

Coconut Coir Dust Ion Exchange Resins for Removal of Ni²⁺ ion from Aqueous Solution

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Abstract

The ability of coconut coir dust, a natural adsorbent made from dried coconut husk as a non conventional and low cost sorbent for the removal of Ni²⁺ from aqueous solution was examined. The adsorption capacity was found to be dependent on the contact time and initial concentration which increased along with contact time and initial ion concentration but latter decreased with 15mg/L for the three resins (coconut coir dust (CCD), carboxylated toluene resin (CTR), and sulphonated toluene resin (STR)). Maximum adsorption was obtained after a contact time of 90minutes at an initial concentration of 15mg/L. The results were analyzed by Langmuir and Flory-Huggins isotherm. The sorption process was best described by Langmuir isotherm which indicates monolayered adsorption and chemisorption. Adsorption was found to follow pseudo-second order kinetics with average rate constant of 1.3287g/mg min. Adsorbents obtained from coconut can be used as sorbent for removing metal ions from aqueous solution.

Keywords: Adsorption, coir dust, ion exchange, Ni²⁺ions, kinetics, isotherms

1.0 Introduction

Nickel is noticeably contained in wastewater streams from industries such as mining, electroplating, metallurgy, pigment and ceramics industries (Akhtar *et al.*, 2004). This metal is non-biodegradable and toxic and may cause dermatitis allergic, sensitization and cancer (Oliver, 1997). It has therefore become very necessary to remove or at least reduce the content of this toxic element from industrial effluents. A number of processes have been confirmed useful for removal of toxic substances from effluents. These include chemical precipitation (Ku & Jung, 2001), reverse osmosis (Shahalam *et al.*, 2002), nanofiltration (Murthy & Chaudhari, 2008) flocculation (Wang *et al.*, 2003), coagulation (Wang *et al.*, 2004), ion exchange and adsorption (Crini, 2005; Kurniawan *et al.*, 2006). In recent times, there has been increasing utilization of bio-waste to develop sorbents for wastewater treatments. Several biomaterials which are discarded as waste from domestic and commercial processing such as rice husk (Khalid *et al.*, 2000), sawdust (Argun, *et al.*, 2007), orange peel (Khaled *et al.*, 2009), pineapple leaves (Hameed *et al.*, 2009), pine leaves (Sen *et al.*, 2011), corncob (Shen & Duvnjak, 2005), have been explored and used as sorbents for wastewater treatments containing coloured compounds such as dye (Sen *et al.*, 2011) and metal ions (Israel *et al.*, 2011). The use of biomaterials is a step towards the realization of the greenness of the environment which is fast replacing the conventional mechanical means of solving this environmental problem. In the present research, coconut coir was utilized as an eco-friendly and technological biomaterial for treating a simulated industrial wastewater by adsorption via ion exchange mechanism. Whereas adsorption is chosen due to its economy, ease of

operation, wide variety of target pollutants, high capacity, fast kinetics, possibly selective depending on adsorbent (Crini, 2005; 2008), ion exchange is given a hand because Ni^{2+} can exchange preferentially with other ions such as H^+ , Na^+ , Ca^{2+} (Chockalingam & Subramanian, 2004; Kwon *et al.*, 2005; Miretzyky *et al.*, 2007). Coconut coir dust is a lignocellulosic material of plant origin obtained from shredding of coconut husk. Coconut husk is discarded as waste in large quantities in coconut processing industries in Nigeria. Similar reports have been documented in other countries such as Philippines, China, Srilanka and India (Tejano, 1985; Vandam, 2002). With the availability of these bio-wastes there is therefore need to investigate how they can be converted to wealth by its utilization as sorbent for metal ions removal. The specific objectives of this work are: (i) prepare a cation exchange resin from coconut coir dust (ii) modify the water/acetone coir extract with toluene diisocyanate (TDI) as cross linking agent for phenol sulphonic acid and hydroxyl benzoic acid (iii) removing of Ni^{2+} from aqueous solutions and (iv) to elucidate the mechanisms of removing Ni^{2+} by ion exchange and adsorption using Langmuir and Flory-Huggin isotherms at 40°C.

2.0 Experimental methods

2.1 Materials and preparation

Coconut coir dust used in this study was sourced locally from coconut processing mill in Akwa Ibom State, Nigeria. It was subjected to screening according to the methods adopted by Etim *et al.*, (2012). Ni^{2+} in the form of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was obtained from Burbidges Burgoyne & Co. Mumbai, India. All reagents including Toluene Diisocyanate (TDI), 4-Hydroxybenzoic acid and 4-Phenolsulphonic acid obtained from Sigma Aldrich chemicals were of analytical grades. De-ionized water was used for preparation of solution in the experiments.

2.2 Extraction of coconut coir dust

(a) Water extract: Coir dust (5g) was added to 100ml of de-ionised water in a beaker on hot plate and stirred for 2 h at the boiling point of water. At the end of this time, the mixture was filtered and the filtrate evaporated to dryness. The weight of the extract was obtained.

(b) Acetone/water extract: Coir dust (5g) was added to (50/50) V/V acetone/water mixture in the thimble of Soxhlet extractor fitted to a flat round bottom flask containing 100ml of the solvent and heated to a temperature of 75° – 85°C until the solution in the arm of the extractor was colourless. Extract was recovered by distillation and drying.

2.3 Characterization of coconut coir dust extract

The coir dust was characterized for lignin, cellulose, elemental contents, and cationic exchange capacity, according to standard methods. The presence of lignin and cellulose in the coir dust was determined using the phloroglucinol solution and Hierberman-Storch method (Browning, 1990), respectively. Cation exchange capacity (CEC) was determined using standard method of Tanigami *et al.*, (2007). The elemental analysis of coir dust based on dry weight (water extract) was carried out according to standard methods [AOAC, 1975].

2.4 Preparation of resins

Coir extract resin (CCD)

Mixed solvent extract (1.45g) was added to 12.5ml re-distilled acetone until completely dissolved and TDI (0.725ml) was added with stirring until it cakes.

Sulphonated Toluene Resin (STR)

Mixed solvent extract (1.45g) was dissolved in 12.5ml of re-distilled acetone and the mixture added to 0.65ml 4-Phenolsulphonated acid in drops and stirring. TDI (0.725ml) was later added and stirred until a brown solid was formed.

Carboxylated Toluene Resin (CTR)

Mixed solvent extract (1.45g) was dissolved in 12.5ml of acetone. 4-hydroxybenzoic acid (0.609ml) was added and shaken for 5mins. TDI (0.725ml) was added and stirred until it cakes.

2.5 Adsorption methods

Batch adsorption was adopted for removal of Ni²⁺ from solution of its aqueous solution. Adsorbents (0.2g) was placed in a 250ml conical flask containing 100ml of predetermined metal ion concentrations ranging from 2-15mg/L and stirred on a magnetic stirrer model 20-880, Japan, for a determined equilibrium time of 90minutes. The mixture after equilibrium was filtered through Whatman-42 filter paper and the filtrate analyzed for residual metal concentration using Atomic Absorption Spectrophotometer 200A. The fraction of the metal ions adsorbed was calculated by the expression in equation 1.

$$M_f = \frac{M_o - Me}{M_o} \quad 1$$

The quantity of the metal ion absorbed by the adsorbents at different times is given by equation 2.

$$q_t = \frac{M_o - Me}{M} * V \quad 2$$

Where M_f is fraction of metal ion adsorbed (no unit), M_o is the initial metal ion (mg/L), M_e residual metal concentration at equilibrium (mg/L), V is volume of metal ion solution (ml), M mass of the adsorbent (g), q_t is adsorption capacity of adsorbent after time (min).

2.6 Effect of contact time

Twenty milliliter (20ml) of Ni²⁺ solution was contacted with 0.2g of the resins in 250ml conical flask containing 100ml of the metal solution and shaken on a mechanical stirrer at 140rpm for different period of time, viz:

0.5, 5, 10, 30, 60, 90 and 120minutes, at pH 7.0 and temperature 40°C. After shaking the residual metal ions in the solution was measured using Atomic absorption spectrophotometer (AAS- Pye Unicam 919), Japan.

2.7 Effect of initial concentration

A fixed mass (0.2g) of the resins were added to 30ml of different concentration (2, 4, 8, 10 and 15mg/L) of the Ni²⁺ solution in a 250ml conical flask and shaken till equilibrium (90mins) at pH 7.0 and temperature 40°C. The residual metal concentration was determined using AAS.

3.0 Results and discussion

3.1 Characterization of coconut coir dust

Results show that hemicellulose and lignin contain 36.45% and 54.3% respectively. These values are close to those reported earlier for the same adsorbent (Israel & Eduok, 2012; Israel *et al.*, 2011; Okon *et al.*, 2012). However, values are higher compared with that of other lingo-cellulosic biosorbents (Table 4.1). The elemental analysis indicates that coconut coir dust contains elements such as Na, K, Ca and Mg. These metals have excellent exchangeable properties which make exchange with heavy metals (Pb, Cd, Zn, Fe and Ni) possible (Israel & Eduok, 2012). Table 1 compares the value of cellulose and lignin composition of coconut coir dust with other bio-sorbents. The cation exchange capacity (CEC) of coconut coir dust obtained in the present study (2.35mmol/g) is comparable to the value obtained by Israel *et al.*, 2011.

Table 1: comparison of cellulose and lignin content of coconut coir dust with other lingo-cellulose materials

Bio-sorbent	Cellulose%	Lignin%	References
Coconut coir dust	35.99	53.5	Israel <i>et al.</i> , 2011
Rice straw	37.20	12.2	Nada <i>et al.</i> , 2002
Bagasse	46.2	20.2	Nada <i>et al.</i> , 2002
Cotton stalk	42.8	27.3	Nada <i>et al.</i> , 2002
Coconut coir dust	36.5	54.3	present study

3.2 Adsorption by the resins

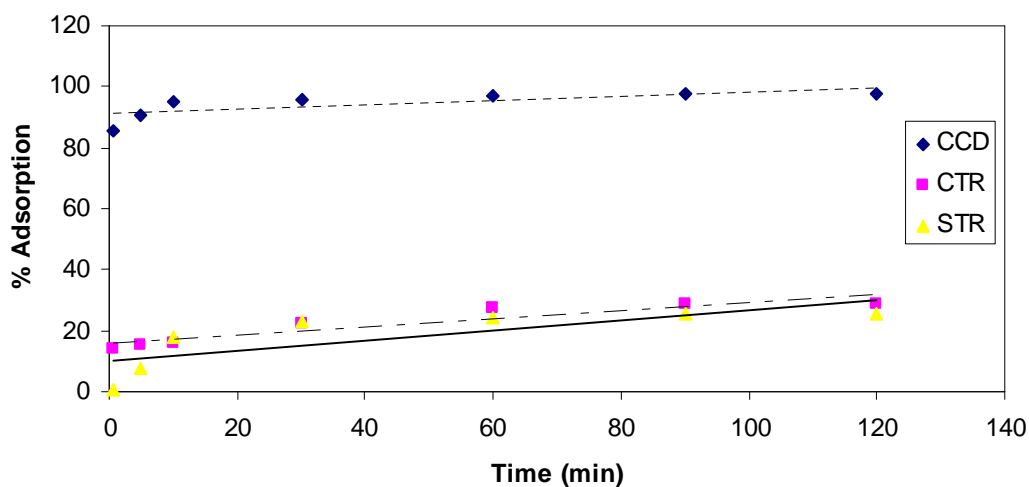


Figure 1: Variation of adsorption by resins with time at 40°C

Figure 1 shows variation of adsorption of the metal ions with time for the resins. The percentage adsorption of the metal ions on the different adsorbent: coir dust, carboxylated toluene resin and sulphonated toluene resin reveals that the adsorption of Ni^{2+} is maximum for CCD (97.5%) followed by CTR (29.0%) while STR adsorbed least (25.5%) (Figure1). The percentage adsorption follows the order $\text{CCD} > \text{CTR} > \text{STR}$. The results indicates that raw CCD provides more accessible surface for the attachment of the Ni^{2+} from the solution in exchange for those on the surface of the resins.

3.3 Effect of contact time on adsorption of Ni^{2+} ion

Adsorption increases with increasing contact time. The Ni^{2+} uptake versus contact time curve is presented in Figure 1 for the different adsorbents namely CCD, CTR and STR. The adsorption of Ni^{2+} at 0.5min is extremely low for STR (0.4%) and increases with increase in contact time. It is therefore evident from the curve that increase in adsorption is a function of prolonged contact time indicating a better and enhanced sorption at higher times (Adekoka, 2005). Increasing the contact time increases adsorption to equilibrium after 90minutes. Maximum uptake of Ni^{2+} occurs with coir dust at the same equilibrium time.

3.4 Effect of initial ion concentration on adsorption of Ni^{2+}

One of the most critical parameter in the adsorption process of metal ions from aqueous solution of its salts is the initial ion concentration. Figure 2 shows the variation of the ion concentration on the adsorption behaviour of CTR and STR. The figure shows that increase in initial ion concentration leads to increase in the adsorption capacity. Maximum adsorption of Ni^{2+} ions occur at concentration of 8mg/l for the adsorbents. This result is in line with report of Israel *et al.*, (2011). Increase in the initial dye concentration caused an increase in the loading capacity of

the adsorbent and this may be due to the high driving force for mass at a high initial dye concentration (Bulut & Aydin, 2006)

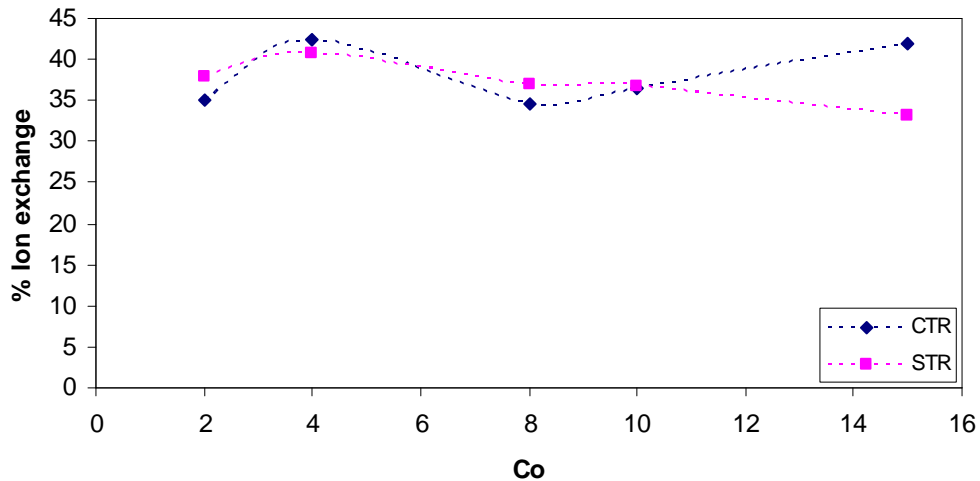


Figure 2: variation of initial concentration of Ni²⁺ with adsorption

3.5 Fractional attainment of equilibrium, α

Table 2: Fractional attainment of equilibrium for adsorption of Ni²⁺ at 40°C

Time (min)	CTR		STR		CCD	
	Co-Ct (mg/L)	α	Co-Ct (mg/L)	α	Co-Ct (mg/L)	α
0.5	8.54	0.88	1.43	0.49	0.04	0.02
5	9.06	0.92	1.51	0.52	0.77	0.30
10	9.53	0.97	1.60	0.55	1.74	0.68
30	9.59	0.98	2.24	0.77	2.28	0.89
60	9.71	0.99	2.74	0.94	2.42	0.94
90	9.75	1.00	2.90	1.00	2.57	1.00

Table 2 above shows the fractional attainment of equilibrium, α , of the resins, CCD, CTR and STR. α is calculated according to equation 3.

$$\alpha = \frac{Co - Ct}{Co - Ce} \quad 3$$

Where C_t is the concentration of metal ion in solution at time t, C_o is the initial concentration of metal ion in solution and C_e is the equilibrium concentration of metal ion. The value of fractional attainment approaches unity with increase in time. After 90minutes maximum adsorption occurred to the creation of some sites on the adsorbent surface by resin modification. Figure 3 shows the fractional attainment of equilibrium versus time plot for the three

resins at 40°C. The value of α (1.00) after 90minutes indicates that equilibrium is completed after this time. Distribution coefficient, D is non-linear showing non uniformity of partitioning with time within the bulk of the solution.

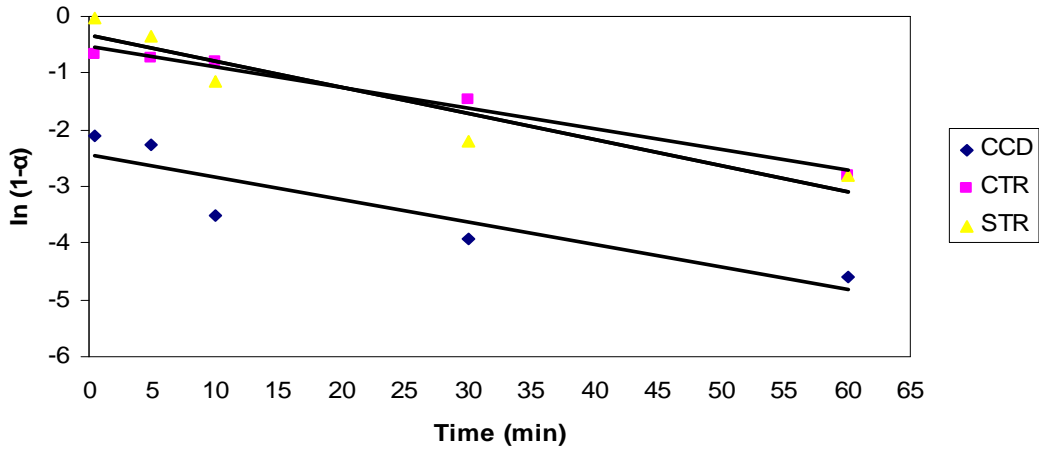


Figure 3: Fractional attainment of equilibrium for Ni^{2+} at 40°C

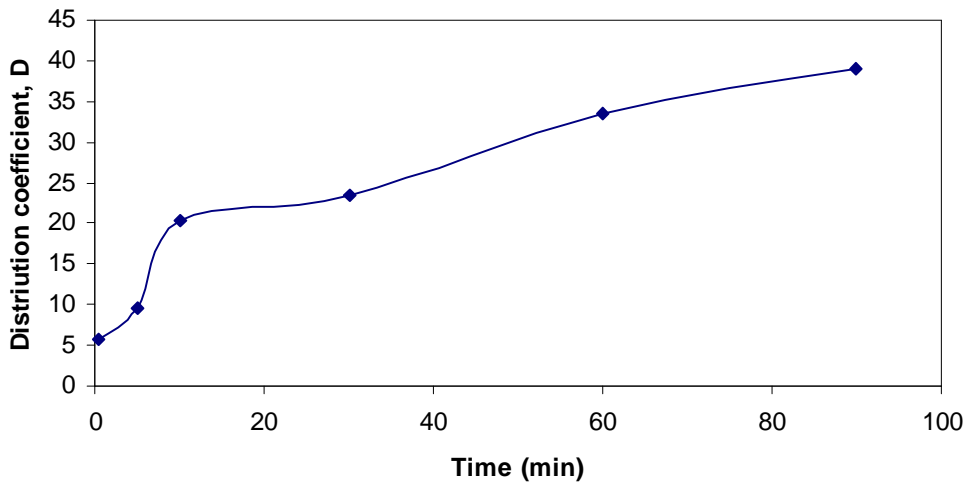


Figure 4: Distribution coefficient plot for Ni^{2+} adsorption

3.6 Adsorption kinetics

Pseudo-first order kinetics

The pseudo-first order kinetics is expressed in equation 4, where q_t and q_e (mg/g) are the amounts of metal ion adsorbed at time, t and at equilibrium respectively. k_1 is the rate constant for adsorption in (/min).

$$\log(q_e - qt) = \log q_e - \frac{k_1 t}{2.303} \quad 4$$

Plot of $\log(q_e - qt)$ versus t give straight lines (Figure 5). K_1 and q_e can be obtained from the slopes and intercepts respectively.

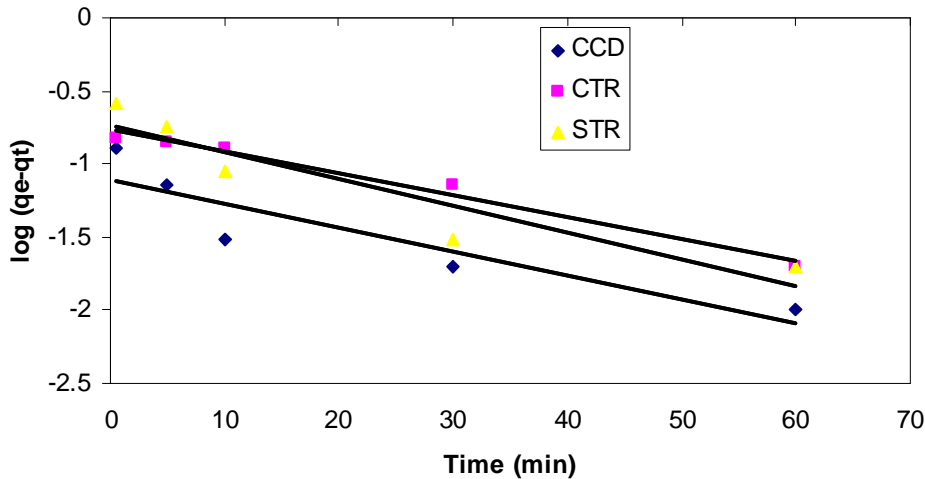


Figure 5: Pseudo-first order plot for adsorption of Ni^{2+} at $40^{\circ}C$

The values of the rate constant (Table 3) show that adsorption is independent of rate and can not be used to explain the process.

Pseudo-second order kinetics was also applied to quantify adsorption of Ni^{2+} by the resins. Figure 6 shows plot of pseudo-second order kinetics. Values of q_e and k_2 are obtained from slope and intercepts of the linear plots respectively.

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad 5$$

Parameters presented in Table 3 clearly show that the pseudo second order kinetics provides a better fit for the adsorption process. Linear correlation coefficient (R^2) values for the three resins are larger than similar ones for the first order models. Values are very close to unity, a justification of fitness of kinetic model. Again, q_e values for the second order model are in close agreement to the experimental values of 0.9591, 0.2980 and 0.2673 for CCD, CTR and STR respectively. The fit of the process to second order kinetic model also point hands to chemisorption (Etim *et al.*, 2012)

Table 3: Kinetic parameters for Ni²⁺ adsorption from aqueous solution

Pseudo-first order kinetics				
Adsorbent	Line equation	R ²	K ₁ (/min)q _e (mg/g)	
CCD	y = -0.063x - 1.1072 0.8284	0.145	0.239	
CTR	y = -0.015x - 0.7662 0.9846	0.042	0.344	
STR	y = -0.018x - 0.7336 0.8683	0.035	0.603	
Pseudo-second order kinetics				
Adsorbent	Line equation	R ²	K ₂ (g/mg min)	q _e (mg/g)
CCD	y = 1.0196x + 0.3687 0.9876	2.8196	0.9808	
CTR	y = 3.3223x + 19.80 0.9889	0.5574	0.3010	
STR	y = 3.6662x + 22.06 0.9999	0.6093	0.2728	

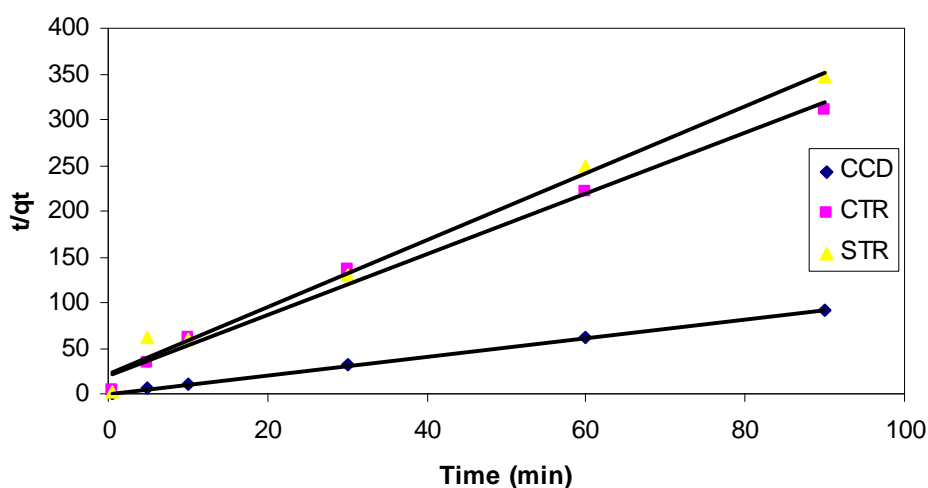


Figure 6: Pseudo-second order plot for adsorption of Ni²⁺ at 40°C.

3.7 Intraparticle diffusion model

The rate constant for intraparticle diffusion, K_a , obtained from the slope of the plot of q_e versus $t^{0.5}$ shows that the adsorption of metal ions follows a fast step and the rate determining step is always a slow one (Weng *et al.*, 2007). Figure 7 shows the plot for the determination of intraparticle diffusion constant and values obtained are 0.854, 0.906 and 0.953 for CCD, CTR and STR respectively. Since the plots do not pass through origin, the process is not a diffusion controlled mechanism. The intraparticle diffusions model is given as:

$$q_e = k_a t^{0.5} + I$$

6

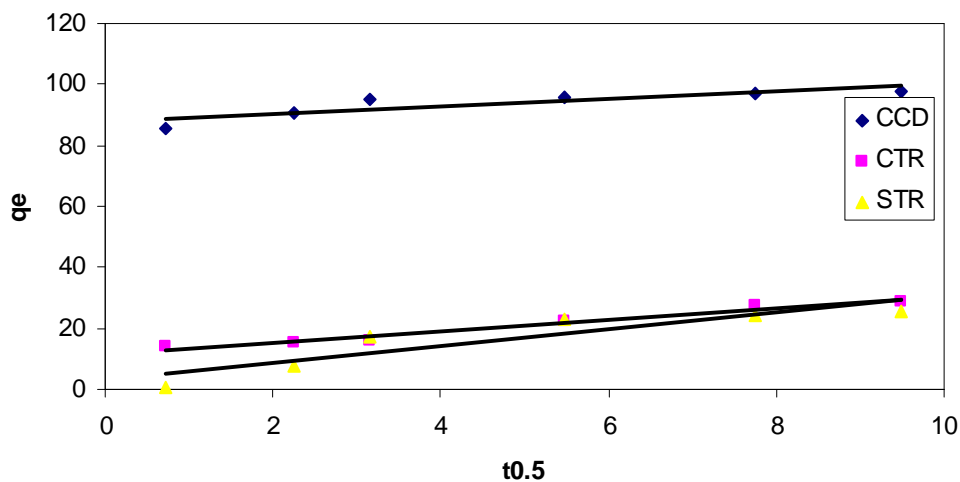


Figure 7: Intraparticle diffusion plot for adsorption of Ni²⁺ at 40°C.

3.8 Adsorption isotherms

Adsorption isotherm explains the relationship between the amount of substance adsorbed by an adsorbent and equilibrium concentration of species. Adsorption data have been analyzed using Langmuir and Flory-Huggins isotherm models.

Langmuir isotherm

The Langmuir equation (Langmuir, 1918) is based on the assumption that the surface of the biosorbent is homogeneous, all sites on it are identical and have equal affinity for the sorbate, and a complete monolayer is formed when the solid surface reaches saturation (Ferreira *et al.*, 2011). Its linearized form is given in equation 7:

$$\frac{C_e}{q_e} = \frac{1}{Q_o b} + \frac{C_e}{Q_o} \quad 7$$

where C_e is the equilibrium concentration (mg/L), q_e is the amount adsorbed (mg/g) at equilibrium, Q_o and b are monolayer adsorption capacity and Langmuir constant related to energy of adsorption calculated from. Figure 8 shows the Langmuir isotherm plot for adsorption of Ni²⁺ for CCD at 40°C. R^2 values are greater than 0.9 which is an indication of co-operative adsorption. Q_o and b are 1.66mg/g and 0.06L/mg respectively.

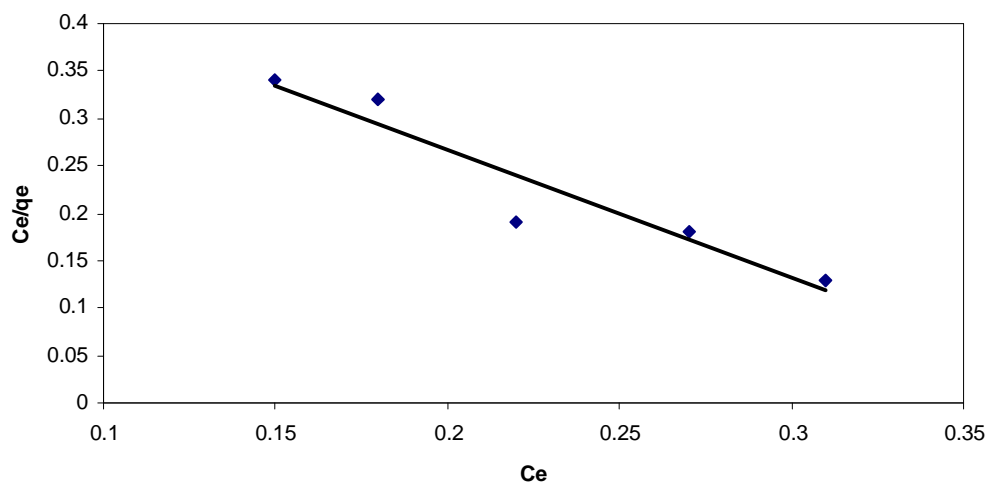


Figure 8: Langmuir isotherm plot for the sorption of Ni²⁺ at 40°C

Flory-Huggins isotherm

Flory-Huggins isotherm model (equation 8) was applied for adsorption of Ni²⁺ from aqueous solution in order to estimate the degree of surface coverage on the resins. A plot of $\log(\theta/C_e)$ against $\log(1-\theta)$ is presented in Figure 9 for STR. Only this resin shows regular adsorption as indicated by its adsorption parameters.

$$\log \frac{\Theta}{C_e} = \log K_{FH} + \alpha_{FH} \log(1 - \Theta) \quad 8$$

$$\Theta = (1 - C_e/C_0)$$

where α_{FH} is the number of metal ions occupying sorption sites, K_{FH} is the equilibrium constant of adsorption and C_0 is equilibrium metal ion concentration and Θ is the surface coverage of the adsorbents by the adsorbent and is defined as:

Θ = total amount of adsorbate per unit area/ total surface area of the adsorbate.

The thermodynamics of the adsorption can be elucidated from Flory-Huggins isotherm model. The value of standard Gibb's free energy change can be calculated from

$$\Delta G^\circ = RT \ln K_{FH}$$

Where ΔG° is the standard Gibb's free energy change and K_{FH} is Flory-Huggins constant, T is the temperature in Kelvin and R is molar gas constant. ΔG° for the process are -10.71, -35.73 and -48.24 for CCD, CTR and STR respectively. The negativity of the values of ΔG° indicates the spontaneity of the adsorption process.

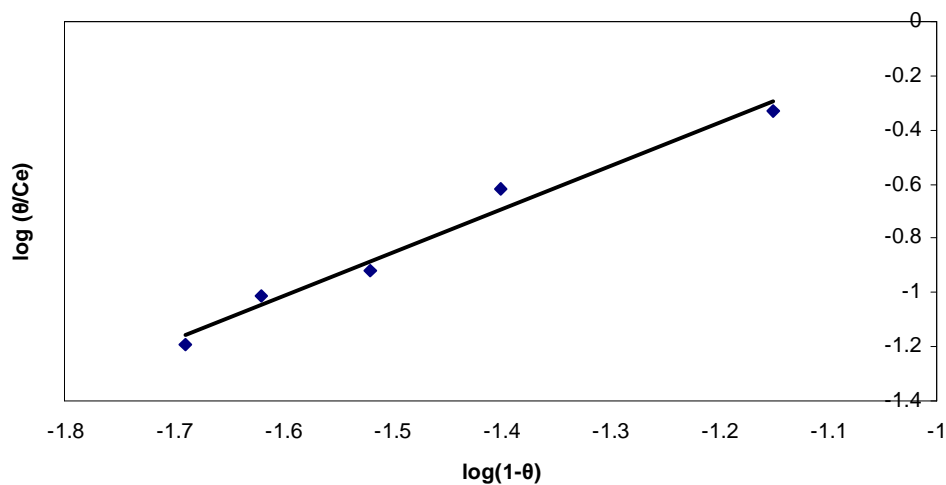


Figure 9: Flory-Huggins isotherm plot for the sorption of Ni^{2+} at 40°C

4.0 Conclusion

The adsorption and ion exchange removal of Ni^{2+} ion from its salt by modified coconut coir dust, carboxylated toluene resin (CTR) and sulphonated toluene resin (STR) has been successfully carried out via the variation of contact time and initial metal ion concentration. The following conclusion has been drawn from the research.

- i. The uptake of Ni^{2+} ion increases with an increase in contact time indicating better adsorption at higher contact times.
- ii. The adsorption capacity of coir dust for the removal of Ni^{2+} ion was found to be concentration dependent.
- iii. The adsorption process followed a second order kinetic and equilibrium sorption was better described with Langmuir and Flory-Huggins isotherm model.
- iv. The adsorption capacity value points to the use of coir dust as an efficient low cost and non-conventional adsorbent for ion exchange from solution.

5.0 References

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