic activities of the synthesized copper(II) complexes were evaluated in the reduction of aromatic compounds in the presence of sodium borohydride which acts as reducing agent. The progress of reaction was monitored using UV-visible spectroscopy and the percentage conversion was determined from the spectroscopy data. The results showed that [C3] has the highest catalytic activities with 97.5% conversion, followed by [C2] complex with 95.2% conversion, then [C1] complex with 90.8% conversion. The optimization of the catalyst dose shows that 1.0 mg of the catalyst dose was the most optimized amount with the highest conversion of 94.6% than other dose of 0.5 mg (92.4%) and 1.5 mg (91.4%). Recyclability and reproducibility tests of copper(II) complexes confirmed that all the three complexes were active, efficient and possess excellent reproducibility with consistent catalytic performances and could be re-used again without significant decrease in the catalytic activities.

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