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Adsorption studies of Fe (II) Co (II) and Ni (II) ions using a Salicylaldehyde -3- amino benzoicAcid Schiff Base ligand

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ABSTRACT
The presence of heavy metals in water, even at permissible concentrations poses significant risk to human and animal health. Consequently, their removal from water sources is very vital for the safety of consumers. Adsorption techniques are increasingly favored due to their design simplicity and availability of effective adsorbents. In this study, Schiff base ligand derived from salicylaldehyde and 3-aminobenzoic acid was evaluated as an adsorbent for the removal of Fe (II), Co (II), and Ni (II) ions from aqueous solutions. Characterization of the ligand before and after the adsorption wasconducted using Fourier transform infrared spectrophotometer (FT-IR), highlighting azomethine (C=N) and hydroxyl (–OH) functional groups as key active sites. Batch adsorption experiments assessed the effect of pH, initial metal ion concentration, contact time, and adsorbent dosage. Optimal removal occurred at pH 6 after 2 hours, of contact time. Kinetic data fitted well to the Pseudo- second order model, indicating chemisorption as rate- limiting step. Adsorption isotherm analysis showed a better fit to the Langmuir model, with correlation coefficient (R^2 0.997, for Fe (II), 0.99 for Co (II), and 0.978 for Ni (II), suggesting monolayer adsorption. Overall, the Schiff base ligand demonstrated significant potential as an efficient and low-cost adsorbent for heavy metal removal from aqueous environments.

Keywords: Schiff base, Adsorption, Fe(II), Co (II), Ni (II),Isotherm, Kinetics.

I. INTRODUCTION

Contamination of water sources with heavy metals has emerged as a critical environmental and public concern. Industrialization, urbanization, and various anthropogenic activities have exacerbated the release of toxic metals into rivers, lakes, and ground water. Chronic exposure to heavy metals such as iron (Fe), cobalt (Co), and nickel (Ni)even at low concentrations can lead to severe health complications including kidney dysfunction, neurotoxicity, immune suppression, developmental abnormalities and carcinogenesis, (Fernandeset al., 2012, Cobbinaet al .,2015,Costa et al., 2019 Gwaziet al., 2020).

Concentration of heavy of metals in water sources, air, and food is assessed with this regard (Mousavitet al., 2013), Ghonia-Azamet al., 2020). Metals among other environmental pollutants may also occur naturally and remain in the environment, hence human exposure to these metals is inevitable, and some studies have reported gender differences in the toxicity of metals (Vahteret al.,2007); Tchounwouet al., 2012).Traditional water treatment methods including chemical precipitation, coagulation-flocculation, ion exchange, membrane filtration, and activated carbon adsorption are often limited by high operational costs and inefficiency at trace concentration levels. Consequently, there is a growing interest in developing alternative low-cost and environmentally adsorbents.Schiff base ligand have attracted attention in recent years due to their facile synthesis, structural versatility, and the ability to chelate metal ions effectively through donor atoms such as nitrogen and oxygen. These ligands are also recognized for their biological activities and catalytic properties. Despite extensive research on Schiff base metal complexes, their application on in water purification, particularly for the removal of Fe (II), Co (II), and Ni (II), remains underexplored.

This study focuses on the synthesis of a Schiff base ligand derived from salicyaldehyde and 3-aminobenzoic acid, and its evaluation as an adsorbent for the removal ofFe (II), Co (II), and Ni

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(II) ions from aqueous solutions. The effects of pH, initial concentration, contact time, and adsorbent dosage on metal ions investigated. Furthermore, adsorption kinetics and isotherm models were applied to elucidate the adsorption mechanism and capacity of the Schiff base ligand.

II. MATERIALS AND METHODS

Reagents

All reagents and solvents wereanalytical grade and used without further purification. Salicylaldehydeand 3-aminobenzoic acid were purchased from Sure chem product (CSP) and Lobachem PVT respectively .Sodium hydroxide, hydrochloric acid, methanol and chloroform were obtained from Sigma Aldrich. Dimethylsulphoxide and dimethylformamide were purchased from Heiltrofen and Eastman chemical respectively .The hydrated salts of the metals used such as $Nicl_2.6H_2O$, was purchased from Guanghua China while $Fecl_2.4H_2O$ and $Cocl_2.6H_2O$ were obtained from Griffin and George.

Apparatus

Beakers, conical flasks, test tubes, 50ml measuring cylinder, round button flask,retort stand ,refluxing condenser , filter paper, funnel , Fourier transform infrared spectroscopy (FTIR) 408 Shimdzu, Atomic absorption spectroscopy (AAS)ZEEnit- model 650P and magnetic stirrer.

Synthesis of Schiff's Base

Salicylaldehyde (1.22g, 0.01mol) was added to a solution of 3-amino benzoic acid (1.40g, 0.01mol) in 20ml of ethanol, and the mixture was stirred constantly with a magnetic stirrer and refluxed at 80^0C for 2 hours. Upon cooling, the precipitate obtained (yellow color compound) was washed several time with ethanol and dried atroom temperature.

Equation of the reaction

Aldehyde + amine → SL + H_2O 1

Where SL = Schiff base ligand

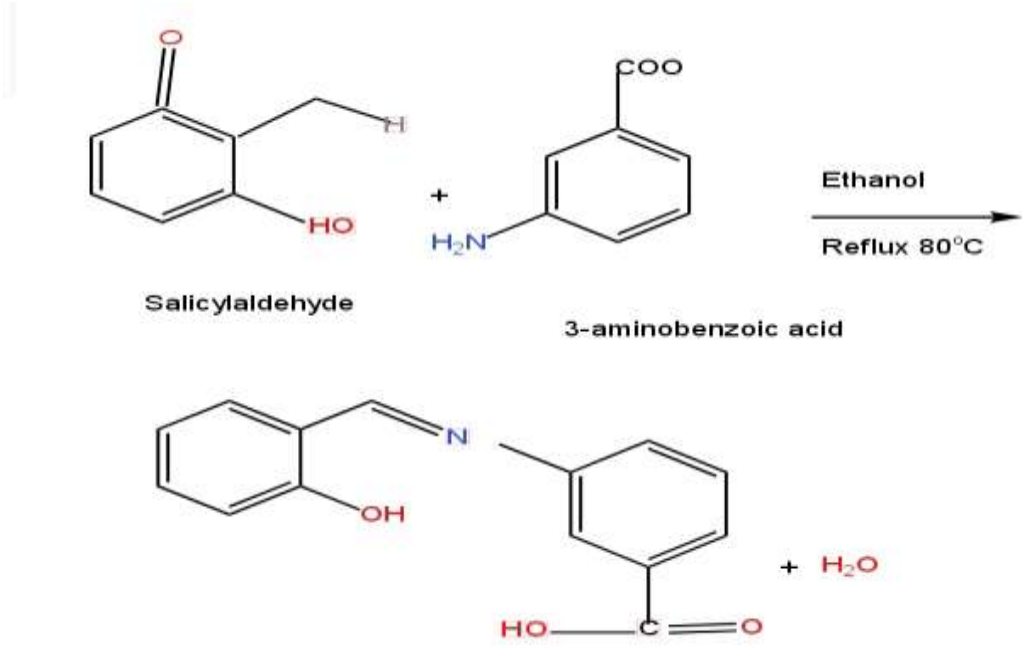

Fig. 1 proposed structure of Schiff base (SL)

Preparation of Standard Solution

A stock solution was first prepared by dissolving 35.60, 40.37, and 40.50 grams of metal salts of $Fecl_2.4H_2O$, $Cocl_2.6H_2O$, and$Nicl_2.6H_2O$ respectively, in distilled water using 250ml volumetric flask to prepare 100mg/L stock solution. The working solutions of 10, 20,30,40,50, and 60mg/L were obtained by dilution of the stock solution to the required concentration using dilution formula C_1 V_1 = C_2 V_2 Ibrahim jimoh (2021).

Where, C_1 = concentration of prepared stock solution, V_1 = initial volume of stock solution needed to prepare standard solution, C_2 = final concentration required and V_2 = final volume.

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Thus, a sample of each standard solution prepared was taken for AAS.

Effect of pH

The effect of pH on the adsorption of metal ions by the adsorbent was investigated at room temperature, at different pH ranges from 1-6 to determine the effect of initial pH on the adsorption of the metal ions. 0.1g of dry Schiff base ligand were added to 15ml of each metal ion standard solution and kept in a mechanical shaker at 150rpm for 2 hours. The filtrate obtained was subjected to analysis for residual metal ion concentration by AAS.

Contact Time

Both the amount of Schiff base used (0.1g) and 15ml solutions of the metal ions was kept constant. Adsorption of metal ions by the adsorbent were studied in batches at various time intervals(20-120mins) at the concentration of 10mg/L. 0.1g of the ligand was weighed in to 15ml of solutions of metal ions and placed in a glass Erlenmeyer flask and kept in a mechanical shaker at different time intervals 20,40,60,80,100,120mins for adsorption. After shaken the samples, each was filtrated one after the other and the filtrate was analyzed using AAS to determine the final concentration.

Effect of Initial concentration

0.1g of the Ligand was shaken in to 15ml of metal ions solutions of various concentration (10, 20,30,40,50 and 60mg/L) at constant speed of 150rpm, for 2 hours under optimized pH condition. The filtrate obtained was analyzed using AAS.

Adsorbent Dosage

The measurement of the metal ion uptake using batch method was performed by placing different weight of ligand 0.1, 0.25, 0.5, 0.8, 1, 1.5g in to 15ml solutions of each metal ions in a stopper flasks at optimum pH within 2 hours .The amount of metal ions adsorbed was calculated based on the differences of the concentrations in aqueous solutions before and after the adsorption.

Batch Adsorption Experiment

Batch method were carried out with 100ml of working adsorbates solutions (Fe (II), Co (II) and Ni (II)) ions at different concentrations ranging from 10-60mg/L. 0.1g was added to the solutions. The flasks were agitated at a constant speed of 250rpm for 120min in a mechanical shaker. This was carried out for kinetic and isotherm studies.

III. RESULTS AND DISCUSSIONS

After following the standard procedure,a light yellow colored compound was isolated in a good yield as 41%.

Table 1. Summary of FT-IR result of the ligand before and after adsorption.

Compound	v(C=N)cm^{-1}	v(O-H) cm^{-1}	v(M-O) cm^{-1}	v(M-N) cm^{-1}
Salicylaldehyde-3-aminobenzoic acid Schiff base	1677.99	3456.33	-	-
After adsorption with Ni	1677.51	3449.63	676.81	488.51
After adsorption with Co	1608.71	3328.51	680.62	418.51
After adsorption with Fe	1627.58	3443.27	674.44	444.60

The ligand was characterized using FT-IR spectrophotometer in the region 4000-500cm^{-1}to determine the main functional groups responsible for the Schiff base formation and other important functional groups available. The vibration of imine (C=N) group were observed at 1677.99cm^{-1}indicating that the Schiff base was formed from Salicylaldehyde and 3-amino benzoic acid. This is in line with (Shamply.pet al., 2018) The IR spectra of the ligand exhibited a broad band of stretching frequency of OH group at 3456.33cm^{-1}. These bands slightly shifted to the lower frequency after adsorption. The IR spectral bands for v(C=N) stretching vibration of the ligand after adsorption was observed at 1677.5cm^{-1}, 1608.71cm^{-1}, and 1627.58cm^{-1} for Ni-L, Co-L and Fe-L respectively, (Narendra Kumar 2018). In fact, in all the complexes, the absorption band for v(C=N) stretch has shifted to lower wave numbers, indicating their participation in the coordination with the metal ions. The IR absorption frequency at 3456.33cm^{-1} due to (OH) stretching in ligand has undergone a

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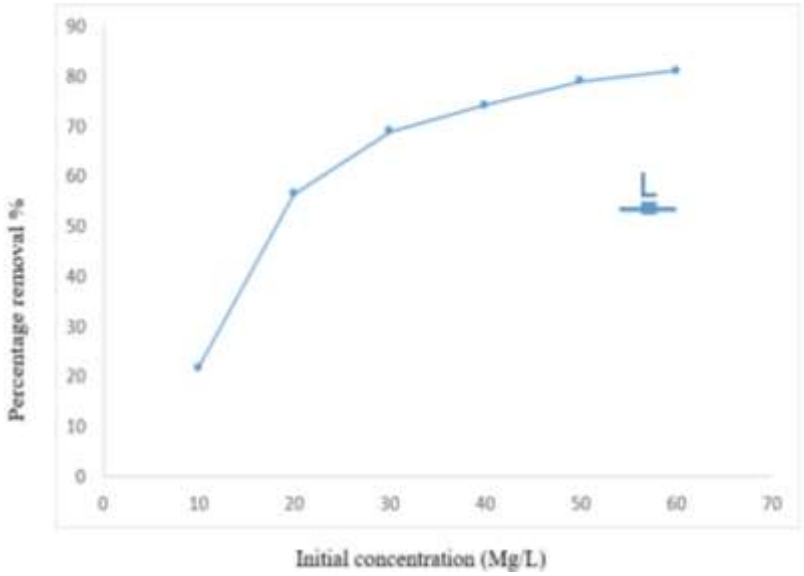
shift at 3449.63cm^{-1}, 3328.51cm^{-1},3443.27 corresponding for Ni- L, Co –L, and Fe –L respectively. The shifting of bands in the compounds as compared to the free ligand, is due to reduction of double bond character of the carbon-nitrogen bond of azomethine group, Abdunnasir. Etokiet al., (2012). Another band also appeared in the region 650-680 cm^{-1} and 440-490 cm^{-1} which is attributed to v (M-O) and v (M-N) stretching vibrations respectively, conforming the coordination of Schiff base to metal ions. These bands are absent in the IR spectra of the free ligand.

Effects of Initial Concentration on the Adsorption of the Metals Ions

The percentage removal of Fe^{2+} by the ligand increased with increasing initial concentration and slow down when the concentration reached 30 mg/L. Thepercentage removal of Ni^{2+} and Co^{2+} decreased as the initial concentration increases. As can be seen in Figure 2a, the adsorption in the beginning is zero, and increases gradually with respect to the contact time due to the large number of vacant active sites between the adsorbate surface and adsorbatein suspension. With proceeding time, there is a reduction in concentration due to the accumulation of metal concentration on vacant sites of the adsorbent and hence the gradient of adsorption rate decreases when the concentration reached 30 mg/L. Asconcentration of Ni (II) ions increases, the binding sites becomes more quickly saturated as the amount of ligand concentration remain constant, this corresponds to the findings of (Rehamet.,al 2013) and the amount of metal ions removal decreased with increase in initial concentration. Fig. 2c showed that at low concentration of Co (II) ion, there are sufficient binding **s**ite**s** and the adsorption process is controlled by ion diffusion (Noor Raid et al., 2021) while at higher concentration, the availability of the binding sites decrease until the binding sites are almost saturated and the adsorption is controlled by the adsorbent dosage. For this reason, at a concentration higher than 10 mg/L, Co (II) removal rate decreased.



(a) Fe (II). Figure (a) shows the effect of initial concentration for the adsorption of Fe (II) by the ligand.

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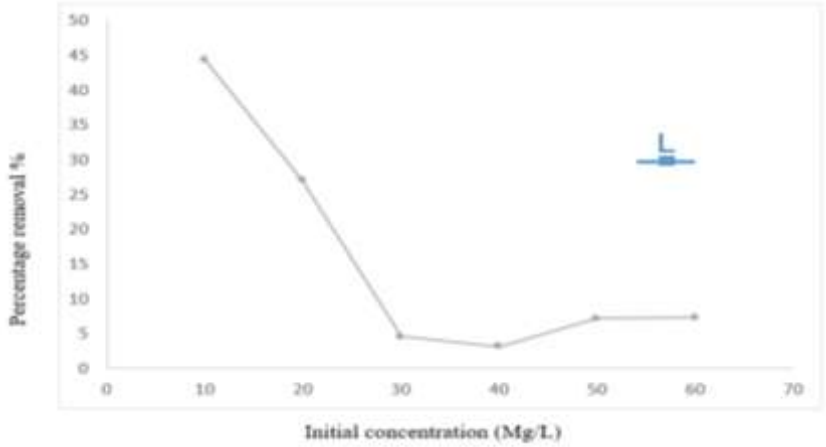
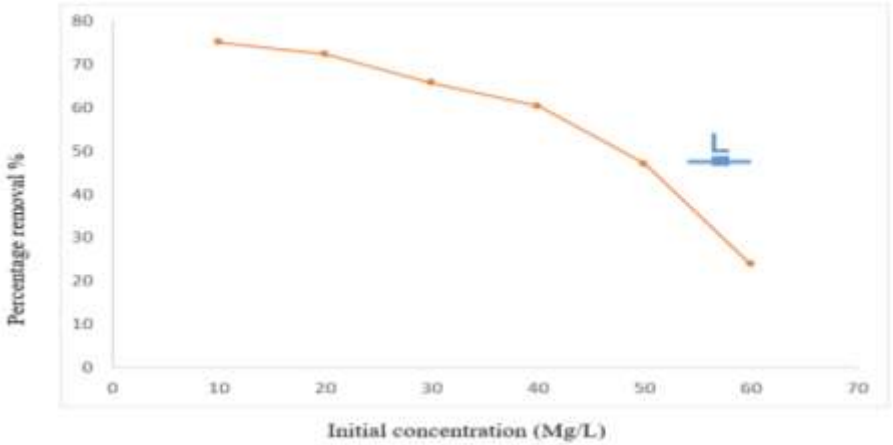


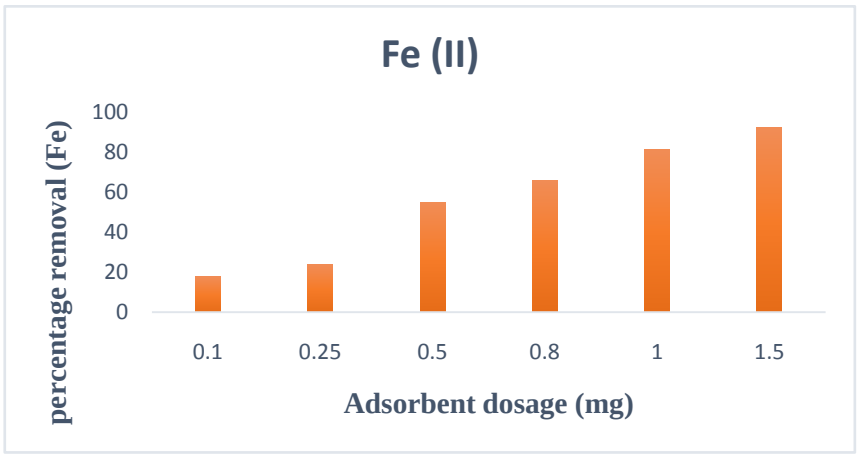
(b)Ni (II). Figure (b) shows effect of initial concentration for the adsorption Ni (II) by the ligand



(c) Co (II). Figure (c) Indicates the effect initial concentration for Co (II) by the ligand.

Figure 2.Adsorption of the metal ions with variation in initial concentration by the adsorbent.

Effect of Adsorbent dosage, Contact time and pH



(d) Fe (II) Figure (a) shows the effect of the adsorbent dosage for the adsorption of Fe (II)

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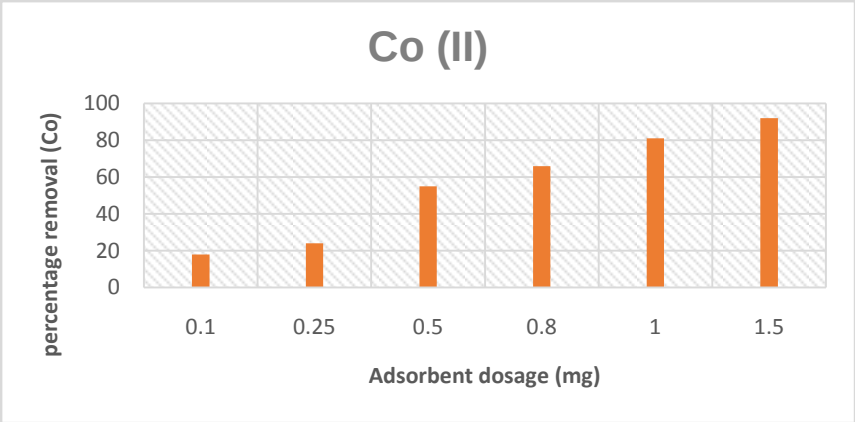
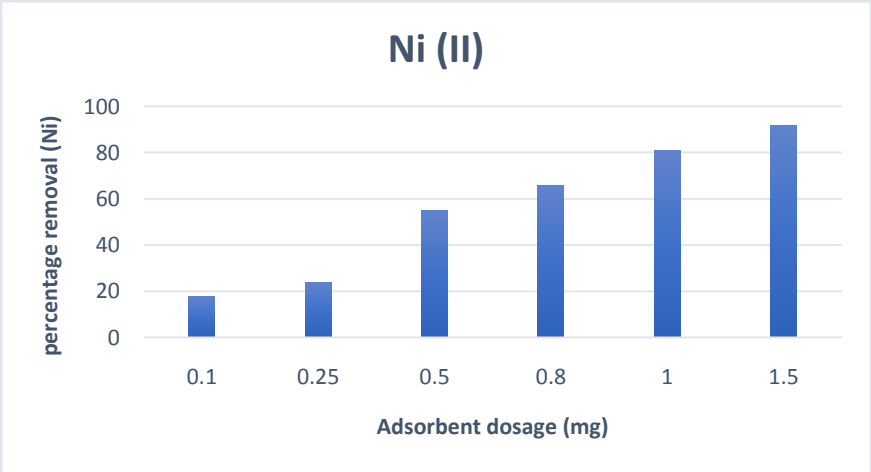
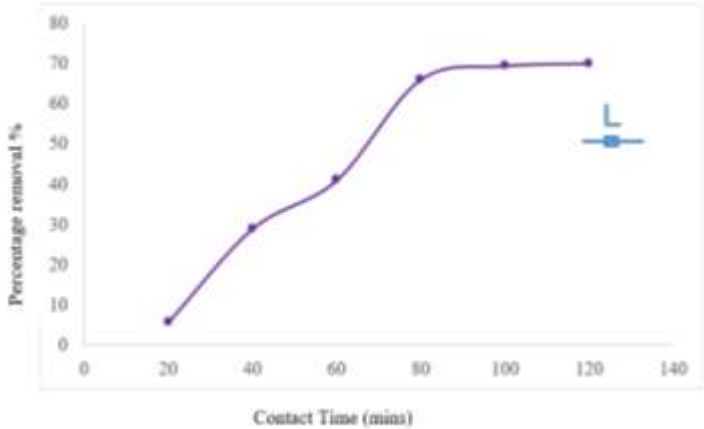


(e) Co (II) Figure (e)shows the effect of the adsorbent dosage for the adsorption of Co (II)



(f) Ni (II) Figure (f) shows the effect adsorbent dosage for the adsorption of Ni (II)

Figure 3 (d – f). Adsorption of the metals ions with variation in adsorbent dosage by the adsorbent.



(g)Fe (II) Figure (g) showsthe effect of contact time for the adsorption of Fe (II)

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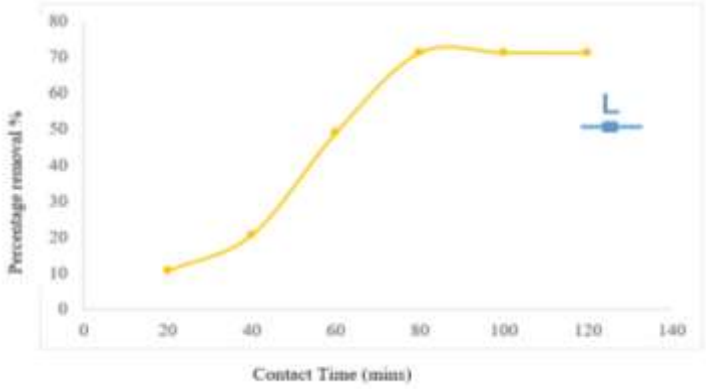
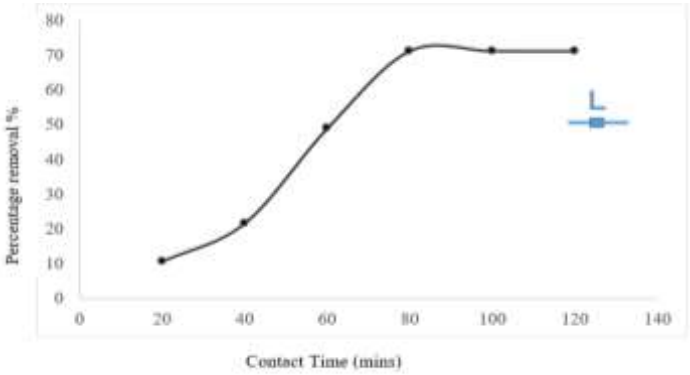
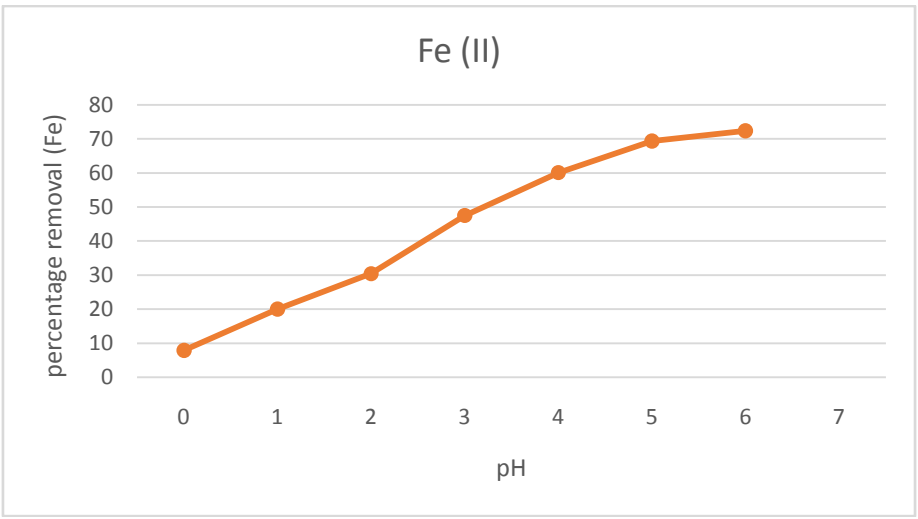


(h) Co (II) Figure (h) showsthe effect of contact time for the adsorption ofCo (II)



(i) Ni (II) Figure (i) showsthe effect of contact time for the adsorption ofNi (II)

Figure 4 (g – i). Adsorption of the metals ions with variation in contact time by the adsorbent.



(j) Fe (II). Figure(j)showsthe effect of pH for the adsorption of Fe (II**)**

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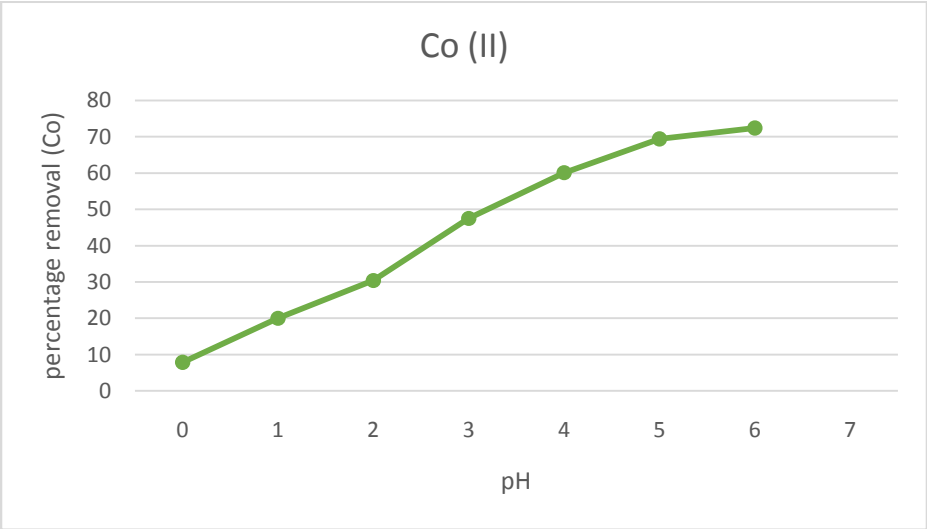
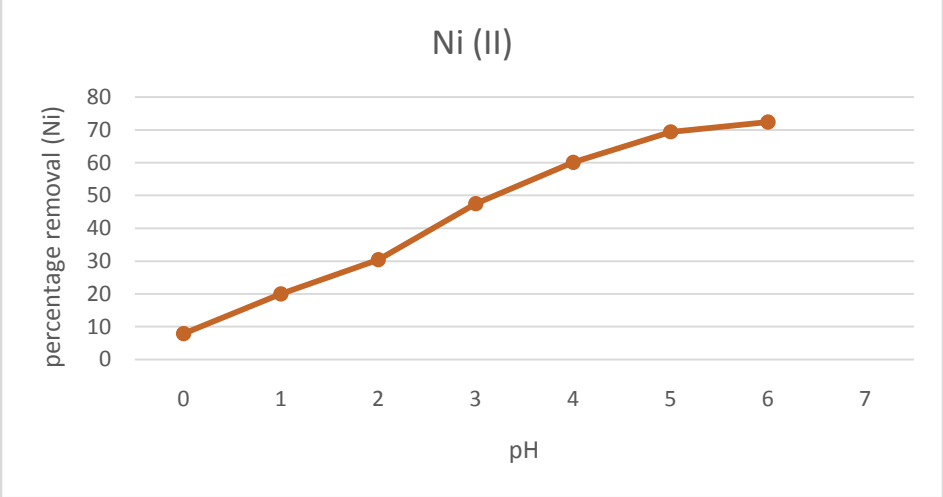


(k) Co (II). Figure(k)showsthe effect of pH for the adsorption of Co (II)



(l) Ni (II). Figure(l)showsthe effect of pH for the adsorption ofNi (II)

Figure 5 (j– l). Adsorption of the metals ions with variation in pH by the adsorbent.

The percentage removal of the metal ions increases with increase in adsorbent dosage, and the optimum dosage of the Schiff base for the metal ion removal was 1.5g. This is attributed to the fact that at high adsorbent dosage, available metal ions are sufficient to cover all the exchangeable sites on the adsorbent.Any further of higher concentration of adsorbent beyond 1.5mg/L would not cause any significant change in the adsorption due to the overlapping adsorption sites of adsorbent particles. Moreover, the adsorption efficiency of Fe^{2+}, Co^{2+}, and Ni^{2+} was evaluated as a function of contact time and result showed that the percentage removal was found to be higher after 80 minutes and then increases slowly until it reach saturation level after 2 hours indicating equilibrium point.The nature of adsorbent and its available sorption sites affected the time needed to reach equilibrium.The pH of the metal solution is the most important parameter governing metal adsorption due to the fact that pHof solution affects the solubility of metal ions, and the surface charge of the adsorbent. Therefore, as pH of the solution increases, the percentage removal of metal ions increases .At pH 6 the, percentage removal of Fe (II), Co (II), and Ni (II) were 80.46, 89.30, and 80.46% respectively.

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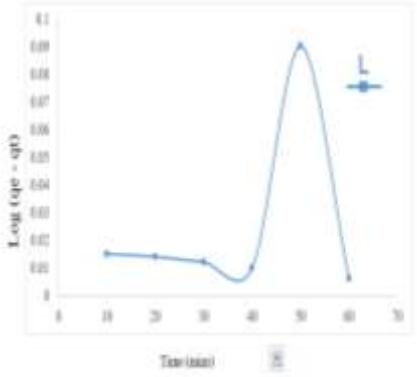
Table 2 The Optimum levels of adsorption for the factors

Metals	Concentration(mg/L)	% removal	pH	% Removal	Contact time(Min)	% Removal	Adsorbent dosage (g)	% Removal
Fe^{2}	50	49.02	6	80.46	120	69.87	1.5	93.7
Co^{2+},	10	75.04	6	89.30	80	71.11	1.5	92
Ni^{2+}	10	44.35	6	80.46	80	71.10	1.5	90

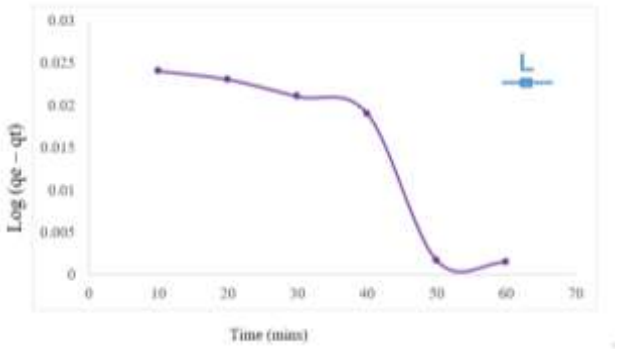
Tale 2 shows the summary of the optimum conditions of the factors for the adsorption of the metal ions by the ligand

Adsorption Kinetic Studies of the Metals Ions by the Adsorbent

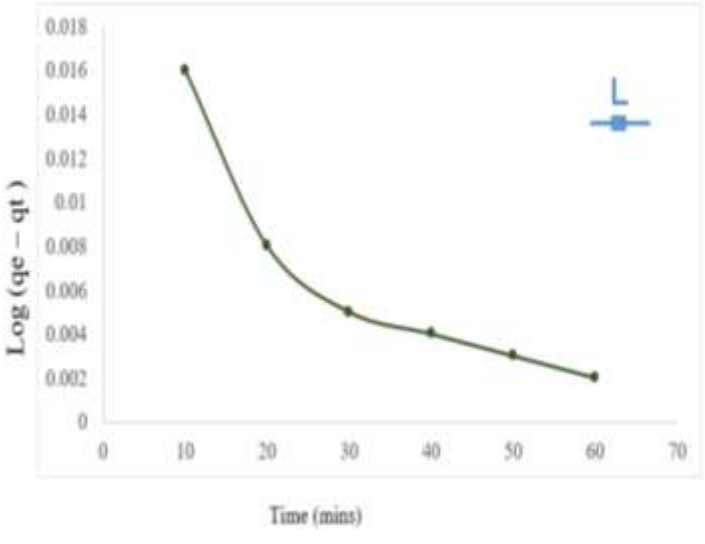
In order to analyze the rate of adsorption and possible adsorption mechanism of Fe^{2+}, Co^{2+}, and Ni^{2+} of the adsorbent, the pseudo- first order and pseudo- second order kinetic models were applied.



Pseudo-first order plot for of Fe (II)



Pseudo-first order plot for of Co (II)



Pseudo-first order plot for of Ni (II)

Fig. 3Pseudo-first order adsorption Kinetic Models of the metals ions on the Adsorbent

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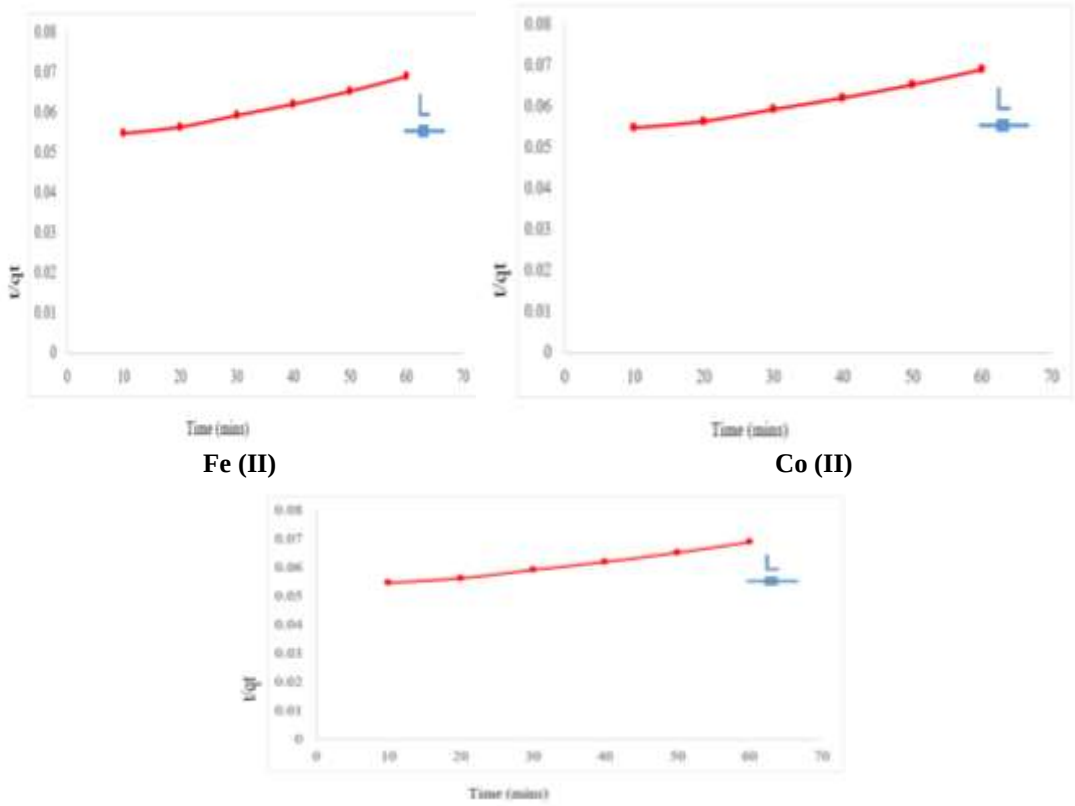

Fe (II) **Co (II)**

Ni (II)

Fig 4.Pseudo- second order adsorption Kinetic Models of the metals ions on the Adsorbent

The two modals used were pseudo-first order and pseudo-second order model (Ho and McKay, 1998). The pseudo- first order model is based on the sorbent capacity and considers that the rate of occupation of adsorption sites is proportional to the number of unoccupied sites. The pseudo-second order equation is also based on the adsorption capacity of the solid phase (Aksu and Tunc 2005). The linearized forms of the pseudo-first order and pseudo- second order are represented by Equations 3 and 4, (Ho and McKay 1998).

$\ln (qe-qt) = \ln qe - K_1 t$ 3

$(\ln /qt) = 1 / (k_2 qe_2) + (t / qe)$ 4

Where qe is the amount of metal adsorbed at equilibrium (mg/g); qt is the amount of metal ions adsorbed at time t (mg/g) k_1 is the rate constant of pseudo- first order adsorption (min^{-1}); k_2 is the rate constant of pseudo-second order adsorption ($g/mg/min^{-1}$). The two models were applied to the adsorption of metals in this study.

Adsorption Isotherm. The distribution of adsorbate from liquid/ suspension to solid phases was described mathematically by models known as isotherms. The coverage type, solid surface heterogeneity / homogeneity and interaction possibility are set of assumptions forthese models. Data were analyzed using Langmuir and Freundlitch isotherm expression in the present study.

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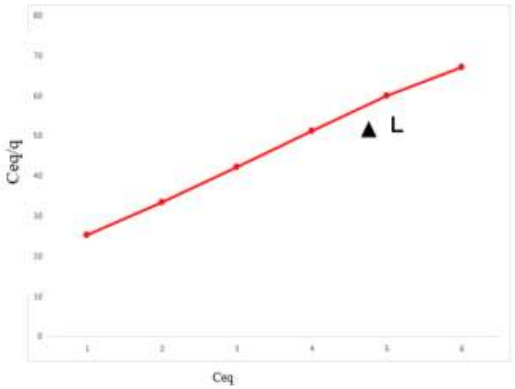
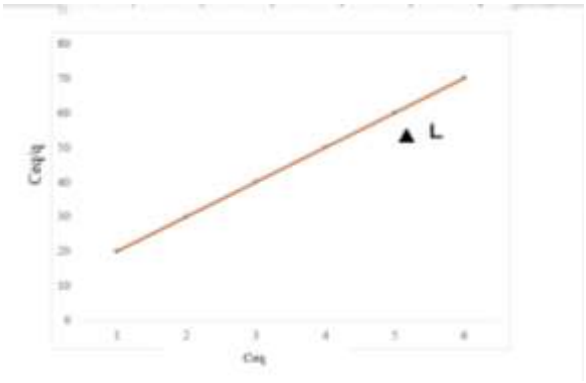
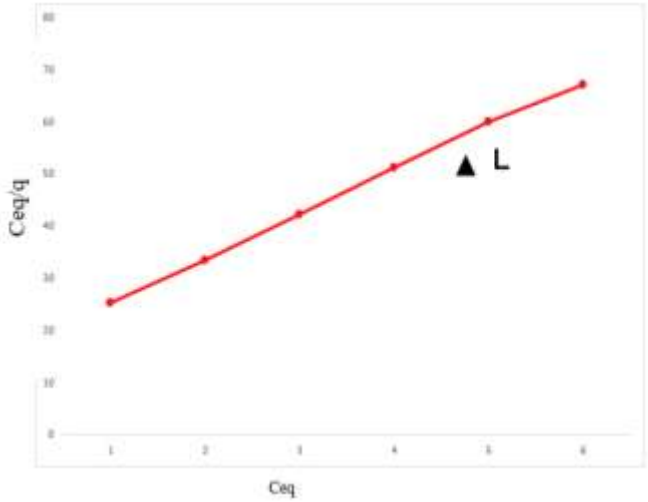


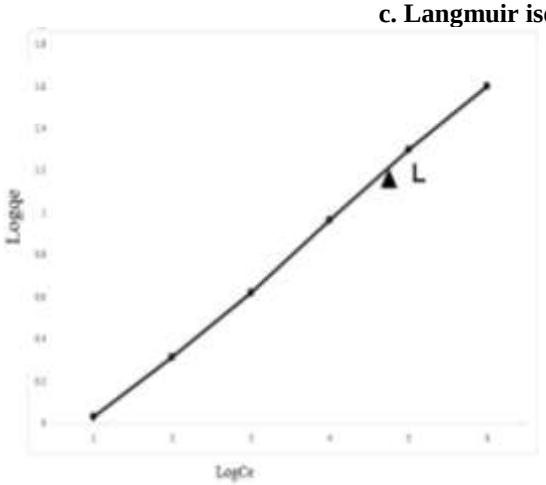
a.Langmuir isotherm Fe (II)



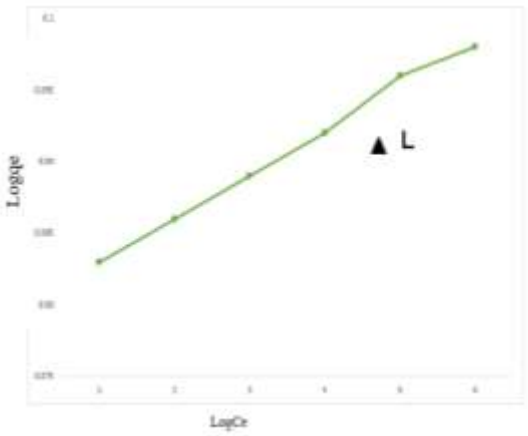
b. Langmuir isotherm Ni (II)



c. Langmuir isotherm Co (II)



d. Freundlitch Isotherm Fe (II)



e. Freundlitch Isotherm Ni (II)

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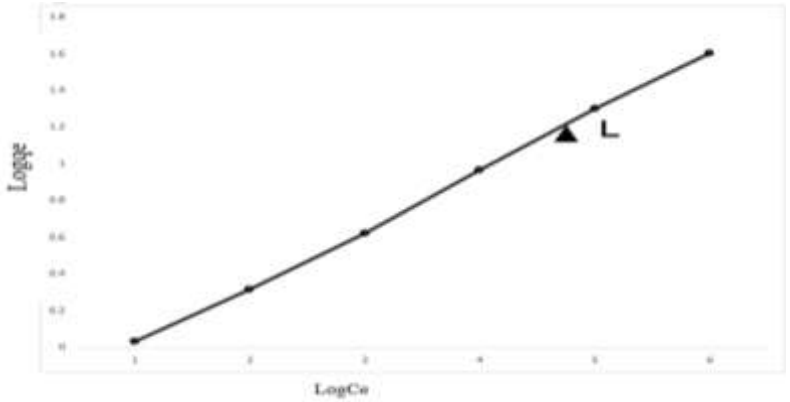

F. Freundlitch Isotherm Co (II)

Fig. 5.Langmuir and Freundlitch models of Fe (II), Ni (II) and Co (II) on the ligand.

Table 3. Summary of Langmuir and Freundlitch isotherms data for the metal ions

Langmuir Isotherm					
Fe^{2+}	R^2	Co^{2+},	R^2	Ni^{2+}.	R^2
	0.964		0.982		0.975
	0.997		0.971		0.978
	0.978		0.947		0.967
	0.991		0.961		0.976
	0.948		0.984		0.978
	0.987		0.99		0.976
Freundlitch isotherm					
Fe^{2+}	R^2	Co^{2+},	R^2	Ni^{2+}.	R^2
	0.516		0.69		0.889
	0.517		0.797		0.872
	0.556		0.870		0.885
0.523		0.821		0.883	
0.543		0.748		0.886	
0.665		0.787		0.876	

Table 3 Shows the variation of the correlation coefficient values for Langmuir and Fruindlitch isotherm models.

Langmuir Isotherm model. The Langmuir isotherm model was tested to estimate the adsorption

Capacity with respect to monolayer coverage or target active sites on the Schiff base ligand. The plots (Ceq/q) versus Ceq for the three metals were used to calculate linear isotherm parameters,qm, K_1 and R^2 values. According to plots, in view of the values of linear regression coefficient (R^2), it is realized that the exhibited Langmuir model fitted well to the adsorption data compared to Freundlutch model for Fe (II), Ni (II) and Co (II).

Freundlich Isotherm. Freundlitch isotherm model interprets the adsorption on heterogeneous surfaces withinteraction occurring between the adsorbed molecules and is not restricted to the formation ofmonolayer. According to plots, in view of the values of linear regression coefficient (R^2), it is realized that the exhibited Freundlitch model was not able to adequately describe the relationship between the amount of the metal ions (Fe (II),Co (II), and Ni (II)) adsorbed by the adsorbent and the equilibrium concentration in the solution.

IV. CONCLUSION

The present study was based on the estimation of feasibility of adsorption of the metal ions,(Fe (II), Co (II), and Ni (II)) using the aqueous solutions of the metals by the Schiff base ligand ;synthesized from salicylaldehyde and 3-aminobenzoic acid. The adsorption study was conducted using parameters of such as: pH, contact time, adsorbent dosage, and initial concentration. The present investigation shows that, the percentage removal of (Fe (II), Co (II), and Ni (II) increased with increase in adsorbent dosage, due to availability of more active sites and the percentage removal of Co (II) and Ni (II) decrease with increase in initial concentration due to the presence of fixed number of active sites. The percentage removal of Fe (II) increased with increase in concentration which may be as a result of nature of the metal or large surface area of the adsorbent to accommodate the metal.The percentage removal of Fe (II), Co (II), and Ni (II) increased with increase in pH and it was found that the optimum pH condition for the removal of Fe (II), Co (II), and Ni (II) was at pH 6 with percentage removal of 80.46, 80.43, and 80.45% for the metal ions, respectively.The results were analyzed using two adsorption isotherm models naming as the Langmuir and Freundlitch isotherm models, the experimental results are in good agreement with Langmuir model for the metal ions. The kinetic study also obey pseudo-second model. Therefore, study concluded that pollution caused by heavy metals may be removed using Schiff base ligand derived from salicyaldehyde and 3-aminobenzoic acid, since its easy to prepare and used in smaller quantity.

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