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Adsorption of Nickel (II) ions from aqueous solution using Potassium Carbonate activated *Tectona grandis* leaves carbon

C. Pratheep [1], K. Ramesh[1]*, M. Kiruthiga [2], M. Saravanan [1], M. Sugirtha [1] and K. Suridha [1]

[1]Department of Chemistry, Poompuhar College (Affiliated to Bharathidasan University), Melaiyur, Nagapattinam, India
[2]Department of Chemistry, Government College for Women, Kumbakonam, Thanjavur, India

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ABSTRACT

Activated carbon has been prepared from *Tectona grandis leaves* and characterized. This carbon is ready to sorb metal ions from its aqueous solution. It has been noted that a increasing the carbon dose with constant Ni (II) ion solution concentration, or a rise in the Ni (II) ion solution concentration with constant carbon concentration resulted in a higher and lower nickel uptake per unit weight of carbon respectively. The Langmuir and Freundlich isotherm models of this adsorption system were studied and reported. The surface adsorption capability (Q_0) calculated from Langmuir isotherm was 38.025 mg Ni(II) g^{-1} at initial hydrogen ion concentration of 20 mg/l at 305 K for the particle size 150 300im. Dynamics of the system was studied with linearised forms of Lagergren, Ho and Webber Morris models.

Key words : *Adsorption, Carbonate activated*

Introduction

Fresh water is a renewable resource, when water is contaminated, it causes illness and disease. The treatment of coloured effluents has been investigated by various physico-chemical methods and purely chemical methods like coagulation, flocculation, adsorption, ion exchange, reverse osmosis and electrochemical coagulation in treating the textile several studies demonstrated the practicability of action and precipitation for colour removal (Gleeson *et al.,* 2012). Rebhun *et al*. (1970] found action on the C resulted in complete removal of the colour. The foremost wide used adsorbents are carbon because of their high area thanks to the presence of very little and meso pores kind of studies need to boot been performed follow carbon ready from agricultural wastes for the removal of dyes from liquid solution. Nickel is a silvery-white, hard, malleable, and ductile metal. It is of the iron group and it takes on a high polish. It is a fairly good conductor of heat and electricity. In its familiar compounds nickel is bivalent, although it assumes other valences. It also forms a number of complex compounds. Most nickel compounds are blue or green (Ghaleb *et al*., 2015). The major use of nickel is in the preparation of alloys, rechargeable batteries, catalysts and other chemicals, coinage, foundry products and plating. Nickel occurs combined with Sulphur in millerite, with arsenic in the mineral niccolite, and with arsenic and Sulphur in nickel glance (Moreno-Castilla, 2004). Most ores from which nickel is extracted are

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iron-nickel sulphides, such as Pentlandite. Foodstuffs naturally contain small amounts of nickel. Chocolate and fats are known to contain severely high quantities. Nickel uptake will boost when people eat large quantities of vegetables from polluted soils. Plants are known to accumulate nickel and as a result the nickel uptake from vegetables will be eminent (Abechi *et al*., 2011). Humans may be exposed to nickel by breathing air, drinking water, eating food or smoking cigarettes. Skin contact with nickel-contaminated soil or water may also result in nickel exposure. In small quantities nickel is essential, but when the uptake is too high it can be a danger to human health. An uptake of too large quantities of nickel has the following consequences: - Higher chances of development of lung cancer, nose cancer, larynx cancer and prostate cancer, Sickness and dizziness after exposure to nickel gas, Lung embolism, Respiratory failure, Birth defects, Asthma and chronic bronchitis, Heart disorders Nickel fumes are respiratory irritants and may cause pneumonitis. Exposure to nickel and its compounds may result in the development of a dermatitis known as "nickel itch" in sensitized individuals. The first symptom is usually itching, which occurs up to 7 days before skin eruption occurs. The primary skin eruption is erythematous, or follicular, which may be followed by skin ulceration. Nickel sensitivity, once acquired, appears to persist indefinitely. Carcinogenicity- Nickel and certain nickel compounds have been listed by the National Toxicology Program (NTP) as being reasonably anticipated to be carcinogens (Susan Verghese *et al*., 2016). The International Agency for Research on Cancer (IARC) has listed nickel compounds within group 1 (there is sufficient evidence for carcinogenicity in humans) and nickel within group 2B (agents which are possibly carcinogenic to humans) (Smith *et al*., 2000). *Tectona grandis* leaves is a tropical hardwood tree species in the family *Lamiaceae*. Recently, microwave energy has been wide employed in analysis and industrial processes (Ramesh *et al*., 2016). Compared with standard heating techniques, microwave heating has the subsequent further blessings as follows: interior heating, higher heating rates, selective heating, larger management of the heating process, no direct contact between the heating offer and heated materials, and reduced instrumentality size and waste. Therefore, microwave radiation is employed to arrange carbon from the material rather than customary heating methods (Qi Hu *et al*., 2021).

Experimental

Preparation of Activated Carbon

The activated carbon was synthesized using the following process: Initially, 25 g of *Tectona grandis* leaves were combined with a 25% K_2CO_3 (Donni Adinata *et al*., 2007) solution. The resultant slurry was left undisturbed at room temperature and atmospheric pressure for 24 hours, allowing the K_2CO_3 to fully react with the *Tectona grandis* leaves. After the sample had dried well, it was carbonized in a tubular furnace by heating for two hours at 500 °C while being exposed to an N2 environment. After carbonization, 0.5 M HCl was used to wash the samples, followed by hot distilled water and then cold distilled water until the pH of the solution reached 7. The carbon was filtered and dried at 150

Table 1. Data Processing Tools.

S. No.	Parameters	Formula	
1.	Mass balance relationships	% of Removal	$(C_i - C_t)100/C_i$
		Quantity adsorbed at equilibrium, q_e	$(C_i - C_e)\ V/W$
		Quantity adsorbed at the time t, q_t	$(C_i - C_t)\ V/\ W$
2.	Kinetic Models and SSE	Pseudo First order kinetics (Legergren equation) (Hamou Moussout *et al*., 2018).	$\log (q_e - q_t) = \log q_e - k_1\ /2.303 \times t$
		Pseudo Second order kinetics (Ho equation) (Nahid Ghasemi *et al*., 2013)	
		The initial adsorption rate h	$t/q_t = 1/\ k_2.q_e^{\ 2} + 1/q_e\ th = k_2 q_e^{\ 2}$
		Intra particle diffusion (Weber–Morri's equation) (Feng-Chin *et al*., 2009)	$q_t = k_p t^{1/2} + C$
		Mean Sum of error squares	$MSSE = \sqrt{\Sigma[(q_e)_{exp} - (q_e)_{cal}]^2}/\ N$
3.	Isotherms	Langmuir (Ramesh *et al*., 2016)	$C_e/Q_e = 1/Q_0 b + C_e/Q_0$
		Freundlich (Ramesh *et al*., 2016)	$\log Q_e = \log K_f + 1/n \log C_e$

Table 2. Nomenclature

C_i,C_t and C_e	Initial Concentration, at the time 't' and at equilibrium respectively
q_e and q_t	Quantity adsorbed at the time 't' and at equilibrium respectively
V	Volume of the metal ion solution in litter (L)
W	Mass of the adsorbent in gram (g)
Q_e	Amount of solute adsorbed per unit weight of adsorbent (mg/g)
C_e	Equilibrium concentration of solute in the bulk solution (mg/l)
Q_0	Adsorption efficiency
b	Adsorption energy
C_0	Initial concentration of the metal ion solution
K_f and n	The constants incorporating all factors affecting the adsorption capacity and intensity of adsorption respectively
q_m	Constant related to adsorption capacity (mg/g)
R	Gas Constant
T	Temperature (K)
k_1	Rate constant of adsorption (l/min)
k_2	Second-order constants
t	Time in minutes
h	Initial adsorption rate (mg/g min)
k_p	Intra-particle diffusion rate constant
C	Thickness of the boundary film
N	Number of data points
K_c	Equilibrium constant

°C for 5 hours. The prepared carbon was designated as TGLAC (*Tectona grandis* leaves activated carbon).

Adsorption Studies

Adsorption experiments were carried out by batch mode technique because of its simplicity. 50 mg of adsorbent was taken in 250 ml iodine flask and 50 mL of the dye solution was poured into the flask. Then the content of the flask was agitated using rotary shaker with 180 rpm for 180 min duration. Then1mL of aliquot was taken from sample and concentration of Ni (II) ions was estimated spectrophotometrically, the absorbance at 470 nm on UV spectrophotometer. The samples were withdrawn from the shaker at predetermined time intervals, the supernatant solution was separated by centrifugation at 180 rpm for 15 minutes and the remaining Ni (II) ions was analysed. The percentage removal of the Ni (II) ions from the solution was calculated by the mass balance relationship.

Results and Discussion

Effect of pH

It has been shown in the Figure 1 that the uptake of Ni depends on pH, it increases with the increase in pH and reaching maximum adsorption of Ni around pH 5. The continued increase in adsorptive capacities of Ni with increase in pH is due to the decrease in hydrogen ion concentration as pH value increases. At lower pH higher concentration of H+ ions present in aqueous medium will compete with the positively charged M^{2+} ion for the surface adsorbing sites thereby leading to a decrease in the removal of metal ions (Halil Hasar, 2003). In other words, under acidic condition solutions both adsorbent and metal ions are positively charged (M^{2+} and H^+) and hence their interaction is via electrostatic interaction. The final pH values at equilibrium after adsorption were lower than the initial pH value, indicating that the Ni ions are adsorbed and hydrogen ions are released from the adsorbent. This is also confirmed by the result that the increase of acidic

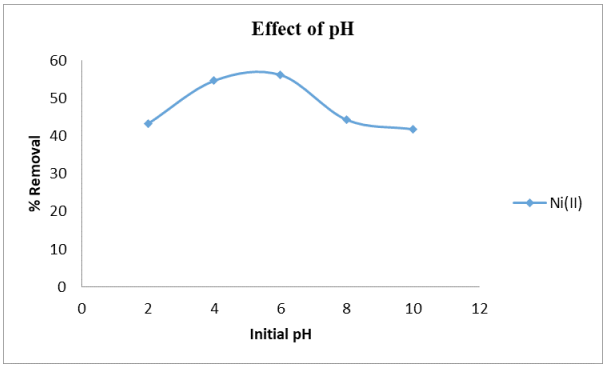

Fig. 1. Effect of pH for Ni(II) ions onto TGLAC

surface groups favoured the adsorption process. The effect of pH on Ni (II) adsorption onto TGLAC was studied over a pH range 2-10 at 305K in order to avoid metal hydroxides (Halil Hasar, 2003).

Effect of adsorbent dosage

The adsorption of Ni (II) onto TGLAC was studied by varying the dose of the adsorbent from 10 mg/50 ml to 30 mg/50 ml by taking 50 mg/l of Ni (II). The percentage of removal of Ni (II) from aqueous solution increased with an increase of carbon dose are shown in Figure 2. This is due to the increased carbon surface area and the availability of more adsorption sites. The curve is smooth for TGLAC indicating the uniform adsorbing sites. Based on these results, the remaining parts of the experiments were carried out with the adsorbent dose of 25 mg/ 50 ml for both the adsorbent TGLAC which gave the percentage of removal between 60 and 70% (Manal El-Sadaawy and Ola Abdelwahab, 2014).

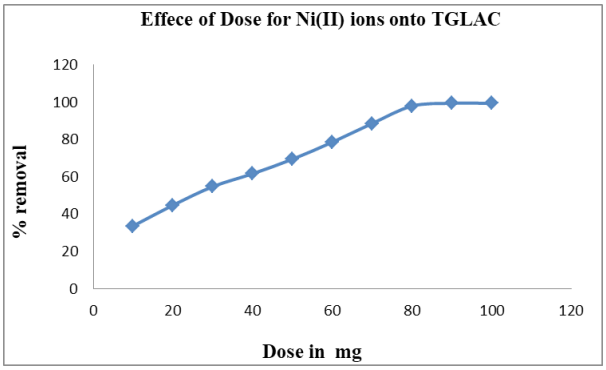

Fig. 2. Effect of Dose for Ni(II) ions onto TGLAC

Effect of contact time

The effect of contact time on the percentage removal of Ni(II) ion from aqueous solution was studied by taking 10 mg/l, 20 mg/l and 30 mg/l solutions as initial concentrations for TGLAC. The results of the above study are shown in Figure 3. The extent of metal ions removal by activated carbon increased with the increased of contact time. The removal of metal ions by adsorption using activated carbon was found to be rapid at the initial period of contact time and then become slower with the increase of contact time. At the initial stage, the ratio of surface area of the adsorbent to the amount of solute in liquid phase is high and hence the concentration driving force makes solute to rush towards the adsorbent surface (Kadirvelu *et al*., 2001).

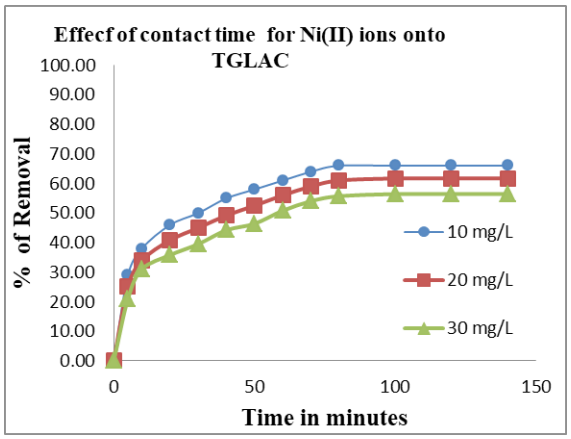

Fig. 3. Effect of contact time for Ni(II) ions onto TGLAC

As the time increases the above ratio begins to decrease due to adsorption and hence the rate of adsorption becomes slow. This is due the strong attractive forces between the metal ions and the adsorbent. The results showed that the contact time required to achieve equilibrium was about 80 minutes for adsorption using TGLAC. This may be due to difference in pore structure and sizes as a result of activation treatments. The results strongly indicate that the activated carbons used in this study are effective in removing Ni (II) ion

Effect of initial concentration

The percentage of the removal of metal ions decreased with the increase of initial concentrations of adsorbate solution. This is because of the following reason. The amount of solute in the liquid phase is low at a lower of initial concentration and the ratio of available adsorbent surface to the concentration of solute is high. Hence large fraction of solute transferred to the adsorbent. But at higher initial concentration the amount of solute in the liquid phase is high and the ratio of available adsorbent surface to the concentration of solute is low and hence small fraction of solute transferred to the adsorbent.

However, the quantity adsorbed by the adsorbent increased with the increase of initial concentration of adsorbate solution. This is because the amount of solute adsorbed is proportional to the fraction of adsorbate transferred from liquid phase to solid phase. This fraction increases with an increase in the concentration of solution. The time to attain equilibrium was found to increase when the initial concentration of the metal ions increased. This is because an additional amount of solute to be adsorbed, take

extra time to penetrate into the pore and find the place to get adsorbed (Halil Hasar, 2003).

Adsorption Kinetic study

Pseudo first- and second-order and intraparticle diffusion kinetic models were tested against the experimental data to explicate the kinetics of the adsorption process. The adsorption was rapid during 30 minutes of contact time, and then slowed, because numerous vacant surface sites were available for adsorption during the initial stage, and then, the remaining vacant surface sites could not be easily occupied because of the repulsive forces between the adsorbates on the adsorbents and in the bulk phase.

Pseudo first and second-order plots drawn for both adsorbents are depicted in Figure 4 & 5. The different theoretical values of the constants linked to the Legergren first order and the second-order models are determined using the slopes and intercepts of these model fits, and are recorded in Table 3 .The regression coefficient values (R^2) are around 0.9 describe the well-fitting of the experimental data; moreover, the $q_{(cal)}$ value calculated from the pseudo second-order model was consistent with the experimental $q_{(exp)}$ values for both adsorbent TGLAC and the SSE (%) values are small for the pseudo second order kinetic model. SSE values for pseudo second order model is 0.89 for TGLAC, where as these values for pseudo first order model is for TGLAC. Accordingly, this study suggests that the pseudo second-order model best represented the adsorption kinetics TGLAC (Hamou Moussout *et al*., 2018; Nahid Ghasemi *et al*., 2013).

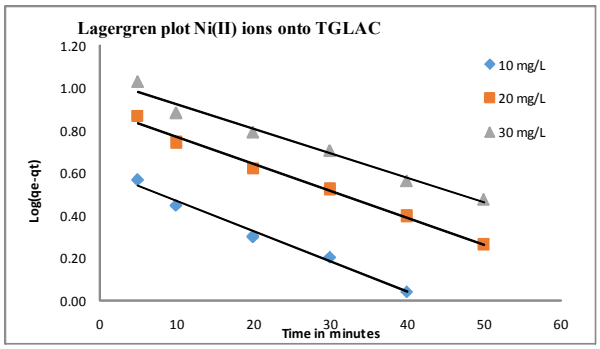

Fig. 4. Legergren for Ni(II) ions onto TGLAC

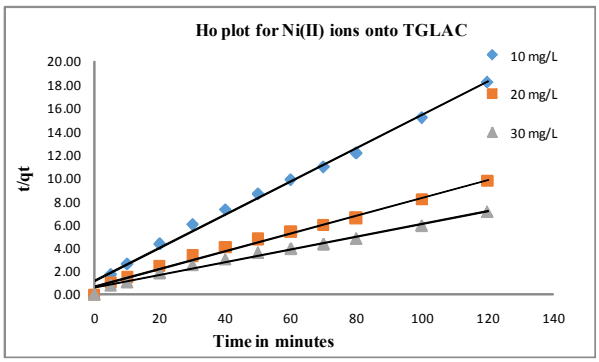

Fig. 5. Ho plot for Ni(II) ions onto TGLAC

Intra Particle Diffusion

Various mechanisms determine the adsorption kinetics; the most limiting mechanisms are the diffusion mechanisms, including external diffusion,

Table 3. Pseudo first order Kinetics results for Ni (II) ions onto TGLAC
[pH = 7; Dose = 25 mg/ 50 ml; Agitation time = 160 min; Temp=305K]

Initial Concentration C_i(mg/l)	Pseudo First Order Kinetics					
	$k_1\times10^{-2}$ (min^{-1})	$q_{e(cal)}$ (mg/g)	$q_{e(exp)}$ (mg/g)	Δq_e	R^2	SSE
10	0.0329	4.08	6.60	6.30	0.9863	2.63
20	0.0290	7.86	12.34	20.01	0.9904	
30	0.0265	10.93	16.92	35.83	0.9772	

Table 4. Pseudo second order Kinetics results for Ni (II) ions onto TGLAC
[pH = 7; Dose = 25 mg/ 50 ml; Agitation time = 160 min; Temp=305K]

Initial Concentration C_i(mg/l)	Pseudo Second Order Kinetics					
	$k_2\times10^{-3}$ (g/mg.min)	$q_{e(cal)}$ (mg/g)	$q_{e(exp)}$ (mg/g)	Δq_e	R^2	SSE
1020	0.0182	7.01	6.60	0.17	0.9929	0.53
	0.0082	13.19	12.34	0.73	0.9898	
30	0.0053	18.21	16.92	1.68	0.9864	

boundary layer diffusion and intraparticle diffusion. If the plot of q_t as a function of $t^{0.5}$ is linear and passes through the origin, then intraparticle diffusion is the sole rate-limiting step. Therefore, the intraparticle diffusion model was applied to determine the rate limiting step of the adsorption process. The values of kd were calculated from the linear portion of the graphs by drawn between q_t and $t^{0.5}$ which are shown in Figure 6. In the intraparticle diffusion plots, the first, sharper region is the instantaneous adsorption or external surface adsorption. The second region is the gradual adsorption stage where intraparticle diffusion is the rate limiting. In some cases, the third region exists, which is the final equilibrium stage where intraparticle diffusion starts to slow down due to the extremely low adsorbate concentrations left in the solutions. As seen from Figure 6, the plots were not linear over the whole-time range, implying that more than one process affected the adsorption. In the Table 5, the k_p values were found to increase with an increase of Ni (II) concentration which revealed the rate of adsorption governed by the diffusion of adsorbed Ni (II) within the pores of the adsorbent (Feng-Chin *et al.*, 2009).

Table 5. Intra Particle Diffusion results for Ni (II) ions on TGLAC

[pH = 7, Dose = 25 mg/ 50 ml, Agitation time = 160 min; Temp=305K]

Initial Concentration C_i(mg/l)	Intra Particle Diffusion	
	k_p (mg/g.min)	R^2
10	0.4737	0.9935
20	0.9082	0.9989
30	1.268	0.9891

Equilibrium studies

Adsorption of heavy metal ion/dye is considered to be a fast physical/chemical process, it is a collective term for a number of passive accumulation processes which include ion exchange, co-ordination, complexation, chelation, Vanderwaal's attraction and micro precipitation. Proper analysis and design of adsorption separation processes require relevant adsorption equilibria as one of the vital information.

In equilibrium, certain relationship prevails between solute concentration in solution and in adsorbed state (i.e., the amount of solute adsorbed per unit mass of adsorbent). Equilibrium concentrations are the function of temperature. Therefore, the adsorption equilibrium relationship at a given temperature is referred to as adsorption isotherm.

Adsorption Isotherms

Adsorption isotherm is basically important to describe how solutes interact with adsorbents, and is critical in optimizing the use of adsorbents. Adsorption isotherm study is carried out on two well-known isotherms, Langmuir and Freundlich isotherms. Applicability of each isotherm gives certain information about the adsorption. Inference obtained from each isotherm is discussed in detail one by one.

Langmuir isotherm

The results obtained from Langmuir model for the removal of Ni (II) ion onto TGLAC are represented in Table 6. Concerned isotherm plots were shown in Figure 7.

The mono layer adsorption capacity q_m values (mg/g) for adsorption of Ni (II) ion onto TGLAC system seems to have a higher adsorption capacity

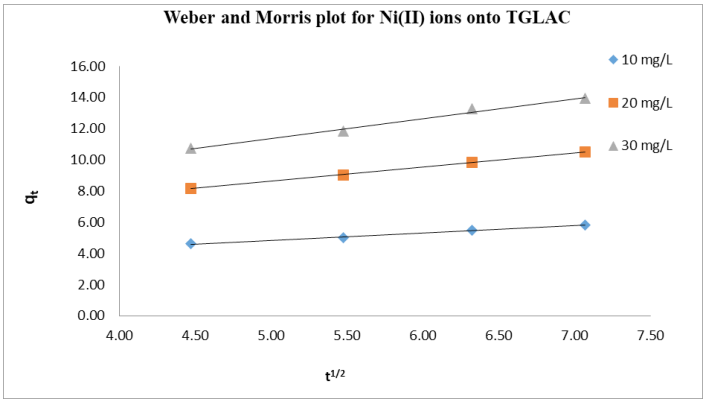

Fig. 6. Weber and Morris plot for Ni(II) ions onto TGLAC

Table 6. Langmuir isotherms for Ni (II) ions onto TGLAC
[Ni(II) ions, pH = 7, Dose = 25 mg/ 50 ml]

Adsorbents	Temperature (K)	Q_0(mg/g)	b(L/mg)	R^2
TGLAC	305	37.313	0.068	0.9995
	315	37.453	0.063	0.9988
	325	37.736	0.071	0.9997
	335	38.023	0.076	0.9997

Table 7. Freundlich isotherms for Ni (II) ions onto TGLAC
[Ni (II) ions, pH = 7, Dose = 25 mg/ 50 ml]

Adsorbents	Temperature (K)	n	K_f(mg/g)	R^2
DRPAC	305	1.4192	2.8294	0.9951
	315	1.4341	2.9888	0.9956
	325	1.4428	3.1485	0.9957
	335	1.4533	3.3481	0.9955

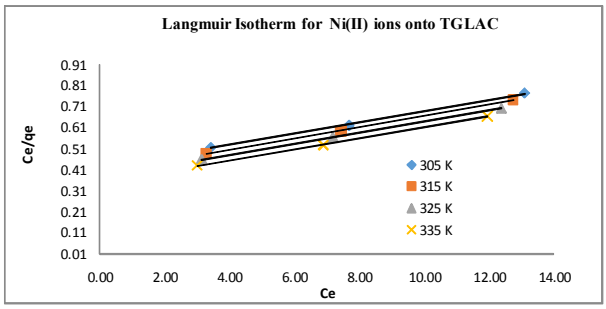

Fig. 7. Langmuir Isotherm for Ni(II) ions onto TGLAC

with respect to the adsorption of Ni (II) ion for all the studied temperatures.

Further it is noticed that adsorption capacity in slightly increased with an increase of temperature. Table 6 reveals that adsorption capacity of TGLAC are quite reasonable when compared to adsorption capacities of other adsorbents at room temperature for the adsorption of Ni (II) ion reported in previous studies

The low values of Langmuir constant 'b', the adsorption energy ranges indicate that the apparent energy of sorption is less and rules out the possibility of strong interaction between the solute and adsorption site.

The regression coefficient (R^2) values are ranged from 0.9 to 1 for the four studied temperatures viz. 305, 315.325 and 335 K for the adsorbent TGLC with Ni (II) ions. These results show the best fitting of the equilibrium data with Langmuir isotherm (Ramesh *et al.*, 2016).

Freundlich isotherm

The results obtained from Freundlich model are given in Table 7. The concerned isotherm plots are shown in Figures 8.

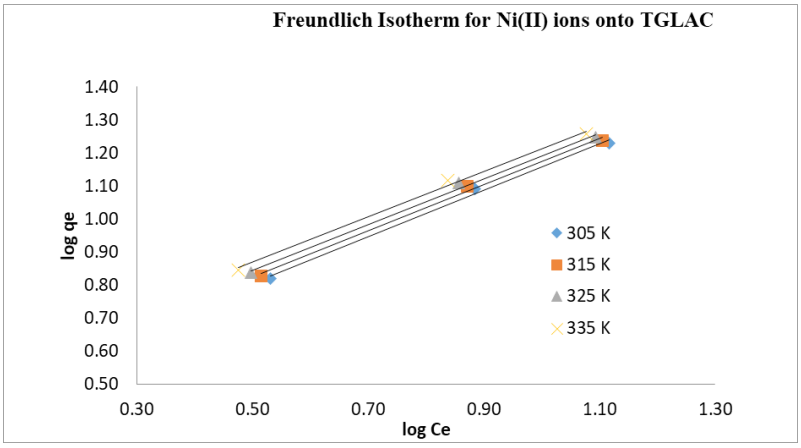

Fig. 8. Freundlich Isotherm for Ni(II) ions onto TGLAC

The regression coefficient (R^2) for Freundlich isotherms is around 0.9 for all the studied temperatures viz. 305, 315 and 325 and 335 K. It indicates that the experimental data fit well into Freundlich model.

TGLAC seems to have s higher adsorption capacity k_f (mg/g) for all the studied temperatures. Further it is noticed that the adsorption capacity increased with the increase of temperature.

The adsorption intensity constant 'n' values are in between 1 and 10, which indicates the favourable physical adsorption. 'n' value increases with an increase of temperature for all the studied systems (Ramesh *et al*., 2016).

Conclusion

In the present study Potassium Carbonate activated *Tectona grandis leaves* carbon (TGLAC) was prepared and investigated its potential to abate chosen adsorbate Ni(II) ions from aqueous solution. The values of n were found to be greater than one indicating a favourable adsorption. Theoretically evaluated quantity adsorbed at equilibrium from first order and second order rate equations were compared with the quantity adsorbed at equilibrium in the actual experiments. The statistical tool 'Summation of error square test' revealed that present studied all adsorbent- adsorbate system followed second order kinetics. The variation of k_p values was proportional to the initial adsorbate concentrations which indicates that the intra-particle diffusion limits the rate of the process.

Conflict of Interest : None

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