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Thermodynamic Parameters of Adsorption of Nickel and Lead onto Cinnamomum Camphora Seeds Powder

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Abstract: ***Adsorption is one of the best technologies for the removal of nickel and lead water decontamination because it is an efficient, cheap and eco friendly treatment method. It is a good technique strong adequate to recognize water reuse requirement and high overflow standards in the industries. In the present work Cinnamomum Camphora seeds powder was studies as adsorbent for the removal of nickel and lead ions from aqueous solutions. The batch adsorption studies were carried out to examine the adsorption of nickel and lead on Cinnamomum Camphora seeds powder for the effect of initial concentration, contact time, adsorbent dosage, pH, temperature and also studied thermodynamic parameters. The maximum adsorption removal of Cinnamomum Camphora seeds powder was found to be 90 % and 91% for lead and nickel at the initial concentration of 50 mg/L. For the effect of temperature, the percentage removal of lead and nickel was noticed as 92% and 96% at the temperature of 62°C. The removal of nickel ions decreased with increasing in pH, thus pH of 10, which gives maximum percentage removal of nickel, is 90% and for lead is 89% at the optimum pH of 6. The adsorbent dosage of Cinnamomum camphora seeds powder increases with increasing the percentage removal of lead and nickel and it is found to be 88 % and 85 %. The correlation coefficient of Langmuir model for lead and nickel were found to be 0.986 and 0.9936 and the correlation coefficient of Freundlich model for lead and nickel were found to be 0.9905 and 0.9647 respectively. The Langmuir adsorption model which exhibited outstanding indication of adsorption data than the Freundlich adsorption model for Cinnamomum Camphora seeds powder in the nickel. The plot of log qe/ce versus 1/T was obtained with R-squared value 0.988 and 0.985 for nickel and lead. The slope & the intercept which gives the value ΔS° of ΔH° and ΔS°, respectively. The decrease in ΔG° with the increase in temperature which shows good adsorption at high temperatures. .The positive value for change in enthalpy is due to endothermic nature of adsorption of cadmium***

Keywords: Thermodynamic Parameters, Cinnamomum Camphora Seeds Powder, Lead and Nickel, Langmuir and Freundlich Isotherm Models, Wastewater

1. Introduction

The most important thing required in all the existing organisms is the contact to clean water. Due to increase of population, not only the stress on the reserves raise but contaminations of these natural resources is increasing day by day. Nickel is an element which occurs in the environment only at extremely low down levels [1]. The nickel metal ion content in soil the can be as small as 0.2 ppm or as elevated as 450 ppm in few clay and loamy soils. This nickel metal ion which causes serious health troubles such as damage to lungs, kidneys, gastrointestinal distress, e.g., nausea, vomiting, diarrhea, pulmonary fibrosis and skin dermatitis [1]. Lead is a growing toxicant which can affects the multiple body systems and is mostly injurious to young children.

This metal ion also used in several other products such as paints, pigments, stained glass, lead crystal glassware, ceramic glazes, jewellery, toys and in few cosmetics and traditional medicines [2]. At elevated levels of introduction, lead will attacks the brain and central nervous system which can causes coma, convulsions, death, Lung and liver damage; loss of appetite. The permissible level of lead is 0.15 mg/L. The heavy metal ions separated from inorganic waste matter can be achieved by using conventional treatment methods [3]. Separations of heavy metal ions from industrial wastewaters can be consummate through different treatment methods which includes like unit operations as chemical precipitation, complexation, coagulation, activated carbon adsorption, ion exchange, solvent extraction, foam flotation, electro-deposition and membrane operation methods [4].

Among these methods, adsorption has been accepted as a successful and cost-effective mode to treat the wastewaters which containing poisonous metals. It gives flexibility in the design and operation, as the process of adsorption can be operated in batch operations (5). The adsorption is more cost-effectively favourable; when the adsorbent is reused and the adsorbate can be recovered, as it makes the process of adsorption is more suitable [6]. Cinnamomum Camphora seeds powder used as a low cost and eco-friendly adsorbent for the removal of lead and nickel from industrial wastewater [7].

2. Materials

Lead Nitrate, Nickel Nitrate Hexahydrate, Dimethyl glyoxime, Sodium Hydroxide, Sulphuric acid, distilled water and Cinnamomum Camphora seeds powder are purchased online from Nursery live in Kerala. All chemicals were used of analytical reagent grade only for the conduction of experiments

3. Methods

3.1 Adsorbent Preparation

The Cinnamomum Camphora seeds are purchased online from Nursery live in Kerala. Then the seeds are washed with distilled water to remove the dust and then soluble impurities. After this, seeds are permitted to dry in an air oven at 80 °C for a period of 24 hrs. These seeds were crushed into a fine powder in a mechanical grinder to get the seed powder. It is sieved using a 200 Tyler mesh and the mesh opening is 72 µm. The activation of adsorbent is carried out by treating it with 0.1N Sulphuric acid and dried at room temperature [8]. Again it is washed with distilled water to remove acid traces.

Figure 1: Cinnamomum Camphora Seeds Powder

3.2 Preparation of Adsorbate Solution

Lead Stock Solution

All the required solutions are prepared with double distilled water. 1.5980 g of lead nitrate ($Pb(NO_3)_2$) is dissolved in distilled water and made up to exactly1000ml. Samples of different concentrations of lead are prepared from the stock solution by appropriate dilutions.

Nickel Stock Solution

The aqueous solutions of Nickel were prepared by 10g of Nickel Nitrate Hexahydrate dissolved in 1 liter of double distilled water to prepare 1000 mg/L of Nickel stock solution. 1000 mg/L Nickel solution is prepared by adding 50 ml of 1000 mg/L Nickel stock solution with distilled water in 500 ml volumetric flask to the required concentrations. The different initial concentration of 50, 100, 200 300, 400 and 500 mg/L were prepared by adding 100 mg/L nickel stock solution. Solutions of 0.1 N Sodium Hydroxide and 0.1 N Sulphuric acid were used for pH adjustment.

Characterization of the Adsorbent

To study the various physical and chemical factors which affecting the adsorption, it is required to characterize the adsorbent [9,10] Hence, physical and chemical categorization was done by using scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR).

3.3 FTIR Spectra for Cinnamomum Camphora Seeds Powder

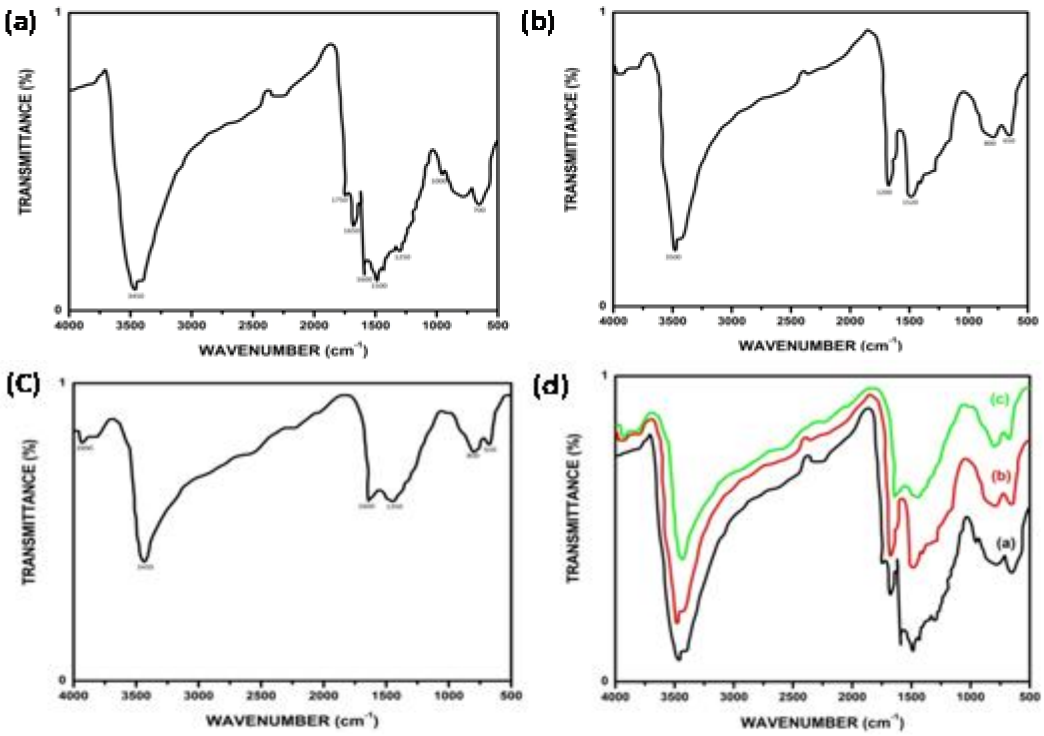

Figure 2: FTIR spectra for samples of Cinnamomum Camphora Seeds Powder (a) Untreated (b) Treated with NaoH (c) Treated with H_2So_4.

Functional groups which are present in organization of sorbent were studied by Fourier transform infrared spectrophotometer (FTIR). The above figure 2 shows the FTIR spectra of Cinnamomum Camphora Seeds Powder and the peak assignments in FTIR spectra are verified at the strong peak 3450 (N-H/C-H/O-H) in the figure a. The different functional groups like

Phenols, Ketones, Aromatic Compounds, and C-CI are observed at different peaks in the figure b. The appearance of OH Stretching, N-H/C-H/O-H, Diketones, C-O, Aromatic Compounds, C-I groups shows the good indication on adsorption of Cinnamomum Camphora Seeds Powder. These functional groups are responsible for adsorption of sorbate on the surface of sorbent, and their detection helps in determining the nature of binding interactions between sorbate and sorbent surface.

SEM images of Cinnamomum Camphor Seeds Powder
Analysis of Surface was investigated by using scanning electron microscope JEOL model 2300. This SEM technique which provides the information regarding the surface area available for morphology and adsorption of adsorbent [11, 12]. Analysis of each sorbent was carried out in optimized conditions under argon atmosphere. One treated adsorbent (acid- and base-treated Cinnamomum Camphora Seeds Powder) were examined through SEM to investigate the surface morphology. Large pore size available on the native surfaces and base-treated adsorbent was liable for increased adsorption on the surface of these materials [9]. From the below figure 2, it is observed that the hollow cavities are become visible in the structure of untreated adsorbent, which were responsible for binding of adsorbate onto the adsorbent surface. But the acid treatment decreases the hollow cavities by deforming the adsorbent surface. Hence adsorption decreases after treated with acid because the surface becomes smooth and thin layer of adsorption is formed on the adsorbent surface.

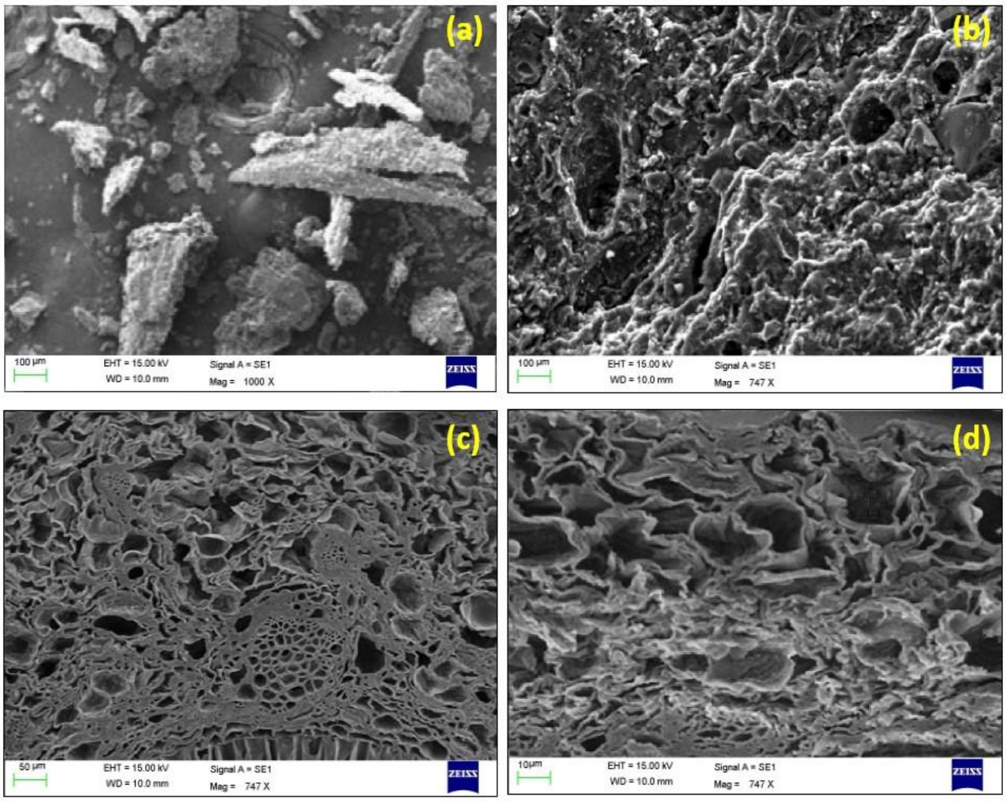

Figure 3: SEM images of Cinnamomum Camphor Seeds Powder: (a) Untreated (**b)** Treated with NaoH (c) Treated with H_2So_4 (50 μm) and (d) Treated with H_2So_4 (10 μm)

Batch Adsorption Experiment
The present investigation was carried out in batch studies on adsorption of lead and nickel from aqueous solutions onto Cinnamomum Camphor Seeds Powder. Batch adsorption studies were carried out to find out the adsorption isotherms of lead and nickel metal ions onto the Cinnamomum Camphor Seeds Powder as adsorbent in 250 ml glass flask. The glass flasks were shaken at a stable rate, which allows the sufficient time for the constancy of adsorption. The parameters of effect of contact time and pH was studied with a lead and nickel concentrations of 100 mg/l and an adsorbent dosage of 2 g/100ml, the aqueous solution pH was adjusted in the range of 2 to10 by using dilute Sulphuric acid and Sodium hydroxide solutions. The adsorption experiments were carried out by varying the adsorbent amount from 2-10 g/100 ml with lead and nickel concentration of 100 mg/l. Finally, lead and nickel concentrations were analyzed by using spectrophotometrically.

4. Results and Discussions

Effect of contact time
The parameter of effect of contact time on fraction removal of lead and nickel was considered at initial concentration 100 mg/L and adsorbent dose 2g/100ml. The contact time

this varies from 0 to 80 minutes. From the graph, it is observed that the removal of lead and nickel ions increases with increasing in effect of contact time, attaining equilibrium from 120 to140 minutes. The percentage removal of lead (26% to 80%) and nickel (26% to 80%) metal increases for Cinnamomum Camphor Seeds Powder at 0 to140 minutes. Ultimately we concluded from the below graph, that the percentage removal of lead and nickel is 80%. The time required for contact is 120 to 140 minutes in both the metal ions.

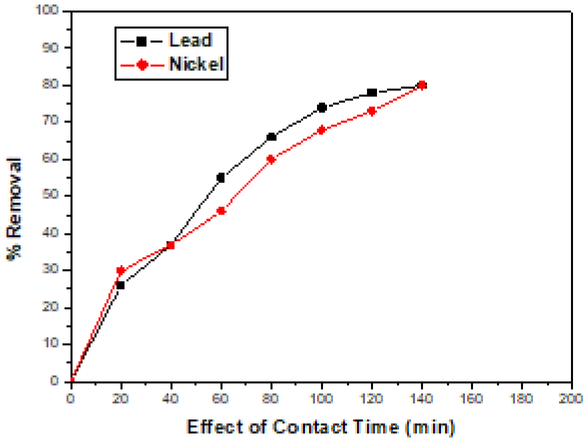

Figure 4: Effect of Contact time on lead and nickel at initial concentration 100 mg/L, adsorbent dose 2g/100ml, temperature 32^0 C by Cinnamomum Camphora Seeds Powder

Effect of initial concentration

The parameter like initial concentration on percentage removal of lead and nickel were studied at the adsorbent dosage of 2 g/100ml. The initial concentrations of lead and nickel were varies from 50 mg/l to 500 mg/l. From the graph, it is observed that the percentage removal of lead decreased (46% to 90%). For nickel, the percentage removal decreased and then slowly declines from (42% to 91%) with increasing initial concentrations 50 mg/l to 500 mg/L for Cinnamomum Camphora Seeds Powder. At small concentration of 50 mg/L, the nickel percentage removal was maximum i.e 91% than the lead for Cinnamomum Camphora Seeds Powder. The decrease in percentage removal of lead and nickel for the adsorbent of Cinnamomum Camphora Seeds Powder are found to be 90% to 91% respectively.

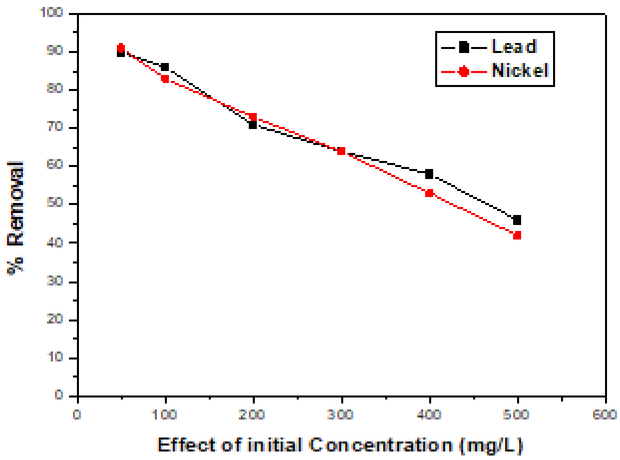

Figure 5: Effect of initial concentration on percentage removal of lead and nickel at adsorbent dose 2 g/100 ml, temperature 32^0 C by Cinnamomum Camphora Seeds Powder

Effect of adsorbent dosage

The effect of adsorbent dosage on removal of lead and nickel was considered by changing the adsorbent dose from 2 to10 g/100ml at fixed adsorption conditions altering Cinnamomum Camphora Seeds Powder. The adsorbent dose on percentage removal of lead and nickel was studied at the initial concentration of 100mg/L and temperature 32^0C by using Cinnamomum Camphora Seeds Powder as adsorbent. The utmost separation of lead and nickel were found to be 88% and 85% achieved at the higher adsorbent dosage of 10 g/100ml. The maximum removal of lead was observed more than the nickel on adsorption process with mounting in adsorbent dosage for Cinnamomum Camphora Seeds Powder. The percentage removal of metal ions increased with increased in the dose of adsorbent and maximum adsorption was occurred at the dosage of 10 g/100ml solution, beyond which no further increase was noticed. It is due to the raise in the surface area and adsorption sites accessible for adsorption. Particularly at higher adsorbent dose the account of active sites and surface area results from accumulation of the adsorbents.

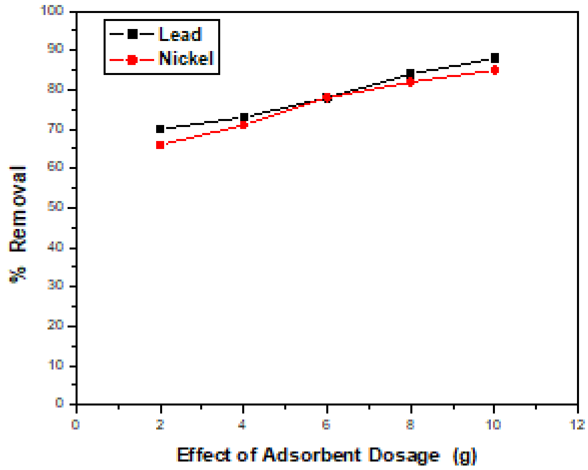

Figure 6: Effect of adsorbent dose on percentage removal of lead and nickel at initial concentration of 100 mg/L, temperature 32^0 C by Cinnamomum Camphora Seeds Powder

Effect of pH

The batch adsorption studies were carried out at various pH ranges from 2 to 10 and maintenance all other adsorption conditions are steady. The solution pH is one of the most important significant parameters in the adsorption of metal ions such as lead and nickel from aqueous solutions. Approximately in lead and nickel metal ions at Cinnamomum Camphora Seeds Powder, a considerable enhancement and diminish in adsorption was noticed as pH increased with optimum pH of 6. The processing parameter such as pH enhance from 2 to 10 with reducing in percentage of lead and nickel ions. From the below, it is concluded that the removal of nickel ions decreased with increasing in pH, thus pH of 10, which gives maximum removal of nickel is 90%.The percentage removal of lead (89%) was observed at optimum pH 6 for Cinnamomum Camphora seeds powder.

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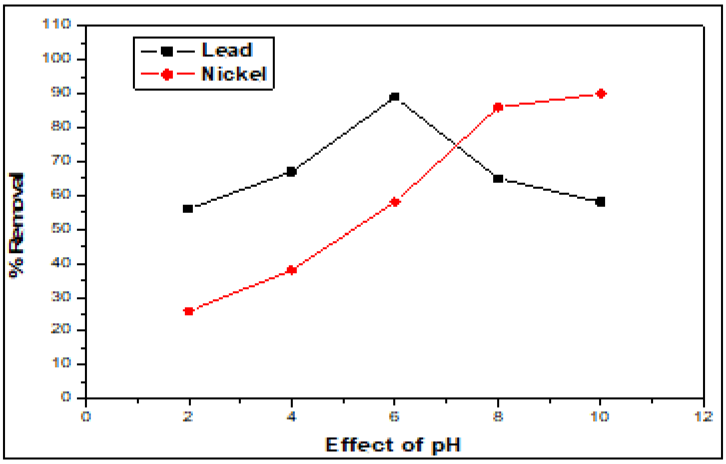

Figure 7: Effect of pH on percentage removal of lead and nickel at initial concentration 100 mg/L, adsorbent dose 2 g/100 ml and temperature 32^0 C by Cinnamomum Camphora Seeds Powder

Effect of temperature

The batch adsorption experiments were performed at various temperatures 32°C, 42°C, 52°C and 62°C and maintaining all other adsorption conditions are stable. The effect of temperature on percentage removal of lead and nickel was carried out at initial concentration of 100 mg/l and adsorbent dosage of 2 g/100ml.The utmost percentage removal of nickel (96%) was noticed at the temperature of 62°C for Cinnamomum Camphora Seeds Powder. The maximum percentage removal of lead and nickel were found to be 92% and 96% at the temperature of 62°C. Further enhance in temperature (beyond 52°C) though activate the adsorption sites but at the same time reduces the physical forces responsible for the adsorption as well as increases the kinetic energy of the adsorbate molecules resulting there by in a net decrease in the adsorption.

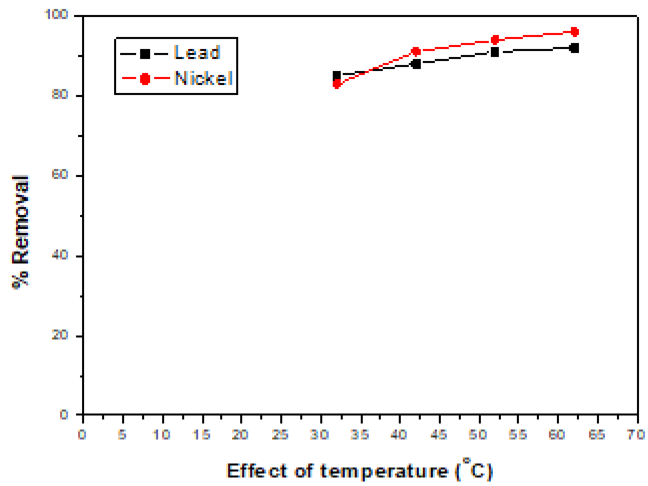

Figure 8: Effect of temperature on percentage removal of lead and nickel at initial concentration 100 mg/L and adsorbent dose 2 g/100ml by Cinnamomum Camphora Seeds Powder

Equilibrium Isotherms

The system of adsorption which can be designed by the isotherms of adsorption commonly is known as equilibrium isotherms which signifies the solute amount adsorbed per unit weight of sorbent (13,14). In order to remove the effluents from the system, particular design is optimized to generate the suitable correlation for investigational data which is known as adsorption isotherm.

Researches projected several isotherms in this regard which are based on the system of adsorption such as Langmuir and Freundlich isotherms. These isotherms were applied to fit the experimental data of adsorption. The Langmuir and Freundlich adsorption isotherms are the plot between Ce/qe versus Ce and lnqe verses lnCe respectively. The following Langmuir and Freunlich adsorption equations are written as

$$Ce/qe = (1/bQ)+(1/QCe) \text{ ------------------ } (1)$$

$$\ln qe = \ln K_f + 1/n \ln Ce \text{ ---------------------- } (2)$$

Table 1: Isotherms constant data for lead and nickel with CCSP

Isotherms	Constants	Lead	Nickel
Langmuir Isotherms	Q	4.72	10.04
	b	0.008	0.017
	R_L	0.51	0.38
	R^2	0.986	0.9936
Freundlich Isotherms	n	1.425	2.282
	K_f	0.0088	0.746
	R^2	0.9905	0.9647

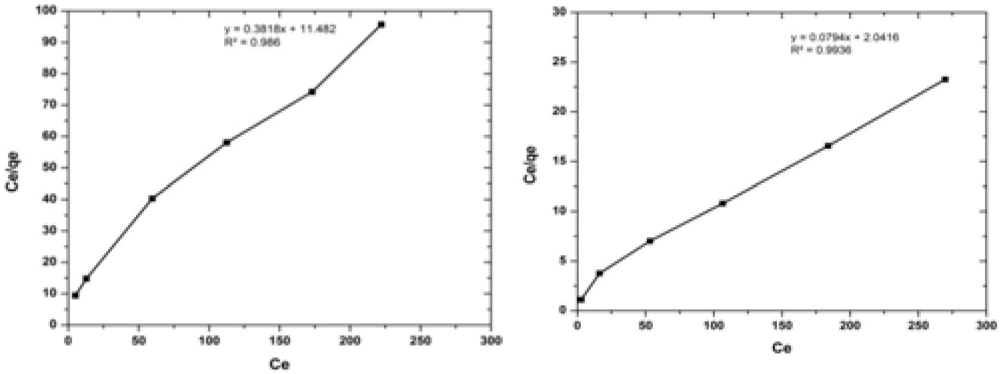

Figure 9: Langmuir isotherms for Lead and Nickel by Cinnamomum camphor powder at initial concentration 100 mg/L, temperature 32^0 C and adsorbent dose 2g/100 ml

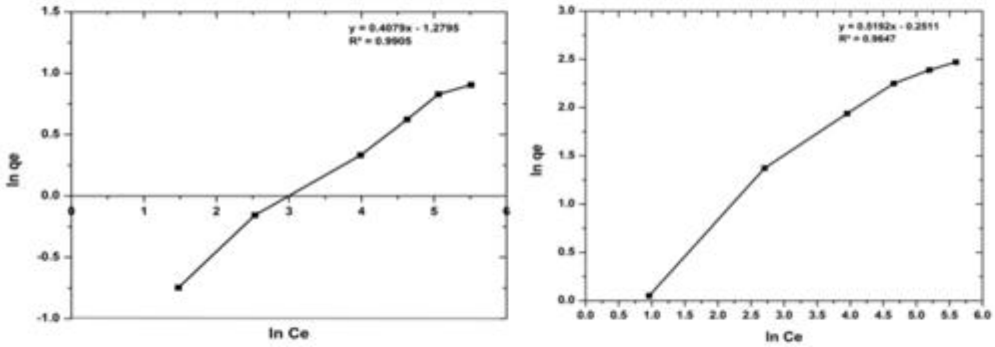

Figure 10: Freundlich isotherms for Lead and Nickel by Cinnamomum camphor powder at initial concentration 100 mg/L, temperature 32^0 C and adsorbent dose 2g/100ml.

Freundlich Isotherms

The Parameters determined for Freundlich isotherm by utilizing its linear form are given in the Table 1. Freundlich isotherm was obtained by plotting ln qe versus ln Ce. K and *n* are constants which are obtained from intercept and slope, respectively in the present study. The values of K_f (0.0088 & 0.746) and n (1.425 & 2.282) for lead and nickel are obtained from the above equation 2 and it is linear approaches of Freundlich adsorption isotherm. In the same way, the adsorption intensity showed by n which indicates the fitness of model for adsorption purposes if *n value* is more than 1. The values of correlation coefficient R^2 (0.9905&0.9647) for lead and nickel obtained from the plot.

Langmuir Isotherm

Langmuir adsorption model is based on monolayer adsorption of metal ions on the surface of Cinnamomum camphora seeds powder. In order to determine the Langmuir model, initial concentrations were varied from 50 to 500 mg/L with 2g adsorbent amount. Langmuir adsorption model was obtained by plotting Ce/qe versus Ce as shown in Figure 9. R^2 value obtained for plot was found satisfactory showing fitness of model on the adsorption research. *Q* represents metal ion uptake per unit mass of adsorbent (mg/g) and b is Langmuir constant. A dimensionless constant R_L is determined by using Langmuir constant, and initial concentration represents model fitness for a specific system. If the value of R_L which falls between 0 and 1, the system is measured appropriate for adsorption purpose and Table 1 shows the results which are in this range. The correlation coefficient R^2 of Langmuir adsorption model fits the good data for nickel than the lead metal ions.

Thermodynamic Parameters

The parameter like effect of temperature on the process of adsorption was performed by using different temperatures ranges [15, 16]. For thermodynamic investigations, the experiments of adsorption was carried out at various temperature conditions like 32°C, 42°C and 52°C maintaining the initial concentration 100 mg/L and adsorbent dose 8 g/100ml are stable. As it was noticed that with an enhance in temperature then the percentage removal increased. With enhancing temperature the number of active sites available for process of adsorption should have enhanced results in increasing of percentage removal by the adsorption.

The thermodynamic systems of the adsorption were performed as

$$\log [qe/ce] = \Delta S°/2.303R+\{-\Delta H°/2.303RT\} \text{ ------- (3)}$$

$$\Delta G° = \Delta H° - T\, \Delta S° \text{ ------------------------------------ (4)}$$

Where qe = maximum adsorbed per unit mass of adsorbent (mg/g); Ce is the equilibrium concentration, mg/L; R is the gas constant 8.314 J mol/K and Temperature (°K)

Table 2: Thermodynamic Properties for Nickel and Lead

Adsorbent	Metal name	ΔH° KJ/K.mol	ΔS° KJ/Kmol.K	ΔG° KJ/K.mol		
				305	315	325
Cinnamomum Camphora Seeds powder	Nickel	51.752	0.1572	3.806	2.234	0.662
	Lead	25.178	0.0736	2.73	1.994	1.258

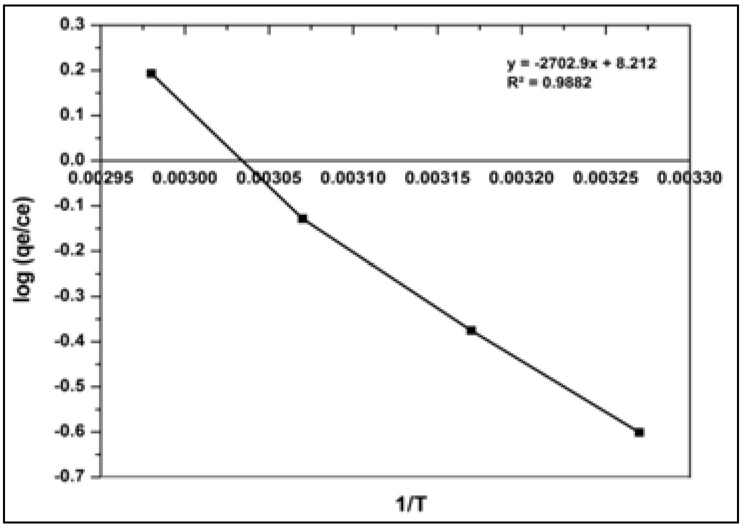

Figure 11: Thermodynamic parameter estimation system: Nickel-Cinnamomum Camphora seed powder, initial concentration = 100 mg/L, adsorbent dose = 8 mg/100 ml, pH= 4

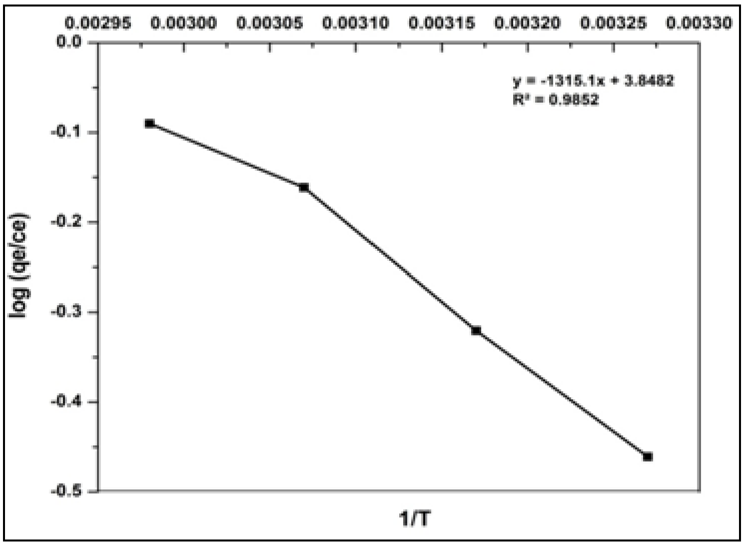

Figure 12: Thermodynamic parameter estimation system: Lead- Cinnamomum Camphora seed powder, initial concentration = 100 mg/L, adsorbent dose = 8 mg/100 ml, pH= 8

From the above figures 11&12, It is observed that the adsorption of nickel and lead was found to enhance with enhancing in temperature. At higher temperatures, further adsorption sites are formed which improve the adsorption phenomenon. The plot of log qe/ce versus $1/T$ was obtained with R-squared value 0.988 and 0.985 for nickel and lead. The Slope and the intercept which gives the value of ΔH° and ΔS°, respectively, as shown in the equation 3. The decrease in $\Delta G°$ with the increase in temperature which shows good sorption at high temperatures. The property of enthalpy was also shows positive since uncertainty in system increases due to solid-liquid contact during adsorption.

5. Conclusions

The percentage removal of lead (26% to 80%) and nickel (26% to 80%) metal increases for Cinnamomum Camphor Seeds Powder at 0 to140 minutes of contact time. At small concentration of 50 mg/L, the nickel percentage removal was maximum i.e 91% than the lead for Cinnamomum Camphora Seeds Powder.

The utmost separation of lead and nickel were found to be 88% and 85% achieved at the higher adsorbent dosage of 10 g/100ml.

The maximum removal of lead was observed more than the nickel on adsorption process with mounting in adsorbent dosage for Cinnamomum Camphora Seeds Powder. For the effect of pH, It is concluded that the removal of nickel ions decreased with increasing in pH, thus pH of 10, which gives maximum removal of nickel is 90%.

The percentage removal of lead (89%) was observed at optimum pH 6 for Cinnamomum Camphora seeds powder. The maximum percentage removal of lead and nickel were found to be 92% and 96% at the temperature of 62°C.

The correlation coefficient R^2 of Langmuir adsorption model fits the good data for nickel than the lead metal ions.

The decrease in $\Delta G°$ with the increase in temperature which shows good sorption at high temperatures. The property of enthalpy was also shows positive since uncertainty in system increases due to solid-liquid contact during adsorption.

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