

# Adsorption/Cation Exchange Removal of Ni (II) Ion from Aqueous Solution Using Modified Coir Dust

<sup>1</sup>\*ESSIETT, A. A., & <sup>2</sup>ISRAEL, A. U.

<sup>1</sup>Department of Physics, University of Uyo, Nigeria

<sup>2</sup>Department of Chemistry, University of Uyo, Nigeria

Corresponding: Author Email: [aessiett2@yahoo.com](mailto:aessiett2@yahoo.com)

## ABSTRACT

*The removal of Ni(II) ion from aqueous solution by ion exchange was studied from the coir dust of extract and residue. The extent of removal of Ni(II) was found to be dependent on contact time and it was found out that percentage of adsorption increases with contact time. The experimental data was described by pseudo 1<sup>st</sup> and 2<sup>nd</sup> orders and intra-particle diffusion as kinetic models while Froehlich, Langmiur and Flory-Huggines model for equilibrium. Intra-particle diffusion model has a correlation coefficient of  $R^2 = 0.956$  and  $R^2 = 0.895$  respectively.*

**Keywords:** Adsorption; Cation-exchange; Coir Dust; Ni(II); Contact time.

## INTRODUCTION

Nickel is a silvery white metal, capable of taking a high polish. It has atomic number 28, chemical symbol Ni, atomic weight of 58.6934 and electronic configuration of  $4s^2 3d^8$ . Nickel has 14 known isotopes which 5 of it are stable and 9 are unstable. Nickel has a melting point of  $1453^\circ\text{C}$ , boiling point of  $2732^\circ\text{C}$ , specific gravity of 8.902 ( $25^\circ\text{C}$ ), with a valence of 0, 1, 2, 3, which as a metal it is hard, ductile, malleable. Nickel is a member of the iron – cobalt group of metal (transition elements). Nickel is the earth's 22<sup>nd</sup> most abundant element and the 7<sup>th</sup> most abundant transition metal. It is silver white crystalline metal that occurs in meteors or combined with other elements in ores. Nickel is known primarily for its divalent compounds since the most important oxidation state of the element is +2. (Abia and asuquo, 2006). Nickel metals becomes toxic when they are not metabolized by the body. It accumulates within the body's fat cells, central nervous system, bones, brain, and glands. It may enter the human body through

food, water, absorption through the skin which many pieces of jewelry contain nickel and wearing them close to skin and some of our cooking utensils have nickel added to them. Nickel can be released in waste water and also from stacks of large furnaces used to make alloys or from power plants and trash incinerators. Nickel toxicity may caused dizziness, heart attack, kidney dysfunction, stress, vomiting, pulmonary congestion, shortness of breath, abdominal pain and cancer (Babel and Kurmlawam, 2003). The primary use of nickel is in the preparation of alloys such as stainless steel, which accounts for approximately 67% for all nickel used in the manufacturing.

Heavy metals “copper, nickel, lead, gold, gallium, cadmium, platinum, mercury, cobalt antimony” etc present in industrial wastewater are hazardous to the aquatic ecosystem, which have posed possible human health risk. These metals are not biodegradable and their accumulation have harmful effect on both animal and plants. The excessive exposure to nickel can lead to severe

damage of lungs, kidneys, skin dermatitis and cancer. This heavy toxic metal entered into the water bodies through wastewater from metal plating industries of Cd-Ni batteries, phosphate fertilizer, mining, pigment, and stabilizer alloys (Balasubramanian and Ahamen, 1998).

Several techniques such as chemical precipitation, oxidation, reduction, coagulation, solvent extraction and adsorption have been commonly employed for the removal of this metal ions. Among these, ion exchange has been thought to be efficient and economically feasible as a wastewater treatment operation. Several resins can be used to remove Ni(II) ions including activated carbons, alumina silica and peat. Ion exchange resins with improved sorption capacity as well as adsorbents. But ion exchange resins hold great potential for the removal of heavy metals from water and industrial wastewater as well as the use of coconut coir dust which is regarded as low cost and non-conventional adsorbent. Thermodynamics, equilibrium, kinetics possible mechanism, design and optimization of the adsorption operation could be used to evaluate the practicability of Ni(II) adsorption by coconut coir dust (Giizel *et al.*, 2008).

Adsorption is the concentration of a substance at the interface between two immiscible phase and the larger the surface area greater will be the adsorption. Similar to surface tension, adsorption is a consequence of surface energy. Adsorption are usually described through isotherms model, that is the amount of adsorbate on the adsorbent as a function of its pressure (if gas) or concentration (if liquid) at constant temperature. The quantity adsorbed is nearly always normalized by the mass of the adsorbent to allow comparison of different materials (Kumar, 2006).

Ions exchange is an exchange of ions between two electrolytes or between an electrolyte solution and a complex. The amphoteric exchangers are able to exchange both cation and anion simultaneously. Coir dust is very similar to peat in appearance. It is light to dark brown in colour and consists of primarily particles in the size range 0.2 – 2.0mm (75-90%). Unlike sphagnum peat there are no sticks or other extraneous matter. Cresswell (1992) looked at coconut coir dust in comparison to sedge and sphagnum peat products and remarked that it has superior structural stability, water absorption ability and cation exchange capacity compared to either sphagnum peat or sedge peat. Coir dust tends to be high in both sodium and potassium (Hume, 1949). The high level of potassium present in coir dust has made it more benefit than being detrimental to plant growth. Activated carbon has been recognized as a highly effective adsorbent for removal of heavy metal from concentrated and dilute metal bearing effluents (Okieimon *et al.*, 1991). Activated carbon production from agricultural waste such as coconut shell has been carried out for the decontamination of wastewaters from heavy metals.

A preliminary study of the removal of heavy metal ion from aqueous solution by modified EDTA combination of cellulosic material (groundnut husk). Coconut Shell Charcoal (CSC) and activated carbon for removal of metal ion have been investigated activated in batch studies, using synthetic electroplating wastewater. Alternatively conventional treatment system for removal of toxic metal such as dyes, Ni, Cu from aqueous solution. Activated carbon can be used as adsorbent for removal or reduction of gaseous pollutants from the exhaust gases of

industrial source such as power stations and sulphuric acid production plants. They can also be used to extract some harmful elements of cigarettes, in refrigerator to remove general food odours.

Ion exchange chromatography (cation and anion) is also one of the methods for metal removal/recovery from solutions includes oxidation-reduction, membrane technologies, evaporative recovery, and filtration. Ion exchange is a chemical reaction wherein ion attached to an immobile solid particle of ion exchange resin (Weixing *et al.*, 1998). Among other conventional adsorbents include alumina, zeolite, silica gel, metal oxides and polymer resin. Conventional methods for the removal of metals include chemical precipitation, chemical oxidation or reduction, (Zacaria, 2002) filtration, ion-exchange, electrochemical treatment and evaporation. These technique become ineffective and non-economical when removal of metal at very low concentration is required.

Adsorption technique for wastewater treatment has received considerable attention for development of an efficient, clean and economical technology. Activated carbon has widely been used for removing various pollutants from aqueous solution. In fact their removal efficiency is not as high as that of activated carbon because of their low adsorption capacity but this fact can be compromised because of their low cost and ease of availability. Nowadays sorption and ion-exchange studies for the removal/recovery of ions (cations/anions) from solution have shifted from the use of conventional adsorbents silica gel, active alumina, zeolites, metal oxides, activated carbon and polymer resins to agricultural wastes (Rafatulla *et al.*, 2009). Many agricultural by-product have modified with acidic functional

groups to act as cationic exchange material. Removal of some heavy metal from solution by modified coconut coir dust has been reported (Rengaray and Moon, 2002). Also cation exchange resin from coconut coir dust has been reported for removal of  $\text{Ca}^{2+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Ni}^{2+}$  from solution (Srivastava *et al.*, 1989).

**Apparatus:** ZD – 8801 model electric shaker, Beaker (100, 250 and 500), Model P165 weighing balance, Spatula, Model SM – IZA water bath thermostatic shaker, Desicator, Gallenkamp oven, Sample bottle, Measuring cylinder, Stirring rod and Conreal flask.

**Material:** Aluminum foils, Hand gloves, Paper cellotape and Handkerchiefs.

**Reagents:** Salt (AICI) – General purpose reagent, BDH London, Deionized water, Nickel.

**Resin:** Modified washed coconut coir dust.

**Method:** The coconut coir dusts were collected from a coconut processing mill at Mkpato Enin Local Government Area of Akwa Ibom State, Nigeria. Coconut coir dust was obtained from coconut husk by severe beating of the husk. The coir were cut into sizable pieces, air dried in sunlight and sieved into various particle sizes. Dried coconut coir dusts (raw) was poured into a beaker containing 250ml deionized water level and placed in an electric shaker for twelve (12) hours. It was then filtered with the used of white handkerchief. The residue was dried and the filtrate was placed on a hotplate until it evaporate to extract. This was known as cold water extraction. Same procedure was repeated for hot water extraction and was placed in an hotplate for twenty four (24) hours. Residue from the hot water extraction method was dried and the filtrate was placed again in a hotplate until it evaporate to extract. The collection of extract of both the cold water extraction and the hot water

extraction was put in a desiccators to dry for modification. Twenty grams (20g) of extract obtained from both the cold and hot water extraction stage of coir dust. 10g of aluminum chloride (AlCl<sub>3</sub>) salt and 200ml of deionized water was added to the beaker. Both the extract weighed to a certain quantity and residue dried were soaked with the dissolved aluminum chloride salt and stirred for 15 minutes before leaving it covered with aluminum foil for forty-eight (48) hours to react before filtration. The beaker that contain the modified extract was placed in a hotplate to obtain an extract while the modified residue was dried and stored.

For kinetics studies, the following procedure for initial concentration was taken at temperature of 30°C. The coconut coir dust of both extract and residue weighed 0.2g was placed in a separate conical flask each with 10ppm (part per million) Nickel solution measured 20ml with measuring cylinder. It was placed in a platform electric shaker to shake at the interval of time from 0.5 minute, then to 5, 10, 30, 60 and 90 minutes. After which it was then filtered with white clean handkerchief with the aid of small plastic funnel. The filtrate was stored in 12 plastic sample bottles for analysis. About 20ml each of Nickel solution measured with conical flask of various concentrations of 2, 6, 10, 15 ppm was poured into 8 different conical flask labeled to the ppm of nickel solution poured into it. 0.2g of both extract and residue of coconut coir dust was also

poured into four (4) of each conical flask and placed on a electric shaker for contact time of 90 minutes, after which the content of the flask was then filtered using clean white handkerchief with the aid of a small plastic funnel into 8 different plastic sample bottles for analysis. 20ml of each of Nickel solution of various ppm (part per million) that is (2, 6, 10, 15 ppm) was poured into 8 different conical flask of the same labeled ppm poured into it. 0.2g of both extract and residue of coconut coir dust was also poured into four (4) of each conical flask and agitated in a water bath thermostatic shaker set to a temperature of 50°C for 90 minutes. The content of the flask was then filtered using white handkerchief into 8 different plastic sample bottles. The same procedure was repeated for temperature of 70°C. Both the content in the same bottle for 50°C and 70°C temperature were analyzed.

## RESULTS

The percentage adsorption of Ni (II) ion onto coir dust extract and residue as a function of initial concentration at 30°C is presented in table 1. This indicates that energy are less favourable in the sites to become involved with increasing metal concentration in aqueous solution. The heavy metal uptake is attributed to different mechanism of ions exchange and adsorption process. During the ion-exchange process, metal ion has to move through pores of extract and residue.

**Table 1: Percentage Adsorption of Ni(II) Ion for Extract and Residue Kinetics**

Time (min)	Extract (30°C)			Residue (30°C)		
	Ce (mg/l)	Xe (mg/l)	% Adsorption	Ce (mg/l)	Xe (mg/l)	% Adsorption
0.5	3.26	4.74	67.40	3.17	6.83	86.30
5	3.02	6.98	69.80	2.94	7.06	70.60
10	1.60	8.40	84.00	2.21	7.79	77.90
30	1.45	8.55	85.50	1.63	8.37	83.70
60	0.36	9.64	96.40	0.75	9.25	92.50
90	0.36	9.64	96.40	0.75	9.25	92.50

The equilibrium data for the metal ions over the concentration range of 2, 6, 10, 15 ppm at 30°C, 50°C, and 70°C have been correlated with the Langmiur, Freunlich and Flory Huggin isotherms and the result presented in Table 2.

**Table 2: Langmiur, Freundlich, Flory-Huggin isotherms**

	Extract (30°C)					Residue						
	Co (mg/l)	Ce (mg/l)	Xe (mg/l)	qe (mg/l)	Ce/qe	Co (mg/l)	Ce (mg/l)	Xe (mg/l)	qe (mg/l)	Ce/qe		
Langmiur isotherm	2	0.85	1.15	0.14	6.07	2	0.90	1.10	0.13	6.92		
	6	2.31	3.69	0.46	5.02	6	1.14	4.86	0.61	1.87		
	10	2.57	7.43	0.93	2.76	10	1.83	8.17	1.02	1.97		
	15	3.49	11.51	1.45	2.41	15	3.46	11.54	1.45	2.39		
The relationship for the langmiur isotherm can be shown in the following liner form.												
	Co	Ce	Ce	qe	Log qe	Log Ce	Co	Ce	Ce	qe	Log qe	Log Ce
Freundlich isotherm	2	0.85	1.15	0.14	-0.85	-0.07	2	0.90	1.10	0.13	-0.89	-0.05
	6	2.31	3.69	0.46	-0.34	0.36	6	1.14	4.86	0.61	-0.22	0.06
	10	2.57	7.43	0.93	-0.03	0.41	10	1.83	8.17	1.02	0.01	0.26
	15	3.49	11.51	1.43	0.16	0.54	15	3.46	11.54	1.42	0.15	0.54
Flory-Huggin Isotherm	2	0.85	1.15	-1.35	0.37	-0.17	2	0.90	1.10	-1.22	0.35	-0.22
	6	2.31	3.69	-1.60	0.42	-0.58	6	1.14	4.86	-4.26	0.75	-0.15
	10	2.57	7.43	-2.90	0.59	-0.54	10	1.83	8.17	-0.22	0.09	-1.66
	15	3.49	11.51	-3.30	0.63	-0.66	15	3.46	11.54	-3.34	0.64	-0.65

$$\frac{C_e}{Q_e} = \frac{1}{Q_{ob}} + \frac{C_e}{Q_o} \quad (1)$$

Where  $C_e$  is the equilibrium concentration of metal ions in solution.  $Q_e$  is the equilibrium quantity of metal ions adsorbed onto the adsorbent at equilibrium,  $Q_o$  and  $b$  are langmiur constant related to sorption capacity and sorption energy respectively.  $Q_o$  is the monolayer coverage of sorbent with sorbet and it represent the enthalpy of sorption and it varies with temperature. A linear plot can obtain for each of the adsorbent when  $C_e/q_e$  is plotted against  $C_e$  over the entire concentration range of metal ions investigated. The Langmiur model parameters and the statistical; fits of the sorption data to the equation are given in table 2 for extract and residue respectively.

The Freundlich sorption isotherm is one of the most widely used mathematical descriptions which usually fit the experimental data over wide range of concentration. Table 2 also shows the Freundlich parameters for the sorption of nickel (II) ion on both extract and residue of modified coir dust. The Freundlich isotherm is illustrated in table 2 for extract and residue at different temperature of 30°C, 50°C and 70°C was applied to the equation.

$$\text{Log } q_e = \text{log } K_f + 1/n \text{ log } C_e \quad (2)$$

Where  $C_e$  is the equilibrium of concentration in mg/L and  $q_e$  is the quantity of the metal ions adsorbed into the adsorbent at equilibrium.

The constant  $k_f$  and  $n$  was calculated for the metal caution from slope and intercept of the plot.  $K_F$  is related to the temperature and is a characteristic constant for the adoption system of study. The correlation values of  $R^2$  for both extract and residue are 0.090, 0.735, 0.757; 0.624, 0.843 and 0.957 respectively, indicating that the adsorption can be effectively described by the Freundlich isotherm. The numerical values of  $1/n < 1$  indicates that sorption capacity is suppressed at lower concentration of coir dust and predict instauration of coir dust by sorbate and also indicting multilayer adsorption,  $1/n > 1$  for extract and residue indicate that the portion capacity has exceed at higher constrains and therefore predicts saturation of the sorbents by sorbate and also indicates multilayer adsorption and finite surface coverage by the adsorbate on the adsorbents.

The Flory-Huggins isotherm is used to described the sorption of adsorbate on adsorbent and the result is presented in table 2. This model helps to account for the degree of surface coverage characteristics of the adsorbate on the adsorbents is expressed in a linear form as:

$$\text{Log } \theta / C_o = \text{log } K_{FH} + C_o = \text{log } K_{FH} + \alpha_{FH} \text{ log } (1 - \theta) \quad (3)$$

Where  $\theta$  is the surface coverage of the adsorbent by the adsorbent  $\theta = (1 - C_o/C_e)$  where  $C_o$  and  $C_e$  are initial and equilibrium concentration of nickel ions concentration respectively. The plot of  $\text{log } \theta / C_o$  versus  $\text{log } (1 - \theta)$  will linear if the adsorption process confirms to this model. As shown in figure 17, 18, 19, 20, 21 and 22 respectively and the correlation value of  $R^2 = 0.543, 0.181, 0.010, 0.409$  and  $0.13$  for both extract and residue indicates that the data can be described using this isotherm model in related to Gibb's free energy by the equation.

$$\Delta G^\circ = RT \text{ In } K_{FH} \quad (4)$$

R is the universal gas constant = 8.314 Jk<sup>-1</sup>mol<sup>-1</sup>. T is the absolute temperature (°K) and K<sub>FH</sub> is the Flory-Huggin’s equilibrium constant.

The integrated form of pseudo first-order kinetic is

$$\text{Log}(q_e - q_t) = \text{In } q_e - K_1 t / 2.30 \quad (5)$$

**Table 3: Pseudo-First-Order**

Extract							Residue						
Time (min)	C <sub>t</sub> (mg/l)	X (mg/l)	q <sub>t</sub> (mg/l)	Log q <sub>e</sub> -q <sub>t</sub>	t/q <sub>t</sub>	t <sup>1/2</sup>	Time (min)	C <sub>t</sub> (mg/l)	X (mg/l)	q <sub>t</sub> (mg/l)	Log q <sub>e</sub> -q <sub>t</sub>	t/q <sub>t</sub>	t <sup>1/2</sup>
0.5	3.26	6.74	0.84	-0.43	0.60	0.71	0.5	3.17	6.83	0.85	-0.51	0.59	0.71
5	3.02	6.98	0.87	-0.47	5.75	2.24	5	2.94	7.06	0.88	-0.55	5.68	2.24
10	1.60	8.40	1.05	-0.80	9.52	3.16	10	2.21	7.79	0.97	-0.72	10.31	3.16
30	1.45	8.55	1.07	-0.85	28.04	5.48	30	1.63	8.37	1.05	-0.96	28.57	5.48
60	0.36	9.64	1.21	0.00	49.59	7.75	60	0.75	9.25	1.16	0.00	51.72	7.75
90	0.36	9.64	1.21	0.00	74.38	9.94	90	0.75	9.25	1.16	0.00	77.59	9.49

Table present the value of constant of kinetics models like pseudo first-order for the sorption of Ni(II) ion onto coir extract and residue. The kinetic rate constant K<sub>1</sub> are for extract and residue respectively which are and indicate that the adsorption process does obey the pseudo-first-order kinetic. The result shows how correlation for pseudo first order R<sup>2</sup> = 0.529 and 0.508 for extract and residue respectively.

The pseudo second order is given in the following linear form;

$$\frac{t}{qt} = \frac{1}{K_2 q_e} + \frac{t}{q_e} \quad (6)$$

**Table 4: Pseudo-Second-Order**

Extract					Residue				
Time (min)	C <sub>t</sub> (mg/l)	X (mg/l)	q <sub>t</sub> (mg/l)	t/q <sub>t</sub>	Time (min)	C <sub>t</sub> (mg/l)	X (mg/l)	q <sub>t</sub> (mg/l)	t/q <sub>t</sub>
0.5	3.26	6.74	0.84	0.60	0.5	3.17	6.83	0.85	0.59
5	3.02	6.98	0.87	5.75	5	2.94	7.06	0.88	5.68
10	1.60	8.40	1.05	9.52	10	2.21	7.79	0.97	10.31
30	1.45	8.55	1.07	28.04	30	1.63	8.37	1.05	28.57
60	0.36	9.64	1.21	49.59	60	0.75	9.25	1.16	51.72
90	0.36	9.64	1.21	74.38	90	0.75	9.25	1.16	77.59

The sorption rate constant K<sub>2</sub> and the sorption capacity q<sub>e</sub> of the pseudo-second-order shown in table 4 for extract and residue. The correlation co-efficient are relatively higher R<sup>2</sup> = 0.998 and 0.967 respectively.

**Table 5: Intra-Particle Diffusion**

Extract					Residue				
Time	C <sub>t</sub>	X	q <sub>t</sub>	t <sup>1/2</sup>	Time	C <sub>t</sub>	X	q <sub>t</sub>	t <sup>1/2</sup>
0.5	3.26	6.74	0.84	0.71	0.5	3.17	6.83	0.85	0.71
5	3.02	6.98	0.87	2.24	5	2.94	7.06	0.88	2.24
10	1.60	8.40	1.05	3.16	10	2.21	7.79	0.97	3.16
30	1.45	8.55	1.07	5.48	30	1.63	8.37	1.05	5.48
60	0.36	9.64	1.21	7.75	60	0.75	9.25	1.16	7.74
90	0.36	9.64	1.21	9.49	90	0.75	9.25	1.16	9.49

The intra-particle diffusion coefficient for the adsorption of Nickel (II) ion onto coir dust extract and residue was calculated, from the slope of the plot of the amount of Ni(II) ion adsorbed  $q_t$ (ml/l) versus  $t^{1/2}$  ( $\text{min}^{1/2}$ ). It is discovered that sorption process of Nickel (II) ion consist of two phase; suggesting that the intra-particle diffusion is into the rate limiting step for the whole reaction. The intercept of the plot which were determined to be 1.438 and 1.499 respectively for coir dust of extract and residue, which provide an estimation of the thickness for the boundary layer. Extract is assumed large thickness and multilayer surface coverage. While for residue is assume monolayer coverage. The two phase in the intra-particle diffusion suggest that the adsorption of Ni(II) ion onto coir dust of extract and residue is proceeds by surface sorption and intra-particle diffusion.

### Conclusion

The sorption process attains equilibrium for the sorption of ions from aqueous solution of water samples treated. Modified and unmodified coir dust resins are very effective for removal of Nickel from aqueous solutions. Coir dust as a natural adsorbent is more efficient on adsorption and it has adsorptive ability for uptake of heavy metal. Notably, the removal efficiency of Ni(II) ion from aqueous solution using the modified coir extract proved to have higher adsorption capacity than the unmodified ones. Also the adsorbent material of ion exchange can be used for the removal of Nickel from wastewater. We conclude that ion-exchange could be used for applications in the advanced treatment of portable water as well as industrial effluents characterization studies on agricultural by-product (coconut coir dust) as an adsorbent for removal of Nickel (II) ion from aqueous solution

to be highly effective and efficient. The result obtained qualified that the resins of extract and residue of coconut coir dust are efficient adsorbent and cation exchange for heavy metals removal. The kinetic data confirm the applicability of the first order, second order and intra-particle rate of expression for the sorption of Ni(II) ions onto the extract and residue of coconut coir dust. The equilibrium data flitted to the Langmiur, Freundlich and Flory-Huggins.

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