Science World Journal Vol 13(No 4) 2018
www.scienceworldjournal.org
ISSN 1597-6343
Published by Faculty of Science, Kaduna State University

Full Length Research Article

SYNTHESIS AND EVALUATION OF THE EFFICACIES OF SOME SCHIFF BASES FOR THE REMOVAL OF HEAVY METALS FROM WASTEWATER

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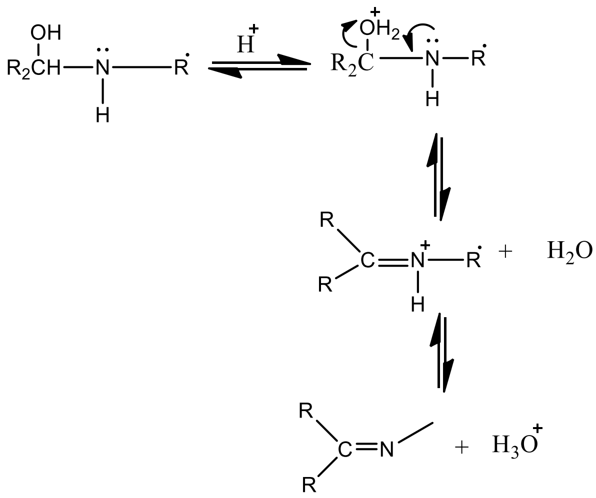
ABSTRACT
The Schiff bases, 2-aminoethyl-1,3-diphenyl-3-((2-(piperazin-1-ylethyl)imino)propylidene)-propylethane-1,2-diamine with 89% yield, and 2,2-ethylideneamino)ethyl)phenol-2,2-hydroxybenzalidene) ethyl)acetimedate salt with 91% yield, represented as L_1 and L_2 respectively were synthesized and characterized. Their metal ion removal competences for five heavy metals (Cu, Fe, Co, Cd and Pb) was investigated, in three categories of wastewater obtained from Lambun Dan Lawal (LDL), Kofar sauri (K/sauri) and Youth Craft Village (YCV). The Schiff bases were characterized before and after complexation, by spectroscopic techniques such as Infrared spectroscopy, Molar conductance, UV-Visible, Melting and Decomposition temperature and solubility test. The concentration of the heavy metals both in the wastewater and the complexes was determined by Atomic Absorption Spectroscopy (AAS). The Schiff bases shows outstanding character, by adsorbing/removing most of the targeted heavy metals at variable concentration, especially copper which shows the highest concentration. The results suggested that, the two Schiff bases can be used as an alternative chelating agents, for the removal of heavy metals from wastewater.

Keywords: Schiff base, Wastewater, Heavy metals, Evaluation, Extraction

INTRODUCTION
Schiff bases are group of compounds, with imine functional group. The preparations of these compounds has attracted the interest of both organic and inorganic chemists for decades. They are prepared by condensing an aldehyde or ketone with an amine, generally refluxed in alcohol. Schiff bases can accommodate different metals in variable oxidation states, there by controlling the performance of the metals in a large variety of important catalytic transformations. (Gaikwad and Yadav, 2014) The formation of this azomethene bond plays important role in organic synthesis, (El-Tab *et al.,* 2016) which normally takes place under acids or base catalysis or with heat. (Sonar *et al.,* 2018) the scheme 1 below, shows an acid catalyzed mechanism of a Schiff base formation.



Scheme 1: Acid catalyzed formation of a Schiff base

Schiff's bases offer a useful and flexible series of ligands that can bind with various metal ions to form complexes, with suitable characteristics for theoretical/practical applications. Since the publication of Schiff base metal complexes to date, a large number of polydentate Schiff's base compounds have been structurally characterized and studied (Tarafder *et al.,* 2001, Mohammed, 2015)

In Schiff base, carbon-nitrogen double bond (C=N) and other donor atoms, like oxygen, plays important role in co-ordination chemistry. (El-Tab *et al.,* 2016) They are generally reported as Lewis bases that normally form insoluble salts with strong acids. (Sonar *et al.,* 2018) The π-system in a Schiff's base often enforces a geometrical constriction and affects the electronic structure as well. Thermochemical properties of Schiff's bases have fascinated numerous researchers, due to their ability to bind through NO or N_2O donor atom sets (Vogel 1991). The knowledge of Schiff's base metal derivatives involving bidentate or tetra dentate bonding of ligands in biological systems, significantly contributed to unravel their nature and behavior in numerous activities (Long, 1995, Mohammed 2015)

Freshwater bodies such as groundwater, ponds, lakes, streams and rivers etc. contain water with a negligible amount of salt. They are source of water for industries, drinking and numerous

Science World Journal Vol 13(No 4) 2018
www.scienceworldjournal.org
ISSN 1597-6343
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agricultural activities. Nevertheless, many water resources comprise wastes like; heavy metals, sewage, fertilizers, decaying animal, industrial pollutants and other waste materials. All these have been proven to be hazardous to the aquatic ecosystem. (Halnor, 2015) The environmental problems due to globalization and speedy industrialization are becoming disadvantageous to human beings due to increase in pollution. Heavy metals present in wastewater and industrial effluent are major concerns of environmental pollution. They are generally considered as those metals whose density exceeds 5g per cubic centimeter, some of them are shown in table 1 below. (Gunatilake 2015) Heavy metals which are harmful to humans generally includes cadmium, chromium, copper, mercury, lead, zinc, iron, arsenic, cobalt, nickel etc. They are found naturally in the soil in minute amounts, and in industrial wastewater due to different chemical processes such as precipitation, filtration, ion exchange etc. They are normally found in limited amount or above the permissible limit which causes few problems. (Halnor, 2015)

The increase of heavy metal pollution in wastewater necessitated scientist to employ adsorption treatment technique, which offered flexibility in operation and design, and in many cases produces high-quality treated effluent. In addition, adsorption can be reversible process, adsorption can mostly be applied to remove metals, because the adsorbents can be regenerated by suitable desorption process. (Fu and Wang, 2011).

The use of organic-based ligands such as Schiff bases that form stable coordination compounds has been suggested as a suitable procedure for the extraction and removal of dissolved heavy metals from contaminated water (Quan *et al*., 2003, Dede *et al*., 2009). Several Macrocyclic compounds have been reported, for the selective adsorption and removal of heavy metals from wastewater, while in this research, we demonstrated a similar extraction or adsorption ability, of some stable non-cyclic Schiff bases towards the heavy metals.

Table 1: The MCL standard of some hazardous heavy metal (Babel and Kurniawon 2003; and SON 2015)

Heavy Metal	Toxicities	MCL (mg/L)
Copper	Liver damage, Wilson disease, insomnia	1.00
Lead	Damage the fetal brain, diseases of the kidneys, circulatory system and nervous system	0.01
Cadmium	Kidney damage, renal disorder, human carcinogen	0.003
Iron	None	0.30
Chromium	Leads to cancer	0.05

Key: MCL = Maximum Permissible Limit, mg/L = Milligram per liter

The three (3) wastewater samples were obtained from agricultural and industrial sites, within Katsina metropolis as shown by the Figure 1, below.

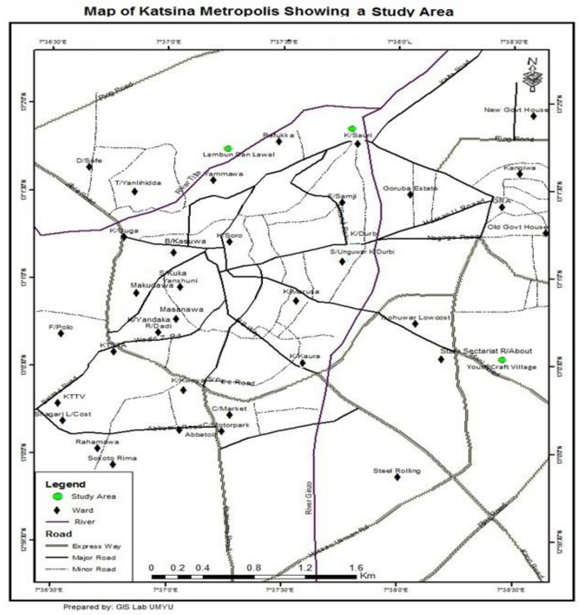

Figure 1: Map of Katsina metropolis showing the study area

MATERIALS AND METHODS

MATERIALS

All reagents used were of analytical grade, procured from Zayo-Sigma and Qualikems and used without further purification. All weighing was done on an electrical meter balance. Melting point/decomposition temperatures were determined using IA9000 series digital melting point apparatus while conductivity measurements were conducted using HI-2300 conductivity meter, pH was measured using a pH HI-99121 meter, The amount of heavy metals were determined by using Flame atomic absorption spectrophotometer. IR measurements were recorded using Shimadzu FTIR spectrophotometer. Others include acetone, methanol, ethanol, benzene, waste water, etc.

METHODOLOGY

Synthesis of the Schiff Base (L_1)

A hot ethanolic solution (20ml) of 1,3-diphenylpropane-1,3-dione (2.24g, 0.02mol) and 20ml ethanolic solution of 2-(piperazin-1-yl)ethanamime (2.58g, 0.02mol) were mixed together and constantly stirred. The mixture was refluxed at 80^oC (±5) for 6 hours in the presence of few drops of HCl (P^H~ 3-4). Progress of the reaction was followed using a paper chromatography. Upon cooling, a yellow colored compound was precipitated, filtered, washed with cold ethanol and dried under vacuum over P_4O_{10} (Rehman *et al.,* 2014). As shown by scheme 2.

Note; Similar procedure was adopted for L_2

Wastewater Sample Preparation for AAS

To each sample of the waste water, (YCV, LDL, and K/sauri), 5ml of 65% HNO_3 was added, and the mixture was boiled gently for 30-45min. after cooling, 2.5ml of 70% $HClO_4$ was added, and the mixture was gently boiled until dense white fumes appeared. Later the mixture was allowed to cool and 10ml of deionized water was added followed by further boiling until the fumes were totally

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www.scienceworldjournal.org
ISSN 1597-6343
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released. Finally, the sample was taken for AAS. The result is shown in figures 2-4.
Similar procedure was adopted for the preparation of the complexes for AAS as shown in figure 5-10.

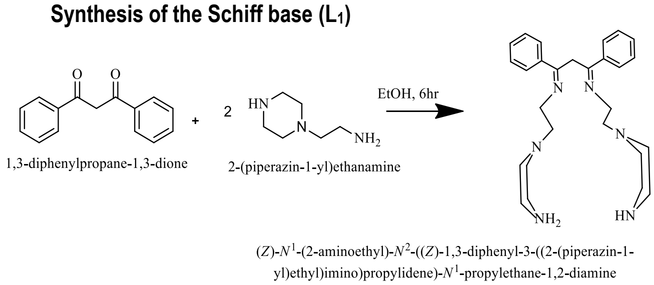
Removal of the Heavy Metals by the Ligands
The metal was extracted by mixing the hot methanolic solution of the ligand with the wastewater, and refluxed for 3 hours. The heavy metals present in the wastewater were detected using AAS.

Procedure
100cm^{3}of the wastewater containing the heavy metals obtained from Youth Craft Village (YCV) was enriched to 50cm^3 and agitated vigorously with 1.0g of the ligand (s), in a stoppered glass conical flask, which was placed on a mechanical shaker for 30min, the mixture was stirred for 2 hours, and then refluxed by dissolving in ethanol-water mixture (40:60) at 80^{o}C ($\underline{+}$5) for 6 hours, and finally left standing for an additional 2 hours. Upon cooling, a colored compound was precipitated, filtered, and dried under vacuum over P_4O_{10}. The concentration of the heavy metals in the compounds were determined using AAS. The procedure was repeated with two other forms of waste water obtained from Lambun Dan Lawal (LDL), and Kofar Sauri (K/sauri) irrigation sites. (Hussein, 2014)

RESULTS AND DISCUSSION

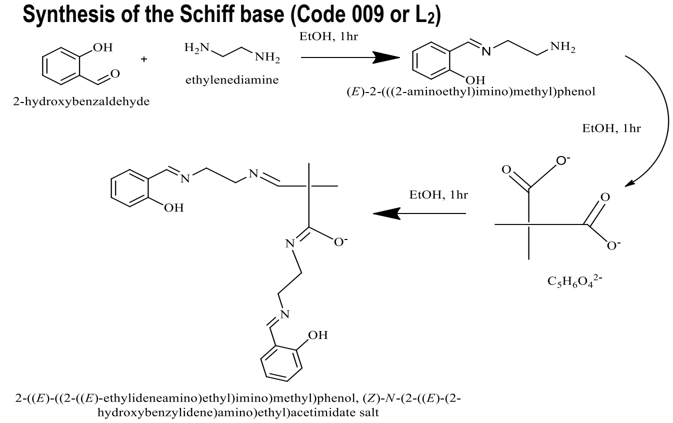
RESULTS

Synthesis of the Schiff base (L$_1$)



Scheme 2: Condensation of 1,3-diphenylpropane-1,3-dione and 2-(piperazin-1-yl)ethanamine

Synthesis of the Schiff base (Code 009 or L$_2$)



Scheme 3: Condensation 2-hydroxybenzaldehyde and ethylenediamine

Note
Both L$_1$ and L$_2$ indicates from the structure, a capability of adsorbing more than one metal, they have more than one metal centers.

Table 2: Wastewater Physical Analysis and the Heavy Metals Present

S/N	Wastewater samples	Color	pH	Heavy Metals Present
1	Kofar Sauri (K/sauri)	Colorless	6.8	Cu, Fe, Co, Cd and Pb
2	Youth Craft Village (YCV)	Dark Purple	6.6	Cu, Fe, Co, Cd and Pb
3	Lambun Dan Lawal (LDL)	Colorless	6.8	Cu, Fe, Co, Cd and Pb

The wastewater samples were obtained from three selected areas within Katsina Local Government Area of Katsina State, as shown by the map in Figure 1. This research was motivated by the regular irrigation and other agricultural activities occurring within K/Sauri and Lambun Dan-lawal, and it serves as a source of vegetables to the people of Katsina LGA. . However, the sewage from Youth Craft Village, industrial activities are taking place around the area, which might be the reason for its pollution. The wastewater has a dirty purple color, and the pH shows a low acidity, with a range of 6.6-6.8, which might be due to the presence of heavy metals, which are Lewis acids, and above permissible limits. The targeted heavy metal ions were Cu, Fe, Co, Cd and Pb, and were proved by AAS.

Table 3: UV-Visible $\lambda_{max\ (nm)}$ of the wastewater and their electronic transitions

S/N	Wastewater	$\lambda_{max(nm)}$	Electronic Transition
1	K/Sauri	250	$n\rightarrow\sigma^*$
2	YCV	250	$n\rightarrow\sigma^*$
3	LDL	200	$n\rightarrow\sigma^*$

Key
K/Sauri = Kofar Sauri wastewater sample, YCV = Youth Craft Village wastewater sample, LDL = Lambun Dan-Lawal

Any wastewater is a combination of different mixtures, supplied to the water bodies from several channels, due to anthropogenic activities. The wavelength of maximum absorption was recorded as shown in table 3. 250nm for the wastewater obtained from K/Sauri and YCV, each which can be attributed to n→σ* electronic transition. Similarly, the wastewater from LDL shows λ_{max} at 200nm which can also be attributed to n→σ* electronic transition, due to the Presence of non-bonding and anti-bonding energy levels available in the structural orientation of the water molecule. There is also possibility of presence of conjugated molecules, in the wastewater, though AAS proves only the presence of the targeted heavy metals namely (Cu, Fe, Co, Cd and Pb). The ions and other molecules in the wastewater were assumed to be in a very high energy, according to Einstein equation, $E = hc/\lambda$, which proves that; energy is inversely proportional to wavelength, and getting a smaller wavelength proves that; the ions are moving in a very high energy.

Table 4: Physical data of the synthesized ligands and their complexes

S/N	COMPOUND	M.P/D.T ^{0}C	CONDUCTIVITY $ohm^{-1}cm^{2}mole^{-1}$	PHYSICAL APPEARANCE	
				STATE	COLOUR
1	L_1	87	-	Solid	Yellow
2	L_1 + K/sauri	182	92	Solid	Pale Red
3	L_1 + YCV	346	82	Solid	Grey
4	L_1 + LDL	160	146	Solid	Pale Red
5	L_2	126	-	Solid	Yellow
6	L_2 + K/sauri	150	97	Solid	Greenish Yellow
7	L_2 + YCV	165	44	Solid	Army green
8	L_2 + LDL	150	103	Solid	Golden

Key
$L_1 = C_{28}H_{42}N_6$, $L_2 = C_{22}H_{27}N_4O_3^-$, K/Sauri = Kofar Sauri wastewater sample, YCV = Youth Craft Village wastewater sample

LDL = Lambun Dan-Lawal, M.P = Melting Point, D.T = Decomposition Temperature

The compounds were all solids, and the ligands appeared yellow in color, which may be assign to the azomethene bond (C=N). The color transition from yellow to pale red and brown for L_1 complexes, after refluxing with the variable wastewater, indicates complexation, or formation of M-N bond. Similar result were obtained with L_2, when it changes from yellow to greenish yellow, army green and golden, just as shown in table 4. The compounds showed higher thermal stabilities due to higher decomposition temperatures recorded, indicated in table 4. The complexes give decomposition temperature range of 150-340^{0}C, a value higher than the melting points of their corresponding ligands. One compound from ligand 1 and two from ligand two had appeared to be electrolytic in nature, by falling within the range of 96-150, and the rest are non-electrolytic as displayed by Table 4.

Table 5: Infrared Absorption Frequencies (cm^{-1}) of the Ligands and their Metal Complexes

S/N	COMPOUNDS	υ (C=N)	υ (M-N)
1	L_1	1595	______
2	L_1 + K/sauri	1525	440
3	L_1 + YCV	1551	447
4	L_1 + LDL	1555	453
5	L_2	1633	______
6	L_2 + K/sauri	1599	440
7	L_2 + YCV	1599	451
8	L_2 + LDL	1603	447

Key
$L_1 = C_{28}H_{42}N_6$, $L_2 = C_{22}H_{27}N_4O_3^-$, K/Sauri = Kofar Sauri wastewater sample, YCV = Youth Craft Village wastewater sample
LDL = Lambun Dan-Lawal

The infrared spectroscopic data indicates the formation of azomethene bond in the Schiff bases (L_1 and L_2) spectra, which are in good agreement with reported literatures. It also shows the complexation, due to the appearance of M-N bond within the range of 440-453 as shown in the table 5. This was further supported by a down field shift observed, in the spectra of the complexes when compared with that of the azomethine bond, after the reflux reaction, as reported by Muaiad, 2011; Malghe *et al.,* 2009 and Shaker *et al.,* 2009. Ilhan *et al.,* 2007 reported the absence of υ(C=O) at 1700cm^{-1} to be an indication of the Schiff base condensation.

Table 6: UV-Visible λ_{max} (nm) of the Compounds and their electronic transitions

S/N	COMPOUNDS	$\lambda_{max(nm)}$	Electronic Transition
1	L_1	400	n→π*, π→π*
2	L_1 + K/sauri	500	π→π*
3	L_1 + YCV	450	π→π*
4	L_1 + LDL	550	π→π*
5	L_2	450	n→π*, π→π*
6	L_2 + K/sauri	450	π→π*
7	L_2 + YCV	400	π→π*
8	L_2 + LDL	500	π→π*

Key
$L_1 = C_{28}H_{42}N_6$, $L_2 = C_{22}H_{27}N_4O_3^-$, K/Sauri = Kofar Sauri wastewater sample, YCV = Youth Craft Village wastewater sample
LDL = Lambun Dan-Lawal

Science World Journal Vol 13(No 4) 2018
www.scienceworldjournal.org
ISSN 1597-6343
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Ahmed *et al.,* 2014 reported The UV-Vis spectra of the ligands exhibit an intense absorptions 263-270 nm and 314-376 nm, which is attributed to n→ π* and π→ π*, electronic transition respectively. The table 6 above shows the UV-visible wavelength of maximum absorption, (λ_{max}) of the Schiff bases (400 and 450nm), which signifies n→ π* electronic transition, that can be ascribed to the presence of lone pairs of electrons on the nitrogen atom of the Schiff bases and C=N. while π→ π* is also possible being it narrower than σ to σ* transition. Muaiad reported that π→ π* electronic transition can be found at 328 while n→ π* electronic transition can be located at 380nm. The complexes appeared to be chromophic, with a λ_{max} range (400-550nm) signifying a π→ π* electronic transition due to the presence of π-bonds. The absorption at 550nm was due to d-d electronic transition as contained in table 5. Moreover, conjugated π bond system is expected to become larger, when the energy gap for π→ π* transition becomes narrower, and the light absorption correspondingly becomes longer. In each case incoming photons match in energy gap, corresponding to a transition from ground state to excited state.

Table 7: Solubility test of the ligands and their metal complexes

S/N	Compound	DMSO	DMF	MeOH	EtOH	Acetone	Chloroform	Diethyl Ether	Benzene	n-hexane
1	L_1	S	S	S	S	S	SS	IS	IS	IS
2	L_1 + K/sauri	S	SS	SS	S	SS	IS	IS	IS	IS
3	L_1 + YCV	S	S	SS	S	S	IS	IS	IS	IS
4	L_1 + LDL	S	SS	S	S	S	SS	IS	IS	IS
5	L_2	S	SS	S	S	S	SS	IS	IS	IS
6	L_2 + K/sauri	S	S	S	S	S	IS	IS	IS	IS
7	L_2 + YCV	S	S	S	S	S	SS	IS	IS	IS
8	L_2 + LDL	S	S	S	S	SS	SS	IS	IS	IS

Key
$L_1 = C_{28}H_{42}N_6$, $L_2 = C_{22}H_{27}N_4O_3^-$,K/Sauri = Kofar Sauri wastewater sample, YCV = Youth Craft Village wastewater sample
LDL = Lambun Dan-Lawal

The compounds were found to be soluble in DMSO possibly due to its high dielectric constant. Also, the compounds were soluble in ethanol, while variable behavior were recorded in methanol, DMF, acetone and chloroform. However, the compounds were insoluble in the rest of the solvents used, as shown by table 7.

AAS RESULTS

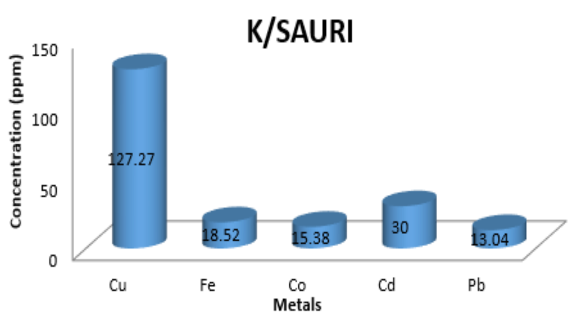

Figure 2: Kofar Sauri Wastewater

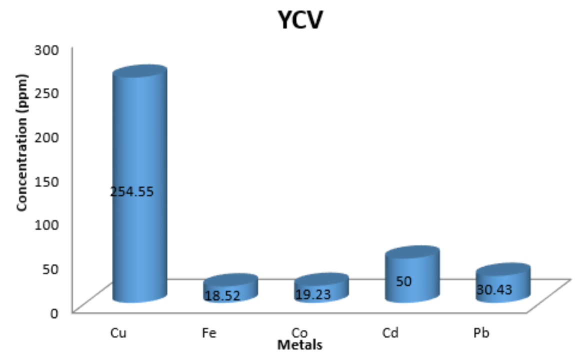

Figure 3: Youth Craft Village Wastewater

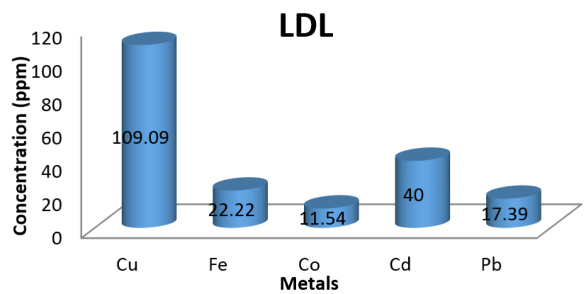

Figure 4: Lambun Dan Lawal Wastewater

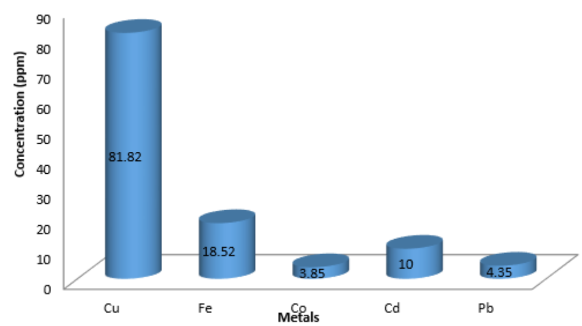

Figure 5: L_1 + K/sauri wastewater sample

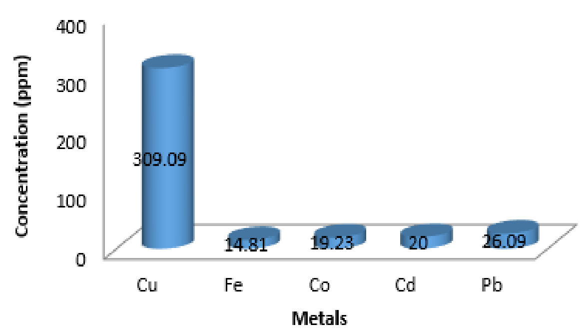

Figure 6: L_1 + LDL wastewater sample

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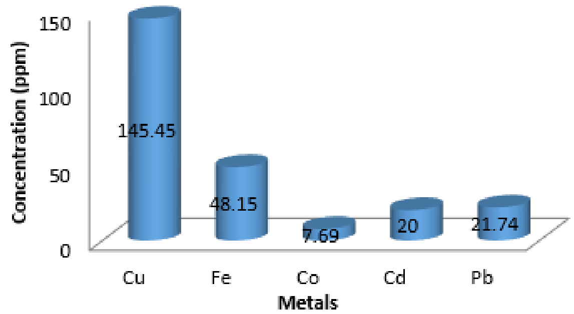

Figure 7: L_1 + YCV wastewater sample

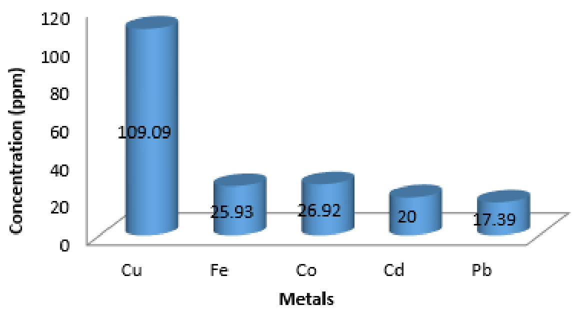

Figure 8: L_2 + K/sauri wastewater sample

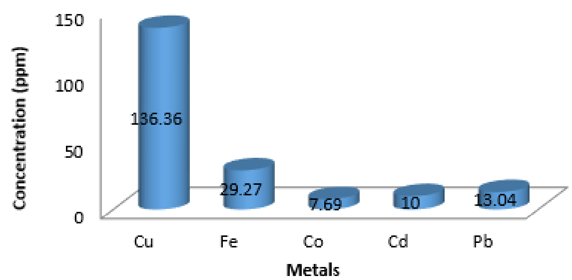

Figure 9: L_2 + LDL wastewater

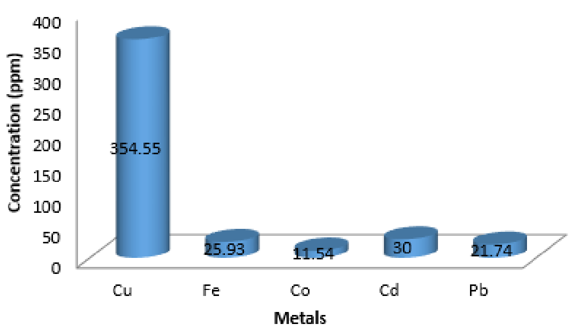

Figure 10: L_2 + YCV wastewater sample

Atomic absorption spectroscopic technique was employed, to detect the presence of the targeted heavy metals (Cu, Fe, Co, Cd and Pb), after the reflux reaction, as shown by the figures 5-10 above. The Schiff bases shows remarkable character; by adsorbing/reacting with all the heavy metals at variable concentration. In each case Cu was found to have higher concentration both in the wastewater, and in the compounds after the reaction.

Conclusion
Schiff bases were reported to have shown, remarkable affinity towards heavy metals in aqueous phase. This paper reported, non-cyclic Schiff bases were found to have demonstrated a characteristic of removing heavy metals from the wastewater, the removing capacity of the two types/kind of Schiff bases manifested that; the non-cyclic Schiff bases can be used in heavy metals removal from wastewater.

Recommendations
There is a need for further investigation of the compounds with characterizations such as ^{1}HNMR, ^{13}CNMR, Crystallography and Mass spectroscopic analysis to reveal the actual molecular structure of the compounds.

Acknowledgement
The contribution made to this research by the TETFUND and Umaru Musa Yaradua University, is highly acknowledged.

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