



Research Paper

Comparative Analysis of the Thermodynamics and Adsorption Isotherms for the Adsorption of Some Metal Ions from aqueous solution using Sawdust Material

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Available online at: www.ijrce.org

(Received 18th June 2011, Accepted 7th July 2011)

Abstract: The experimental results of the thermodynamic and adsorption isotherms of best fit for the adsorption of Cr(VI), Ni(II), Fe(II) and Cd(II) from aqueous solution onto Sawdust were studied. Adsorption experiments were carried out using batch process with a fixed (8g) weight of adsorbent and variable adsorbate concentrations. Changes in concentration of the different adsorbates during the adsorption process were monitored by the use of Shimadzu AA650 Double Beam Atomic Absorption/Flame spectrophotometer. Thermodynamic parameters such as ΔH and ΔS were calculated from Van't Hoff plots which subsequently lead to the computation of ΔG . At high adsorbate concentrations, ΔH varied from 292, 754, 180 and 1,868 Jmol⁻¹ respectively for Cr, Ni, Fe and Cd. While ΔS varied in the form 2.89, 10.04, 0.83 and 35.66 Jmol⁻¹K⁻¹ respectively for the same set of adsorbates. Whereas at the highest temperature studied ΔG changes according to the order -666.45, -2,590.26, -96.69 and -10,006.20 Jmol⁻¹ respectively. This shows that the adsorption process is endothermic and spontaneous, with spontaneity increasing as the temperature increases. Equilibrium data were tested using Freundlich, Langmuir and Dubinin-Radushkevich adsorption isotherms. The adsorptions of Cr, Ni and Fe onto sawdust were found to be a favourable physical adsorption process which fitted well to the Freundlich adsorption isotherm. However, the adsorption of Cd having R_L value of 1.0000 is a linear process as suggested by Langmuir isotherm.

Key words: Adsorption; Adsorbate; Adsorbent; Thermodynamics; Isotherm; Sawdust

Introduction

Although water is abundant on earth, and since almost 97% of the water contains salts, it is not suitable for drinking or for the various industrial purposes. Of the remaining 3%, two thirds is in the form of ice and snow leaving only about 1% of the total water as fresh water. Of this ground water accounts for about 98% and the surface water is only about 2%. Thus, of the total amount of water present on earth, only about 0.02% is available in the lakes and streams^[1]. Therefore such a limited resource is very precious and needs conservation. Industrial effluents contains significant levels of toxic substances, and since the final destinations of effluents are the streams, rivers and the lakes, these toxic substances are finally transported therein and the health risk become more wide. Hence keeping the effluents free of these toxic substances is by extension keeping the environment healthier, and this is the target of this research work.

Pollution has been defined by Wong *et al.*,^[2] as a harmful change in the natural environment caused by human activities which may be as a result of the release of substances, that are toxic to either animals or plants, or energy (heat,

light, radiation, or sound), which interferes with the development of animals or plants. A pollutant on the other hand is any substance produced by human activities, which at an unacceptable level, causes damage to the environment or is harmful to living systems. In general, pollution is usually harmful to one or more species of animal or plant. As such in order to maintain a healthy living environment, a careful study on the sources and nature of pollution and the ways of pollution control should be considered.

Most of the reported work on the use of agricultural by-products for removal of heavy metals has to do with the capability of such materials in adsorbing heavy metals^[3] The principle behind exploring agricultural by-products in this work is the fact that most agricultural by-products are composed of cellulose in the plant cell walls.

The IR spectrum of sawdust according to Abdel-Ghani *et al.*,^[4] showed small absorption bands at 3726.2, 3618.2 and 3525.6 cm⁻¹ assuming the presence of H-bridges and crystal water. A medium band at 2908.5 cm⁻¹ was also

attributed to H- bridges, the weak band at 2125.4 cm^{-1} may be attributed to (C=CH) while the band at 1639.4 cm^{-1} was due to (C=C). The strong band observed at 1033.8 cm^{-1} indicated the stretching of the many C-OH and C-O-C bonds.

The work of Igwe and Abia^[5] on the effect of temperature and particle size on bioremediation of As(III) from aqueous solution using modified and unmodified coconut fiber reported ΔG values which are less than -15 kJmol^{-1} indicating a spontaneous and physical adsorption process, with the modified fiber showing higher remediation of the adsorbate.

The work of Rajmond^[6], utilised a mixed bed ion exchange column for the simultaneous determination of Cr(III) and Cr(VI) using UV detector at a wavelength of 365nm from environmental samples such as rainwater and galvanic sediments. The performance of his method was established for the linearity, limit of detection, limit of quantification and the influence of sample pH.

Activated carbon from cassava peel was found to be effective for the removal of nickel from aqueous solution^[7] and the equilibrium sorption isotherm were found to fit well to three isotherm models, with the Temkin model having the highest correlation coefficient. Their result also indicated that Ni(II) uptake by the adsorbent followed pseudo 2nd-order rate expression.

The use of new commercially available superflow chelating resin with Nitriloacetic Acid (NTA) - type functional groups for concentration of trace metals (such as iron and copper) from seawater has been described by Lohan *et al.*,^[8]. The technique made use of inductively coupled plasma-sector field mass spectrometry (ICP – SFMS) for the determination of low concentrations of dissolved iron and copper in seawater. Trace metal recoveries were found to be pH dependent such that at $\text{pH} \leq 2$, only iron (III) and copper are quantitatively recovered.

The effect of agitation rate on the % adsorption of cadmium was studied by Ibrahim *et al.*,^[9] for cadmium in aqueous solution by using sugarcane bagasse. Results showed that highest Cd^{2+} adsorption was achieved at agitation rate of 150rpm and the adsorption process was relatively fast and equilibrium was achieved after about 90 mins of contact.

Methodology

Material and Methods

All glasswares and plastic containers were washed with detergents, rinsed with distilled water and then soaked in a 10% HNO_3 solution for 24hrs. They were then washed with deionised water and dried in an oven for 24hrs at 80°C ^[10].

Distilled-deionised water, and AnalaR grade reagents were employed for the preparation of all stock solutions. 1000mg/L of Cr(VI), Ni(II), Fe(II) and Cd(II) were respectively prepared by dissolving 2.8290g of $\text{K}_2\text{Cr}_2\text{O}_7$, 4.0503g of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, 4.9780g of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ and 2.1930g of $\text{CdSO}_4 \cdot 8/3\text{H}_2\text{O}$ in small volumes of distilled deionised water in separate beakers and the solutions were transferred to a 1.0litre volumetric flasks followed by the addition of 100cm^3 of 6M HNO_3 and they were made to mark with more water^[11]. Lower working concentrations were prepared daily from the stock solution by appropriate dilution.

Hardwood sawdust material of Mahogany (*Khaya senegalensis*) tree collected from a local sawmill was air dried

in sunlight until almost all the moisture evaporated, then it was washed several times with distilled water in order to remove the water soluble tannins, after which it was dried in air and then in an oven at 80°C . The material was allowed to cool and it was then ground to the working particle size ($850\mu\text{m}$) and kept in a plastic container for subsequent use.

Static adsorption experiments (studied at four different temperatures viz; 303, 313, 323 and 333K) in which the same solution remains in contact with a given quantity of adsorbent till equilibrium between the adsorbate concentration in solution, and the adsorbate adsorbed per unit weight of adsorbent, is reached was employed in this work. This type of equilibrium established is static in character, as it does not change further with time. Procedures described by Bhattacharya, *et al.*,^[12] and El-Nemr, *et al.*,^[13] for testing the adsorption properties of the adsorbent were followed. The procedure involved shaking a fixed amount of the adsorbent (8 g) with 100 cm^3 of the aqueous solutions of the adsorbates (in a screw capped Erlenmeyer flasks) with initial loading concentrations ranging from 20 – 60mg/L on an Innova 4000 shaker from New Brunswick Scientific at a speed of 290 rpm for an equilibrium period of one hour^[14]. Immediately after the shaking process, the samples were separately filtered using Whatman number 1 filter paper, and the filtrates collected in polyethylene bottles were taken for AAS measurements for the residual adsorbate level using Shimadzu AA650 Double Beam Atomic Absorption/Flame spectrophotometer.

Results and Discussion

A Thermodynamic Parameters for the Adsorption Process

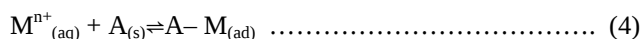
Thermodynamic studies play an important role in determining the spontaneity and the heat change for the adsorption process. The adsorption of the metallic ions (M^{n+}) onto a given adsorbent is a typical example of a heterogeneous equilibrium for which the equilibrium constant for the distribution of the metal ions between the liquid and solid phases K_a , changes in enthalpy (ΔH) entropy (ΔS) and Gibbs free energy (ΔG) can be calculated from thermodynamic relations [15 – 17] such as

$$K_a = \frac{[\text{M}_{ad}]}{[\text{M}_{(aq)}^{n+}]} = \frac{q_e}{c_e} \dots\dots\dots (1)$$

$$\ln K_a = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \dots\dots\dots (2)$$

$$\Delta G_{ad} = \Delta H_{ad} - T\Delta S_{ad} // \dots\dots\dots (3)$$

taking the adsorption process to be represented as



Relevant data calculated from the thermodynamic relations were tabulated in Tables 1 (a & b) respectively. It is evident that the values of ΔH_{ad} were mostly positive, i.e. endothermic, especially at higher adsorbate concentration. One possible interpretation of the endothermicity of the adsorption process was that the metal ions were highly solvated in water. In order for these ions to be adsorbed, the hydration sphere must be removed, and this process requires energy intake. This energy of dehydration supersedes that required for getting the ions attached to the surface of the adsorbent^[15], hence resulting to an overall endothermic nature.

Also evident from the Tables is the positive nature of the ΔS_{ad} values at higher ionic strength i.e. suggesting spontaneous process. This occurs as a result of redistribution of energy between the adsorbate and the adsorbent. Before adsorption occurs, the metallic ions near the surface of the adsorbent were more ordered than in the subsequent adsorbed state. This increase in randomness at the adsorbent – adsorbate interface during the adsorption process leads to the increase in entropy and hence an overall positive ΔS . The adsorption process is therefore likely to occur spontaneously at normal and at high temperatures because $\Delta H > 0$ and $\Delta S > 0$ [18–20].

The Gibbs free energy change (ΔG_{ad}) was negative as expected for a spontaneous process under the applied conditions. The decrease in ΔG_{ad} (i.e. increase negative) values with increase in temperature indicated more efficient adsorption at higher temperature. This is because at higher temperatures ions are easily desolvated and therefore their adsorption becomes more favourable [21, 15, 18].

B. Adsorption Isotherms

Adsorption isotherm studies were carried out with a fixed initial adsorbent dose (8g) and varying adsorbate concentrations [22] and the applicability of the data were tested for the Freundlich, Langmuir and Dubinin – Radushkevich (D-R) adsorption isotherms respectively according to the following relations,

$$\ln q_e = \ln K_F + \ln C_e, \dots\dots\dots (5)$$

$$\frac{C_e}{q_e} = \frac{1}{K q_m} + \frac{C_e}{q_m} \dots\dots\dots (6)$$

$$\text{and } \ln q_e = \ln q_m - K \varepsilon^2 \dots\dots\dots (7)$$

Freundlich Adsorption Isotherm

The various adsorption data for the metal ions onto the adsorbent were tested against the Freundlich model as represented in Fig. 1. Freundlich isotherm gives an expression encompassing the surface heterogeneity and the exponential distribution of active sites and their energies. The numerical values of the constants n_F and K_F for the corresponding plot were determined from the slopes and intercepts of the respective graph (Table 2). The constant K_F is indicative of the relative adsorption capacity of the adsorbent related to the bonding energy and can be defined as the adsorption or distribution coefficient. It represents the quantity of the metal ion adsorbed onto the adsorbent for unit equilibrium concentration. While the constant $a = 1/n_F$ where n_F is the heterogeneity factor, represents deviation from linearity of the adsorption process. Also for all values of $1/n_F$ less than unity, adsorption is the predominant process taking place otherwise desorption becomes predominant [23].

The observed values of $n_F > 1$ for the adsorption of Cr, Ni and Fe onto the sawdust material, the values of the coefficient of variation (R^2) and the linearity of their plots are indicative of the fact that the result of the experimental data in this study fitted well to the Freundlich isotherm. Hence the adsorption of these metal ions onto the adsorbent can be said to be a favourable physical adsorption process.

Langmuir Adsorption Isotherm

The Langmuir model represents one of the first theoretical treatments of non-linear sorption and suggests that the uptake occurs on a homogeneous surface by monolayer sorption without interaction between the adsorbed molecules [24]. The constants k from Langmuir isotherm represents the adsorption equilibrium constant (dm^3/mg) and was computed from the intercepts of the Langmuir plots (Figs. 2) and are presented in Table 2.

The essential characteristics of the Langmuir isotherm may be expressed in terms of the dimensionless separation parameter R_L , computed from the relation;

$$R_L = \frac{1}{1 + k C_0} \dots\dots\dots (8)$$

where k is the adsorption equilibrium constant while C_0 is the initial metal ion concentration. The value of the R_L indicates the type of the isotherm to be either (i) unfavourable if $R_L > 1$, (ii) linear if $R_L = 1$, (iii) favourable if $0 < R_L < 1$ and irreversible if $R_L = 0$. The calculated values of the R_L for the different ions onto the adsorbent (Table 3) under the different conditions studied are in the negative range. This condition indicates that the adsorptions process does not fit to the Langmuir model. However, the case of adsorption of cadmium onto sawdust ($R_L = 1.0000$) points to a linear situation [25, 9, 7].

Dubinin – Radushkevich (D – R) Adsorption Isotherm

The D – R model was applied to the adsorption data to determine if sorption had occurred by physical or chemical processes. The constant K obtained from the slope of the D – R isotherm is a constant related to adsorption energy having units of $\text{mol}^2 \text{kJ}^{-2}$ and it gives an idea about the mean free energy $E(\text{kJmol}^{-1})$ of adsorption per molecule of the adsorbate when it is transferred to the surface of the adsorbent from infinite distance in the solution calculated for the relation

$$E = \frac{1}{\sqrt{2K}} [13, 26, 27, 28].$$

In the D – R isotherm, the sorption energy is independent of the working temperature but varies depending on the nature of the adsorbent and adsorbate [24]. The magnitude of E provides information on the nature of the sorption process, i.e., whether it is chemisorptions or physisorption. If the values of E are in the range of less than 8 kJmol^{-1} the sorption process is physical in nature, otherwise it is a chemical adsorption process [28]. From the D – R plots (Figs. 3) the sorption energies were calculated and tabulated as in Table 4. All the calculated energy values were found to be in the range of less than 8 kJmol^{-1} , which according to Mataka *et al.*, [28] corresponds to physical adsorption process.

Conclusion

Conclusively, it can be observed that the adsorption of the different ions studied is thermodynamically spontaneous and feasible, with spontaneity increasing with increase in temperature and the mean free energy E , of the adsorption suggested that the sorption process is physical in nature and is best defined by Freundlich adsorption isotherms.

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Table 1(a): Thermodynamic Parameters for the Adsorption of the various Metal ions onto Sawdust

	Ka	$\Delta G = \Delta H - T\Delta S$ (Jmol ⁻¹)				ΔH (Jmol ⁻¹)	ΔS (Jmol ⁻¹ K ⁻¹)
		303K	313K	323K	333K		
Cr	0.5708	6199.57	6422.88	6646.19	6869.51	-566.8485	-22.3314
Ni	0.0875	3055.09	3147.46	3239.82	3332.19	256.3206	-9.2369
Fe	0.2375	4249.23	4393.14	4537.06	4680.97	-111.4076	-14.3915
Cd	0.0299	-406.07	-448.81	-491.54	-534.27	888.7666	4.2734

Conditions: 8g Adsorbent, 20mg/L metal ion concentration and 1hr Agitation time

Table 1(b): Thermodynamic Parameters for the Adsorption of the various Metal ions onto Sawdust

	Ka	$\Delta G = \Delta H - T\Delta S$ (Jmol ⁻¹)				ΔH (Jmol ⁻¹)	ΔS (Jmol ⁻¹ K ⁻¹)
		303K	313K	323K	333K		
Cr	0.3156	-581.91	-610.76	-639.60	-668.45	292.2371	2.8850
Ni	0.1125	-2288.96	-2389.39	-2489.83	-2590.26	754.1629	10.0433
Fe	0.4696	-71.7498	-80.0638	-88.3778	-96.6918	180.1644	0.8314
Cd	0.0250	-8936.44	-9293.03	-9649.62	-10006.2	1868.1558	35.6587

Conditions: 8g Adsorbent, 60mg/L metal ion concentration and 1hr Agitation time

Table 2: Numeric Constants for the Adsorption of the Metal ions onto Sawdust Adsorbent

Ion	Freundlich		Langmuir	D – R
	n _F	K _F	k (L/mg)	K (mol ² kJ ⁻²)
Cr	1.4514	0.4325	-0.1595	4 x 10 ⁶
Ni	1.6556	0.2254	-0.0718	1 x 10 ⁶
Fe	1.0627	0.5105	-0.0673	4 x 10 ⁶
Cd	0.5627	0.0015	0.0000	250.9

Table 3: Variation of R_L for the Sawdust Adsorbent with Increase in Initial Metal Ion Concentration

Co	Langmuir Separation Parameter (R _L)			
	Sawdust			
	Cr	Ni	Fe	Cd
20	-0.4565	-2.2941	-2.8947	1.0000
30	-0.2641	-0.8667	-0.9821	1.0000
40	-0.1858	-0.5343	-0.5914	1.0000
50	-0.1433	-0.3861	-0.4231	1.0000
60	-0.1167	-0.3023	-0.3293	1.0000

Table 4: Variation of the Sorption Energy (E) of the Adsorbates onto the Various Adsorbents

Ion	Sawdust	
	K	E
Cr	4 x 10 ⁶	0.0004
Ni	1 x 10 ⁶	0.0007
Fe	4 x 10 ⁶	0.0004
Cd	250.9	0.0446

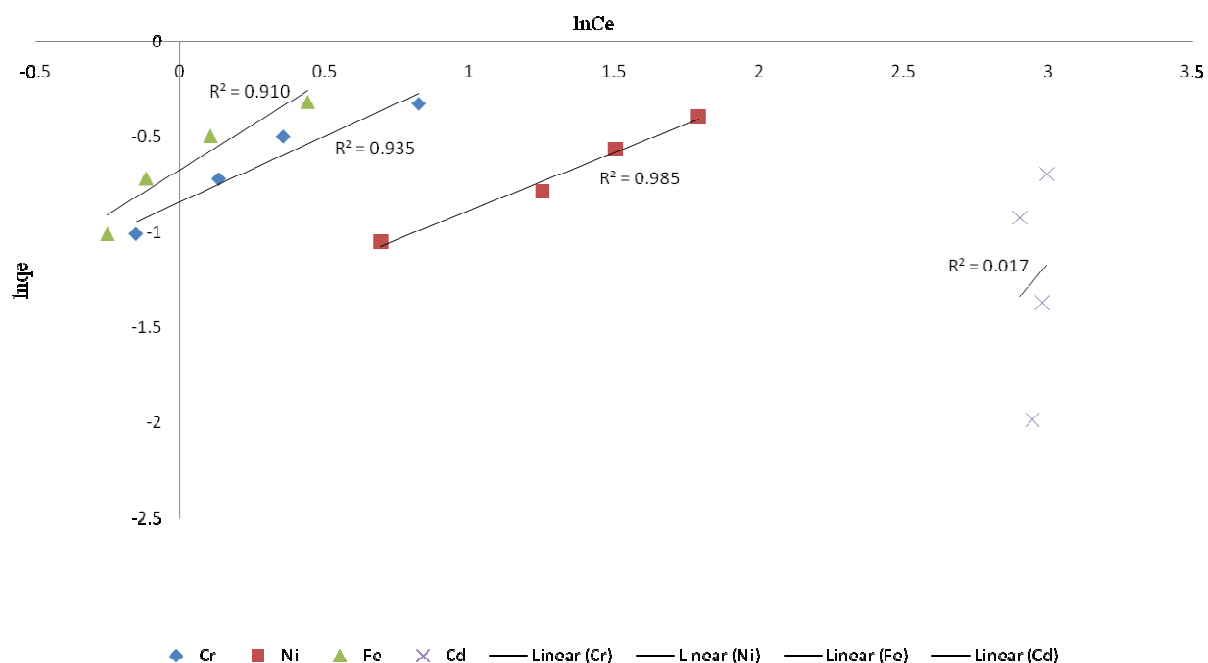


Figure 1: Freundlich Adsorption Isotherm for the Adsorption of the Metal Ions onto Sawdust for 1hr Agitation

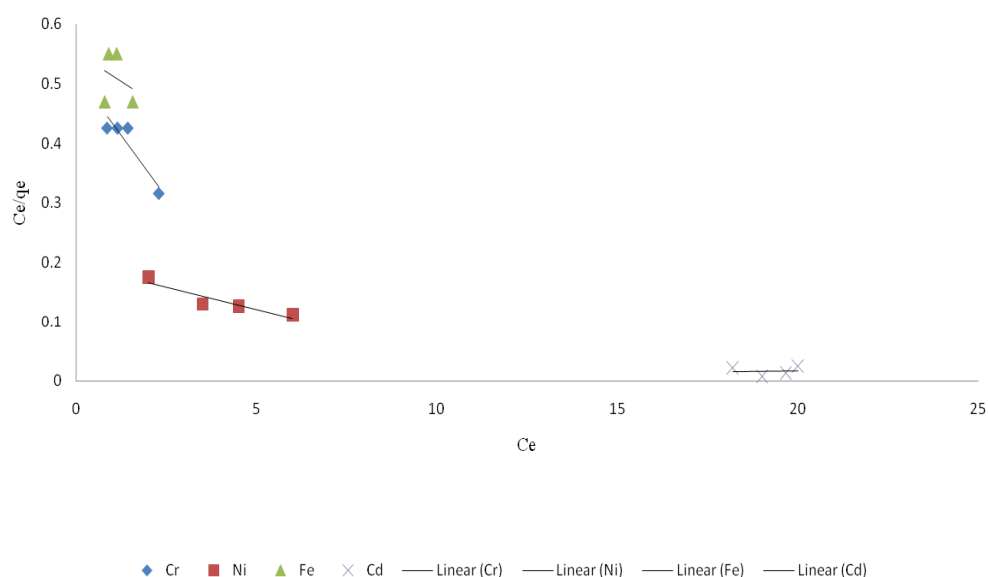


Figure 2: Langmuir Adsorption Isotherm for the Adsorption of the Metal Ions onto Sawdust for 1hr Agitation

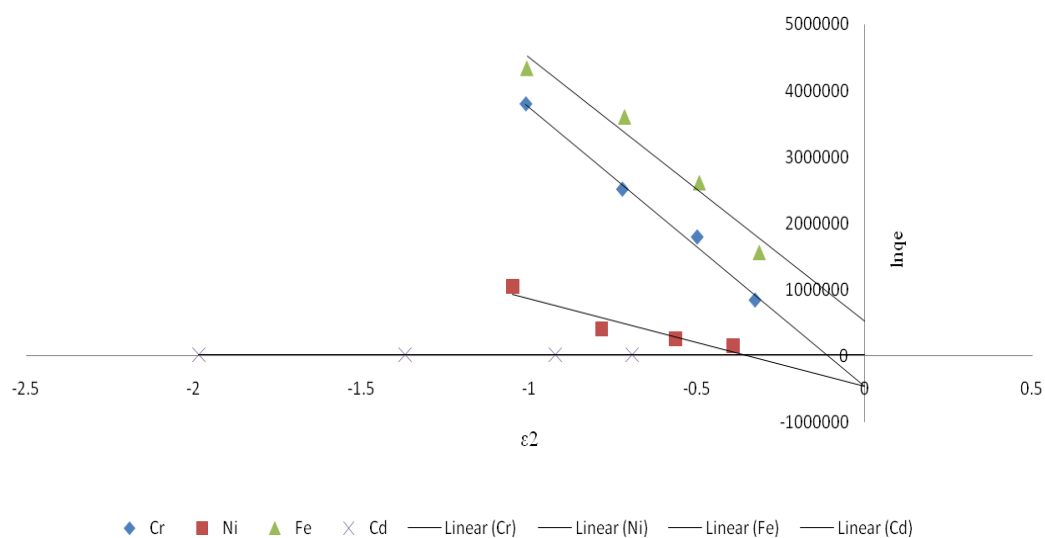


Figure 3: Dubinin - Radushkevich Adsorption Isotherm for the Adsorption of the Metal Ions onto Sawdust for 1hr Agitatio