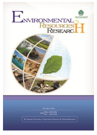

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Application of Nano-lignocellulose for Removal of Nickel Ions from Aqueous Solutions

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Abstract

Nickel is one of the toxin heavy metals in surface waters. Developing new approaches for removing heavy metals from aqueous solutions that are simultaneously economical and environmentally friendly is of great importance. The purpose of this study was to use Nano-lignocellulose adsorbent as a natural material to remove nickel heavy metal. In this study, several important environmental parameters were studied including pH, adsorbent concentration, duration of exposure, concentration and temperature on the uptake, as well as adsorption and Kinetic isotherms. The maximum adsorption efficiency (99.5%) occurred at pH=6. Optimum contact time was measured 30 minutes, which reflects the short duration of the analysis. Results of researches in the field of adsorption isotherm show that, the best fit of the experimental data corresponds to the Langmuir isotherm model. Kinetic studies revealed that the process of elimination well matched with pseudo-second-order kinetic model. So, the Nano-lignocellulose has high ability to remove nickel from aqueous solutions and as a new adsorbent could be used in different industrial effluents.

Keywords: Heavy Metals, Nickel, Nano-lignocellulose, Aqueous Solutions, Adsorption

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1. Introduction

Pollution is one of the most important factors that results in waste of water resources. With the rapid development of science and technology in society, and increased quantity and quality of industrial production, the use of heavy metals has grown dramatically. This has led to the increased concentration of these metals in the environment, particularly aquatic environment (ZavarMousavi and Arjmand, 2010). The importance of preventing the entry of heavy metals into the environment is that, these elements are toxic for environment even at low concentrations. Heavy metals cannot be decomposed in the same way as organic pollutants, namely through chemical reactions or biological processes in nature. Only the nature of these compounds changes in the sense that metals still remain in the environment (Ballay *et al*., 2015).

Nickel is one of the most important heavy metals. If this nickel finds its way to the human body through the food chain or close contact, it will cause severe damage to the heart and liver, while intensifying allergies, cancer and respiratory disorders (Ghafourian *et al.*, 2015). The maximum allowable amount of nickel is 0.07 mg/L. Increasing the value in plants or animals, will result in irreparable damages (Seyyedi, 2011).

Removal of heavy metals through application of various adsorbents has been of interest to many researchers. Akpen *et al.* (2012), used the coconut coir as an eco-friendly biomaterial for the removal of nickel in the aquatic environment. Liu *et al.* (2013) applied the chitin nanofibers to quickly and effectively remove cadmium, nickel, and zinc and lead bivalent and trivalent chromium ions in aqueous systems. Manal *et al.* (2014), to remove nickel from aqueous solutions, used palm kernel shells and seeds as activated carbon. Ravindra *et al.* (2015), used magnetic nanoparticles with the aim of the removal of nickel in an aqueous medium. Their results showed that these small particles are capable of absorbing nickel from aqueous solutions through the simple eco-friendly method of Freundlich deposition method and thermodynamic studies. Swat *et al.* (2014), in their study used activated carbon produced from canola's oil cake to remove nickel ions from aqueous solutions. Their results showed that according to the Freundlich model, activated carbon is environmentally and economically useful and cost-effective for the sequestration of nickel.

A variety of methods have been developed to remove heavy metals, including sequestration, filtration, and oxidation-reduction, ion exchange and membrane separation. When metals are soluble in high volume and low concentration, these methods are often ineffective and expensive. Among these, the adsorption method is more commonly used. Biosorption is an effective technology to remove heavy metals from aqueous solutions. In this process, a very high rate of adsorption and excretion occurs. The simple function has caused this method to be one of the best ways to remove toxins from nature (Marandi *et al.*, 2008).

Therefore, inventing new ways to remove heavy metals from aqueous solutions that are simultaneously economical and environmentally friendly, is of great importance and urgency. Thus, this study tried to use a new method to remove or reduce the amount of heavy metals in aqueous solutions. In this study, Nano-lignocellulose was used to remove nickel heavy metal. Nano-lignocellulose, as a valuable natural material, is derived, mechanically concomitant with various treatments, from plant fibers (hardwood), wood (bamboo, hemp, corn, bagasse, etc.), Kinetic fibers such as polyacrylamide and poly Vinyl. There is no report about usage of Nano-lignocellulose as an adsorbent for removal of heavy metals. Also, this adsorbent has no disadvantage for the environment which is its stength. Hence, the approach can be regarded as new in the field of adsorption.

2. Materials and Methods

2.1. Preparation of adsorbent Nano-lignocellulose

Nano-lignocellulose has been acquired from the Nano Novin Polymer company. Nano-lignocellulose was produced from poplar, beech and hornbeam wood. This compound was selected due to its various advantages compared to other natural Nano-adsorbents in the adsorption process of nickel. In preparing this adsorbent, lignocellulose pulp was prepared from thepseudo-chemical neutral sulfite process containing unbleached cellulose fibers (including lignin and hemicellulose) from wood and paper company located in Mazandaran province. This paste was washed several times with water and then a lignocellulose suspension at a concentration of 1% was prepared andtransferred througha Disk mill machine (MKCA6-3; Masuko Sangyo Co., Ltd., Japan) in order for the Nano-lignocellulose to be prepared.

2.2. Sample preparation to obtain the FTIR, XRD spectrum and TEM images

For the preparation of the samples, after completion of all the tests and determination of the optimum quantity of each parameter, testing was performed. First, 5 mg/L of nickel was made up to the volume and then the pH was set to 6. Then, 0.1 g Nano-lignocellulose was added to Nickelsolution solute and the mixture was stirred at 25 ° C for 60 minutes. Eventually, flasks were removed from the shaker and using centrifuges, adsorbent and dissolved metal ions were separated. Remainingadsorbent was dried after separation from the solution in a glass Petri dish and sent to be imaged.

2.3. Adsorption ofnickel usingNano-lignocellulosein a batch system

To investigate the effects of variables of pH, time, temperature, adsorbent dosage, initial concentration of nickel, 100 ml of metallic nickel was added to a 250 ml flask and a certain amount of adsorbent was added to each flask to be mixed. After the reaction time, the adsorbent was separated from the solution by centrifugation at around 4000 rpm for 5 minutes. The supernatant liquid phase was collected and read by atomic adsorption. All adsorption experiments were conducted in a batch system. The range of parameters is presented in Table 1.

Table 1. Range of studied parameters for nickel adsorption by Nano-lignocellulose

Adsorbent dosage (gr)	time (Min)	temperature (^{O}C)	Initial concentration (mg/L)	pH	Variable
0.1	60	25	5	4-8	pH
0.1	60	25	5-50	6	initial concentration
0.1	60	15-40	5	6	Temperature
0.1	15-120	25	5	6	Time
0.1-0.5	60	25	5	6	adsorbent dosage

2.4. Adsorption isotherm

There are many models that can express the relationship between adsorption and residual concentration in solutions. Langmuir and Freundlich are more common and acceptable adsorption isotherms for the measurement of adsorption of heavy metals in aqueous solutions (Deobandi *et al.*, 2012).Therefore, in this study Langmuir and Freundlich models have been applied to investigate and analyze experimental data and descriptions of the adsorption equilibrium between solid and liquid phases.

2.4.1. Langmuir isotherm

Langmuir is designed for the monolayer adsorption on the surface for a limited number of places, unevenly distributed on the surface of the adsorbent. This hypothesis is based on the fact that adsorption takes place in certain homogeneous places (Yang *et al.*, 2014). Langmuir isotherm for the adsorption ofNano-lignocellulose and its related parameters are given in the equation (1).

$$\frac{C_e}{q_e} = \frac{C_e}{q_{max}} + \frac{1}{b.q_{max}} \qquad \text{eq. (1)}$$

Where $C_e$is the equilibrium concentration of absorbable ions in milligrams per liter, q_e is the mass of metal adsorbed onto the adsorbent (mg.gr^{-1}; the amount of pollutants absorbed), q_{max} is the maximum adsorption capacity in terms of (mg.gr^{-1}) and b is the adsorption equilibrium constant in terms of L.mg^{-1} (at a given temperature; related to theadsorptionenergy (the attraction between adsorbent and adsorbent). After plotting linear function of Langmuir by C_e/q_e against C_e, qmax (maximum adsorption capacity) and b (Langmuir adsorption constant)values are respectively, obtained from the intercept and slope (Jafari *et al.*, 2013).

2.4.2. Freundlich isotherm

This is based on the multilayer and heterogeneous adsorption as well as uneven surface of the adsorbent material. In fact, unlike the Langmuir, Freundlich isotherm is used to describe heterogeneous systems. Adsorbent surface has different places with different potential energy where each absorbed molecule shall be assigned to multiple locations (Javanbakht *et al.*, 2012). Freundlich isotherm for the adsorption Nano-lignocelluloses and its related parameters are provided in the equation (2).

$$Lnq_e = LnK_f + \frac{1}{n} LnC_e \qquad \text{eq. (2)}$$

Where n is Freundlich equilibrium constant that expresses the links between energy and adsorbent metal (adsorption intensity), kf is Freundlich isotherm constant which represents adsorption strength ($mg.gr^{-1}$) (adsorption capacity), C_e is equilibrium concentration of adsorbent ions ($mg.l^{-1}$), q_e is the mass of metal adsorbed onto the adsorbent ($mg.gr^{-1}$). By drawing a Freundlich line graph between Log $q_e$and Log C_e, n and Kf parameters are obtained respectively from slope 1/n and Log $K_f$intercept (Kiarostami *et al.*, 2013).

2.5. Kinetic adsorption models

One of the important studies in the field ofadsorption, is the assessment of the effect of contact time with the adsorption level, which is called Kinetic studies. To investigate the mechanism of adsorption and controlling the response time, Kineticstudies are necessary (Lowe *et al.*, 2014).

Since the most commonly used Kinetic models foradsorption are pseudo-first-order and pseudo-second-order models, these models have been applied to evaluate the adsorption mechanism. In equations (3) and (4), these models are described.

$$\log(q_e - q_t) = log q_e - \frac{k_1}{2.303} t \qquad \text{eq. (3)}$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \left(\frac{1}{q_e}\right) t \qquad \text{eq. (4)}$$

In this equation, q_e and q_t are respectively the adsorption capacity in equilibrium at time t (min), and K1 is speed ratio per minute. In the pseudo-first-order model, q_e and K_1 values are respectively intercept and slope obtained from plotting Ln (qe-qt) vs. t. Q_e and k2 values (pseudo-second reaction constant) in $mg.gr^{-1}.min^{-1}$ are respectively the slope and intercept obtained from linear graph of t/q_t versus t (Shahmohammadi Klalaq *et al.*, 2011).

2.6. Adsorption thermodynamics

Thermodynamics is a branch of the natural sciences discussing heat and its relation to energy and movement. In the adsorption assays in the range of 10-40°C temperature, the evaluation of thermodynamics criteria was conducted by the measurement of the Gibbs' free energy (ΔG). Gibbs' free energy is obtained from equation (5) as follows.

$$\Delta G = -RTMK_C \qquad \text{eq.(5)}$$

Where T is temperature (K), R is ideal gas constant equal to 8.314 $J.mol^{-1}.K^{-1}$. KC is the thermodynamic equilibrium constant which is obtained from equation (6) (Ghasemi *et al*., 2012).

$$K_c = \frac{C_a}{C_e} \quad \text{eq.(6)}$$

Where C_a is the final equilibrium concentration of metal, C_e is mg absorbing material absorbed in the solution. According to the laws of thermodynamics, Gibbs' free energy at a constant temperature is associated to enthalpy and entropy changes which is expressed as Van't Hoff equation (eq. 7).

$$LnKc = \frac{-\Delta H^{\circ}}{R}\frac{1}{T} + \frac{\Delta S^{\circ}}{R} \quad \text{eq.(7)}$$

Where ΔH and ΔS are respectively obtained from the slope and intercept of LnK_c graph based on 1/T.

3. Results and discussion

In this section, the results of the optimization of parameters, affecting the process of elimination, are presented. In the batch system, the effect of various parameters such as pH, initial concentration of nickel, and adsorbent dosage on the removal efficiency of Nano-lignocellulose is investigated. In the next step, by determining the optimal adsorption parameters, effect of time on the nickel adsorption by Nano-lignocellulose was studied. Finally, equilibrium and adsorption Kinetic models were studied. Moreover, the effect of temperature and thermodynamic parameters were determined, as provided below.

3.1. Effect of pH

One of the most important parameters in the process of adsorption is the initial pH of the solution. The removal efficiency and adsorption capacity for nickel, with the range of changes in pH, is provided in figure 1. As is clear, by increasing pH, nickel removal capacity increases, so that at pH 6, removal and adsorption capacity reaches respectively 99.5 and 4.97 $mg.gr^{-1}$, i.e. their maximum level. Then at pH of 7 and 8, adsorption efficiency and capacity reduces. The data showed that the adsorption of nickel is dependent to pH, so that it affects the characteristics of the adsorbent surface and adsorption efficiency. According to the above arguments, pH 6 is selected as the optimal value. Increasing pH give rise to net negative charges on the surface of the adsorbent. This leads to intensification of electrostatic forces in the adsorption process and thus more efficient remove of metal ions (Rozin *et al.*, 2010).

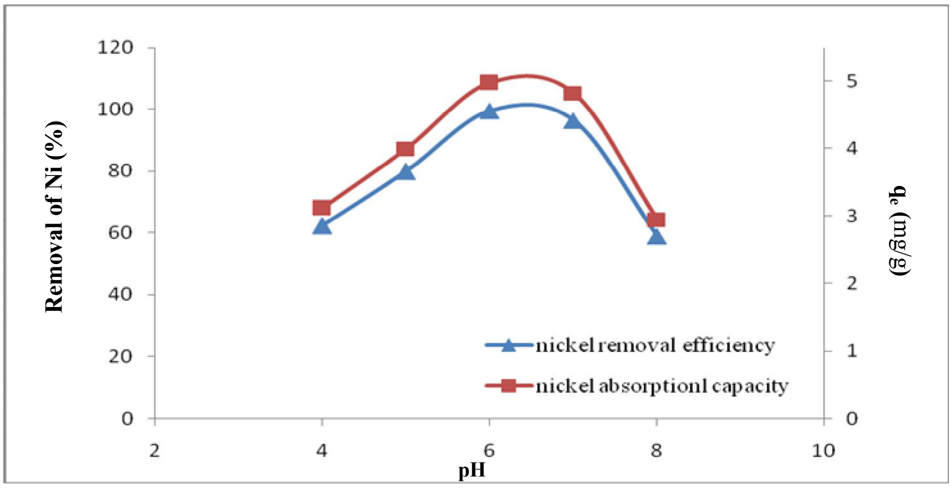

Figure 1. Effects of pH on the Adsorption of nickel by Nano-lignocellulose

2.3 The effects of the initial concentration of nickel

The effect of initial concentration of nickel for the removal and adsorption capacity of Nano-lignocellulose is shown in Figure 2. As can be seen, with increasing initial concentration of nickel, adsorption capacity and efficiency decreases. In fact, the change in the initial concentration of nickel is inversely proportional to the capacity of adsorption and removal. Given that at the concentration of 5 mg, 99.8% of the nickel was removed and then with increasing concentrations from 5 to 50 mg adsorption significantly decreased, concentration of 5 mg/L was selected as the optimal concentration.

It could be said that at the lower concentrations of nickel, more receivers are available for the adsorption of metal cations. As a result, nickel ions are simply able to be attached to the receivers on the surface, and thus, the efficiency of adsorption increases. However, at higher concentrations, metal cations are far beyond the capacity of the receivers. As a result, competition for access to the contact area increases and all binding sites become exposed to ions and turn activated. The findings of this study is consistent with that of Shokouhi *et al.* (2011) and Kumar *et al.* (2014).

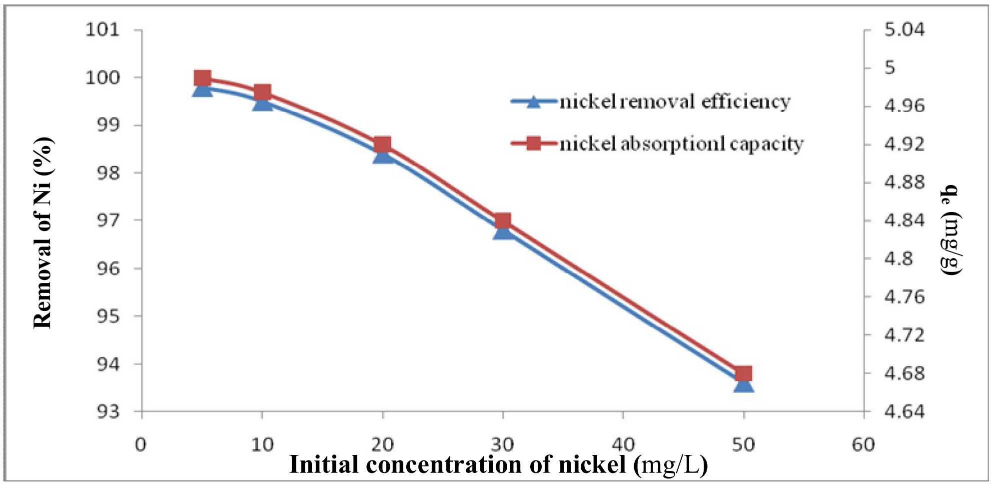

Figure 2. Changes in initial concentration of nickel and its effects on nickel adsorption by Nano-lignocellulose

3.3. The effect of adsorbent dosage

Changes in the efficiency and adsorption capacity of nickel, with variation in the dose of adsorbent are provided in Figure 3. Adsorbent dosage increase is directly proportional to the nickel adsorption efficiency and capacity. With the increase of adsorbent from 0.1 to 0.5gr, the efficiency of adsorption increases from 99.5% to 100%, while adsorption capacity increases from 4.97 to 5 mg. As can be seen, with increasing adsorbent dose from 0.1 to 0.5 gr, little change occurs in nickel uptake efficiency and capacity. Considering the foregoing and also by taking into account the cost of the absorber, 0.1 g dose was selected as the optimal value for nickel adsorbent.

As can be seen, in this study, by increasing the amount of adsorbent, nickel removal efficiency increased, because the number of available adsorption positions increases and thus so does the adsorption efficiency and capacity. However, increasing the amount of adsorbent, the chance foradsorbent to encounter with nickel cations increases, and hence nickel uptake increases. So the most important factor in increasing the efficiency of adsorption, pertains toaccelerated availability of the exchange area athigher level of concentration (Suman *et al.*, 2014).

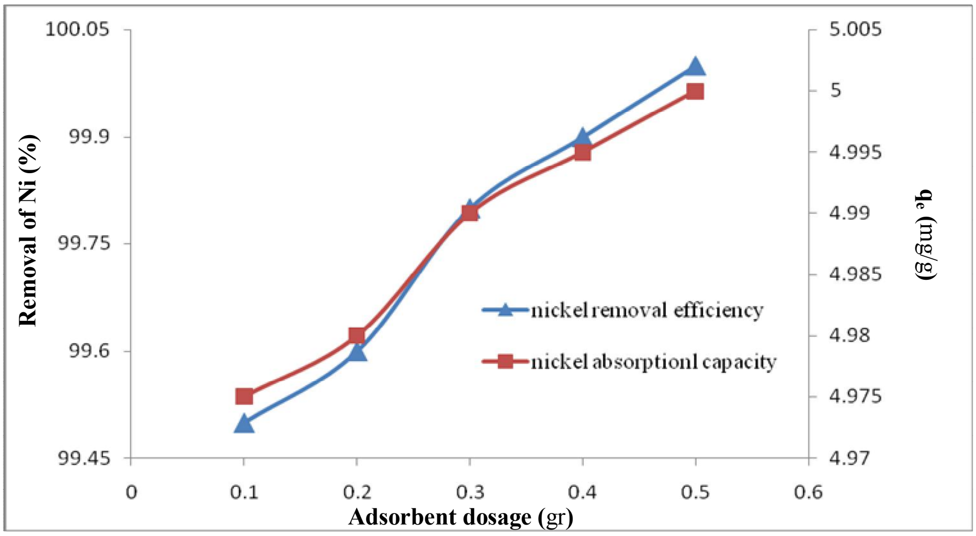

Figure 3. Effects of Nano-lignocellulose adsorbent dosage increase on the adsorption of nickel

3.4. The Effect of temperature

Efficiency and adsorption capacity of nickel with temperature changes is shown in figure 4. As can be seen, temperature increase is directly proportional to the nickel adsorption efficiency and capacity.

Increasing the temperature from 15 to 25°C, the adsorption rate rises from 99.7% to 60.9% and adsorption capacity increases from 3.04 to 4.98 $mg.g^{-1}$. Increasing the temperature from 25 to 40°C, adsorption rate rises from99.7 to 94.7% and adsorption capacity decreases from 4.98 to 4.73% $mg.gr^{-1}$. The highest removal efficiency and adsorption capacity occurs at 99.7% and 4.98 $mg.gr^{-1}$ at 25 ° C (ambient temperature) respectively. As a result, a temperature of 25 °C was considered as the optimal temperature.

In this study, adsorption capacity and efficiency as the temperature increases, firstly increased and then decreased. The reason for this is that before the solution reaches equilibrium temperature up to 25°C, adsorption of nickel speeds up. These are due to the increase in the velocity of nickel ions and their collision to the surface of the absorber, the absorber expansion and therefore increase in the number of receiving locations on the adsorbent. Temperatures above 25°C, reduces the rate of penetration of nickel ions on the outside layer of the adsorbent layers, which reduces the ability to absorb nickel. In fact, high-temperature changes Nano-lignocellulose structure that reduces the capacity of the adsorbent to bind nickel (Alizadeh *et al.*, 2011).

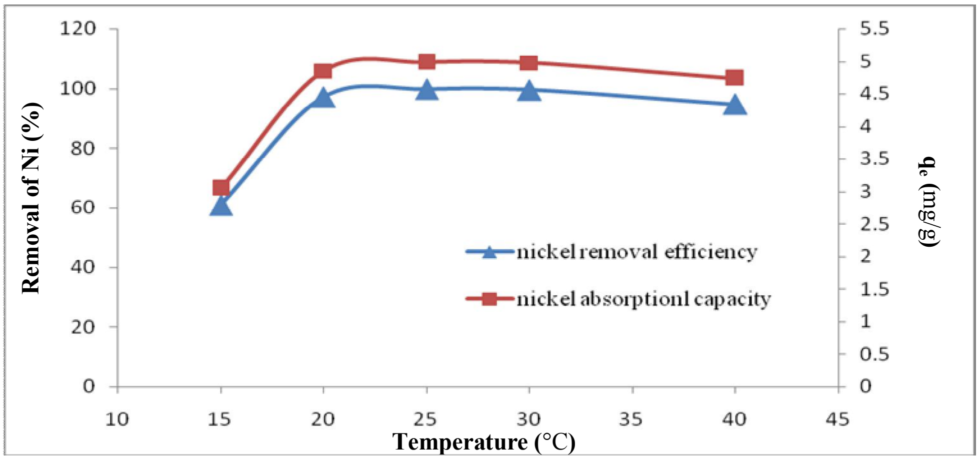

Figure 4. Effect of changing temperature on nickel adsorption by Nano- lignocellulose

3.5. Effect of time

Nickel adsorption efficiency against time is provided in figure 5.As can be seen, during the initial time, adsorption rate is very high. So that in the first 15 and 30 minutes respectively 95.4 and 97.8% of nickel is separated from the solution. From 60 to 120 minutes, adsorption efficiency changes from 99.5 to 99.8, while adsorption capacity changes from 4.97 to 4.99. Since from the time 60 to 120 minutes, only less than 1% was added to the adsorption, for economic reasons and to prevent a waste of time, 60 minutes was selected as the optimum time and further tests were done accordingly.

The results showed that the adsorption of nickel with Nano-lignocellulose occurs quickly. This is due to the presence of many active sites in the early stages. Alovalya (1997), also showed that the maximum removal rates is achieved in the first 30 minutes of reaction. Also Sharma and Singh (2009), reported that the highest adsorption of Zinc, occurs at the time of initial contact which then gradually increases. This might be explained by the disposal of metal ions.

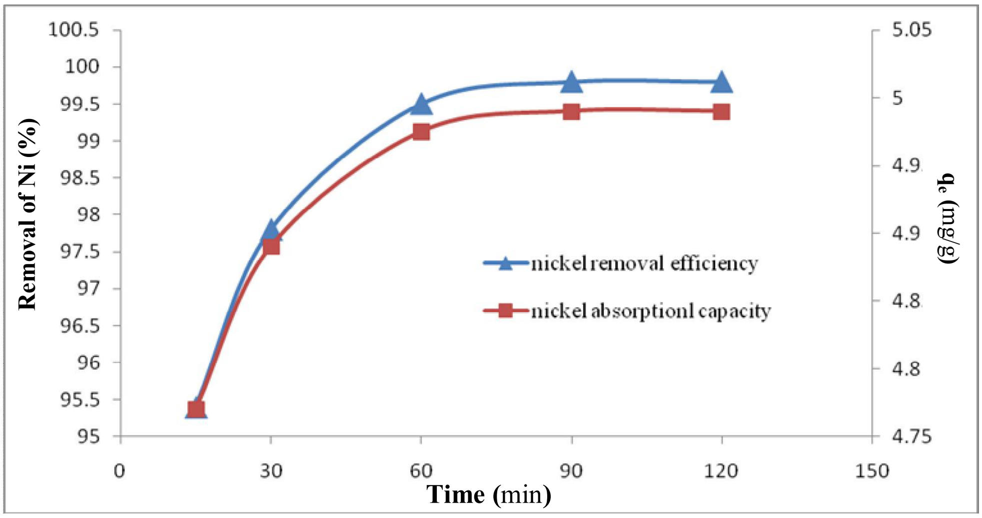

Figure 5. Effect of time on the absorbance of Nickel by Nano-lignocellulose

3.6. Results of adsorption isotherm's studies

According to the equations of adsorption isotherms, corresponding graphs were drawn, and with the help of the slope and intercept, the best straight line was fitted to the points to obtain constant values corresponding to each of the isotherm. Langmuir and Freundlich isotherm model is drawn in figures 6 and 7 to describe the adsorption process of nickel by Nano-lignocellulose.

In Table 2, the maximum values of adsorption capacity (q_m), isotherm constant (b), correlation coefficient (R^2), for the Langmuir and isotherm constant (K_f, a) as well as correlation coefficient (R^2) for Freundlich model are provided. As is evident in the results, the coefficient of determination (R^2) in the Langmuir model and 0.999 and the coefficient of determination (R^2) were obtained Freundlich and Freundlich models were respectively calculated as 0.999 and 0.874. According to the coefficients obtained, both models were able to explain and describe data. Yet, higher adsorption coefficient of determination in Langmuir model, showed a better fit to the Nickel absorbance data. Evidently, R_L values ranges between 0 and 1, denoting an optimal adsorption. Studies conducted by Anondy *et al.* (2010) and Malaruyzi *et al.* (2013), showed proper conformity between adsorption data with the Langmuir isotherm which is consistent with the results from this study. Langmuir model assumes monolayer adsorption in which the adsorbent surface has locations with the same energy where adsorbed molecules are only assigned to a specific location.

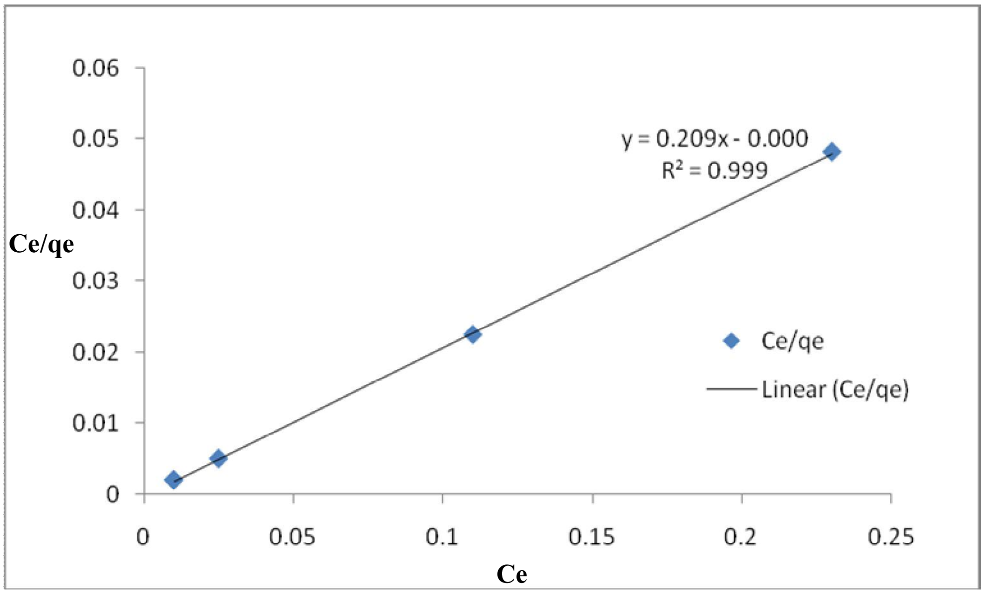

Figure 6. Langmuir isotherm model to describe the adsorption process of nickel by Nano-lignocellulose.

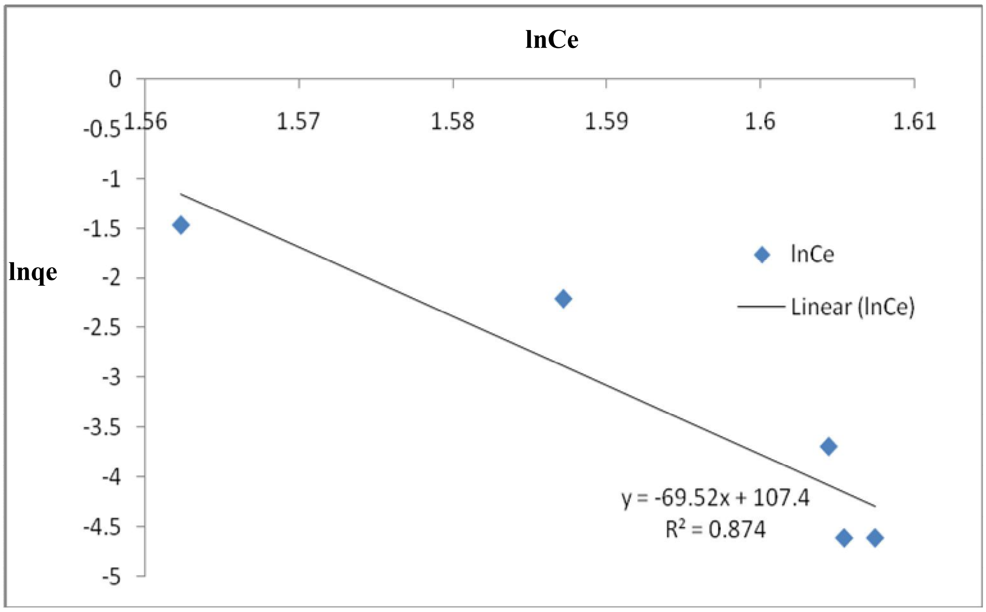

Figure 7. Freundlich isotherm model to describe the adsorption process of nickel by Nano-lignocellulose

Table 2. Parameters and correlation coefficients of Langmuir and Freundlich isotherm model of nickel absorbance by Nano-lignocellulose

Freundlich			Langmuir			
k_f	N	R^2	q_m(mg/g)	R_L	R^2	Nano-lignocellulose absorbant
4.99	0.132	0.8748	4.98	0.2252	0.999	

3.7 Assessment of the Kinetic models of Nickel adsorbance

Pseudo-first-and Pseudo-second-order modelsfor the adsorption of nickel by Nano-lignocelluloseare illustrated in figures 8 and 9. In Table 3, a comparison between the Pseudo-first- and Pseudo-second-order Kinetic models is provided. As given, pseudo-first and pseudo-second correlation coefficients have been respectively measured 0.930 and 1.As a result, according to the higher correlation coefficient of the pseudo-second-order model,it can be concluded that experimental data obtained from the adsorption of nickel possess high conformity with pseudo-second-order Kinetic model and the data could be better described by this model. Malarvyzy *et al.* (2013), in the course of their study on the removal of nickel ions from aqueous solutions via volatile coal ash found pseudo-second-order model to have the highest conformity.

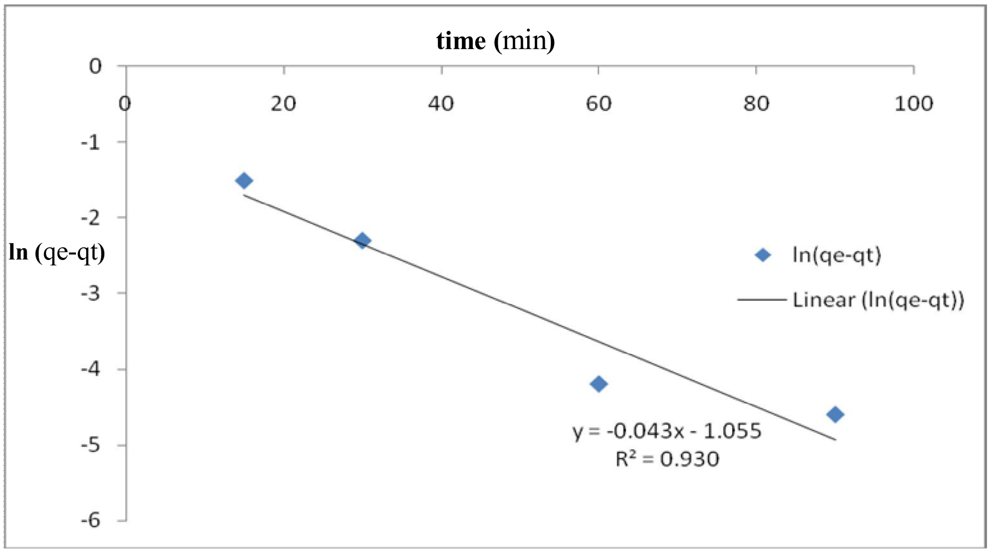

Figure 8. Pseudo-first-order Kineticmodelto describe adsorption process of nickel by Nano-lignocellulose

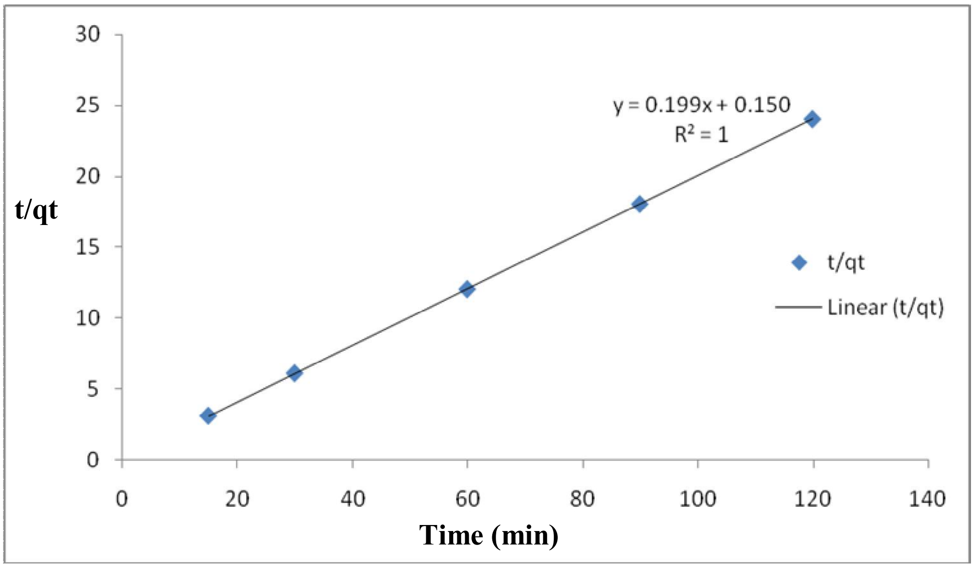

Figure 9. Pseudo-second-order Kinetic model to describe adsorption process of nickel by Nano-lignocellulose

Table 3. Assessment of the kinetic model of Nickel absorbance by Nano-lignocellulose using pseudo-first and pseudo-second-order Kinetic model

Pseudo-second-order			Pseudo-first –order		
q_e(mg/g)	K_2	R^2	q_e(mg/g)	K_1	R^2
5.00	0.3461	1	4.99	-0.0235	0.93

3.8. Analysis of adsorption thermodynamics

Thermodynamics determines the energy level of the reactants and products as ΔH, ΔS and ΔG examine the possibility of reaction. Calculated thermodynamic parameters for Nickel adsorption by Nano-lignocellulose is provided in figure 4. Thermodynamic studies showed that the adsorption of nickel metal via Nano-lignocellulose occurs with increased irregularities spontaneously.

Likewise, negative values of ΔH (enthalpy) showed that the overall process isan endothermic reaction, in the sense that by decreasing the ambient temperature, the removal rate increases. High changes in enthalpy level reflect the sensitivity of the adsorption process to the temperature. Positive values of ΔS (entropy) also showed that the irregularities in the solid-liquid interface during the process of adsorption have increased. As can be seen, negative values of ΔG show the spontaneous nature of adsorption reaction. Kardam *et al.* (2014), used the Nano-lignocellulose as adsorbents for the removal of cadmium, nickel and lead from aqueous solutions. The results of thermodynamics revealed that by positive values of enthalpy and increasing the efficiency of adsorptionin response increasing temperature, the reaction were endothermic which is consistent with the results of this study.

Table 4. The results of the calculation of the thermodynamic parameters for Nickel adsorption by Nano-lignocellulose

Temperature	ΔG	ΔH	ΔS
15	-1061	-34526.81	84.26
20	-8552.88		
25	-14385.1		
30	-13334.6		
40	-7502.4		

4. Conclusion

In the present study, the aim was to investigate the process used to remove nickel by using Nano-lignocellulose adsorbent. The effect of various parameters on the process was studied. The results showed that the adsorption of nickel is dependent on pH, so that pH 6 was selected as the optimal level. Given that at the concentration of 5 mg/L, 99.8% of the nickel was removed, this concentration was chosen as the finest concentration. Also, taking into account the cost of the absorber, the dosage of 0.1 gram was selected as optimal for nickel adsorbent. Optimum contact time was 30 minutes, which reflects the short duration of the analysis. Evaluation of the effect of temperature and thermodynamic studies showed that the adsorbent was removed by spontaneous and endothermic process. Studies on the adsorption isotherms showed the best fit for the experimental data corresponds to the Langmuir isotherm model. Kinetic studies revealed that the process of elimination conforms to pseudo-second-order Kinetic model. According to the results of this research it can be conclude that, Nano-lignocellulose has very high ability to remove heavy metals like nickel from aqueous solutions. Rate and capacity of Nickel adsorption by the adsorbent is high due to the small size, high surface area, unique network arrangement and thus remarkable reactivity of Nanoadsorbent. The results of this study can be used to guide removing nickel from the environment, particularly aquatic environment, to protect human health and aquatic plants, animals, and especially aquatic mammals that are very dependent on the aquatic environment. In a nutshell, Nano-lignocellulose has succeeded in removing nickel and could be used at larger scales to remove heavy metals from industrial wastewater.

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