Studies on Zinc (II) Adsorption Using *Alisma Plantago Aquatica*

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***Abstract*—Adsorption is an effective method to remove heavy metals from wastewater. Alisma Plantago Aquatica was used as an adsorbent to remove Zn(II) from aqueous solution. The equilibrium adsorption were analysed using three isotherm models.The adsorption kinetics followed the pseudo-second order model. Isotherm studies have been used to determine the thermodynamic parameters of the process. Temperature change in the range 20-50^{o}C affected the adsorption capacity. From enthalpy data revealed that the adsorption is endothermic in nature. IR spectrum analysis suggested the presence of hydroxyl, carboxyl and carbonyl groups which are participating in the adsorption of metal ions. The performance of continuous flow packed bed adsorption systems with the biomass was evaluated by plotting the breakthrough curves. It was observed that the breakthrough was strong function of the flow rates and the height of the packed bed. The zinc removal yield decreased with increasing flow rates and initial zinc concentration. Adam Bohart and Wolborska models were applied to the experimental data from dynamic studies on packed bed and were observed to fit the data well with good correlations. The model parameters including the mass transfer coefficient and kinetic parameter were estimated.**

***Index Terms*—Adsorption, heavy metal, isotherm kinetics, thermodynamic.**

I. Introduction

Due to rapid development of industrial activities in recent years, the levels of heavy metals in water system have substantially increased over time. Heavy metals can easily enter the food chain because of their high solubility in water. Due to their toxic effect and accumulation tendency throughout the food chain and the heavy metals pollution represents an important problem, with human health and serious ecological consequences. It is therefore essential to remove heavy metals from industrial waste waters and drinking water.

Zinc is essential in small quantity but when exceeds the prescribed limit it has also detrimental effect on human health. World Health Organization has set a provisional limit of 5mg/l for zinc. It is mostly found in the inorganic form particularly in +2 states. Zinc is used in the process of galvanization,pigment formation,stabilizers, thermoplastics, alloys and batteries. During the metallurgical processes, some amounts of metals are also released into the water bodies.

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Due to their high toxicity, industrial wastewaters containing heavy metals are strictly regulated and must be treated before being discharged in to the environment. A number of technologies have been developed over the years to remove heavy metals from industrial waste water. The most important technologies include chemical precipitation, coagulation process, membrane filtration process etc [1]. These methods are generally expensive, complicated and time consuming. The major advantages of an adsorption system for water pollution control are less investment in terms of both initial cost and land, simple design, easy operation, and the absence of the effect of toxic substances compared to conventional methods [2], [3]. In the last few decades, alternative sorbents for the treatment of heavy metal contamination have been investigated [4]-[7]. There is a large volume of literature relating to the performance of different biosorbents for the removal of variety of heavy metals [8]-[11].

Numerous waste biomass sources are available in nature in which some experimental adsorption properties have been reported e.g rice husk[12], saw dust [13]-[15], tea and coffee waste [16]-[18],orange peel [19] peanut shells[20], activated carbon [21]-[22] dry tree leaves and barks [23]-[25]. When compared with other low cost adsorbents, the results of the present study indicate that adsorbent prepared from A*lisma plantago aquatica* has better adsorption capacity in almost all cases.

Adsorption of heavy metal ions occurs as a result of physicochemical interaction, mainly ion exchange or complex formation between metal ions and the functional groups present on the cell surface.

The main aim of this work is to study the behaviour of a specific biomass. This paper works on the amount of Zn ions that uptake by Alisma Plantago Aquatica by Langmuir and Freundlich isotherm equations for equilibrium, and investigates the kinetic and thermodynamic by different models. The study was carried out in a laboratory fixed bed continuous column using suitable mathematical models [26], [27].

II. Materials and Methods

A. Adsorbent and Instrument.

The experiments were carried out using the adsorbent *Alisma Plantago Aquatica* (AP) .The adsorbent samples were collected from the near by locality and washed several times with distilled water to remove dust and other impurities. After drying, it is then ground using domestic mixer and sieved to 250 mesh size. The sample is washed with distilled water to remove colour and dried in an oven at 60^{o}C for 24

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hours. The dried sample was stored in airtight bottles for further use with out any chemical or physical treatment.

All reagents used in this study were of analytical grade obtained either from Merck Germany. Stock solution of zinc was prepared by zinc nitrate salts in double distilled water. Zn(II) solution of different concentrations were obtained by diluting the stock solution.

Analysis of each Zn(II) remaining in solution and initial metal concentration was determined using Atomic absorption spectrophotometer (Thermofisher iCE:3000).

FT-IR Spectra were obtained on a Perkin Elmer (Spectrum two) spectrometer.

III. Results and Discussion

A. Adsorption Kinetics

To study the adsorption kinetics of Zn(II) onto *Alisma Plantago Aquatica,* first order kinetics and second order kinetics were applied to the batch experimental data. In order to define the adsorption kinetics of heavy metal ions, the kinetic parameters for the adsorption process were studied for contact times ranging between 1 and 150min. The kinetics in most cases follows the first order rate equation.

$$\frac{dq}{dt} = k_1(q_e - q) \tag{1}$$

where q_e and q are the adsorption capacity at equilibrium and at time t respectively, and k_1 is the rate constant of the pseudo first order adsorption process. The integrated linear form of Eq.(1) can be expressed as follows:

$$\log(q_e - q) = \log(q_e) - \frac{k_1}{2.303}t \tag{2}$$

Plot of $\log(q_e-q)$ vs. t gives a straight line for first order adsorption kinetics and the rate constant k_1 is computed from the plot.

The sorption data was also studied by second order kinetics

$$\frac{dq}{dt} = k_2(q_e - q)^2 \tag{3}$$

where k_2 is the second order rate constant.

After integration,

$$\frac{1}{q_e - q} = \frac{1}{q_e} + k_2 t \tag{4}$$

This can be written in the linear form on further simplification

$$\frac{t}{q} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{5}$$

The applicability of this equation can be studied by a plot of t/q vs. t and the rate constant k_2 and q are obtained from the plot.

The intra particle diffusion model was used to understand the diffusion mechanism. Intra particle diffusion was characterized using the relationship between specific sorption (q) and the square root of time ($t^{1/2}$)

$$q = k_d t^{1/2} \tag{6}$$

where k_d is the intra-particle diffusion rate constant and the rate constant k_d directly evaluated from the slope of the regression line.

The second order equation appeared to be the better fitting model than other models because it has higher R^2 value. The values of the first order, second order and Intra particle diffusion kinetic models are tabulated in the Table I.

TABLE I: Kinetics Parameters for the Adsorption of Zn(II) onto *Alisma Plantago Aquatica*

q_{exp} (mg/g)	First order			Second order			Intra Particle	
	R^2	q_{calc}	k_1	R^2	q_{calc}	k_2	k_d	R^2
14.73	0.844	3.78	0.033	0.999	15.27	0.016	0.302	0.918

B. Adsorption Isotherms

During adsorption a rapid equilibrium is established between adsorbed heavy metal ions on AP (q_{eq}) and unadsorbed heavy metal ions in solution (C_{eq}). This equilibrium can be analysed by mathematical representation called adsorption isotherms.

The adsorption coefficient q is calculated from the expression

$$q = \frac{V(C_i - C_f)}{M} \tag{7}$$

where V is the volume of the solution, C_i and C_f are initial and equilibrium concentrations and M is the dry mass of adsorbent.

The most widely used isotherm equation for modeling the equilibrium is the Langmuir equation which is valid for monolayer sorption on to a surface with a finite number of identical sites and is given by equation

$$q = \frac{q_{\max} b C_f}{(1 + b C_f)} \tag{8}$$

where q_{max} is the maximum amount of the metal ion per unit weight of the adsorbent to form a complete monolayer on the surface bound at high C_f and b is a constant related to the affinity of the binding sites q_{max} represents a practical limiting adsorption capacity when surface is fully covered with metal ions and assists in the comparison of adsorption performance particularly in cases where the sorbent did not reach its full saturation in experiments. q_{max} and b can be determined from the linear plot of C_f/q versus C_f. The linearized form of this model equation is given as

$$\frac{C_f}{q} = \frac{C_f}{q_{\max}} + \frac{1}{b q_{\max}} \tag{9}$$

The empirical Freundlich model also considers mono molecular layer coverage of solute by the adsorbent. However, it assumes the adsorbent has a heterogeneous surface so that binding sites are not identical. This model takes the following form for a single component adsorption.

$$q = KC_f^{1/n} \quad (10)$$

where K and n are the Freundlich constants characteristic of the system. K and n are indicators of adsorption capacity and adsorption intensity respectively. Through the Freundlich isotherm is more widely used, it provides no information on the monolayer adsorption capacity, in contrast to the Langmuir model.

$$\log q = \log K + \frac{1}{n}\log C_f \quad (11)$$

Langmuir and Freundlich isotherms are insufficient to explain the physical and chemical characteristics of adsorption. Dubinin – Radushkevich (D-R) isotherm is commonly used to describe the adsorption isotherms of single solute systems.

The D-R isotherm is expressed as

$$q = q_{\max} \exp\left(-B\left[RT\ln\left(1+\frac{1}{C_f}\right)\right]^2\right) \quad (12)$$

$$\ln q = \ln q_{\max} - Be^2 \quad (13)$$

where B is a constant related to the adsorption energy, R is the gas constant (8.314×10^{-3}kJ/molK) and T is the absolute temperature

$$e = RT\ln\left(1+\frac{1}{C_f}\right) \quad (14)$$

$$E = \frac{1}{(2B)^{0.5}} \quad (15)$$

Study of variation of the initial heavy metal concentration at a fixed amount of 0.25g/100ml was carried out at room temperature. Adsorption isotherms show the distribution of solute between the liquid and solid phases and can be described by several mathematical relationships such as the standard Langmuir and Freundlich models. The linearized Langmuir and Freundlich adsorption isotherms obtained shown in the Table II with the values of linear regression coefficients. In view of the values of the linear regression coefficients, both the models fitted very well to the sorption data in the studied concentration range.

An adsorption isotherm is characterized by certain constants the values of which express the surface properties and affinity of the sorbent. The Freundlich constants k and $1/n$ are adsorption capacity and adsorption intensity are determined for Zn(II) at all values of temperature. The Langmuir constants of q_{max} and b were determined from C_f/q versus C_f plot. The applicability of both Langmuir and Freundlich isotherms implies that both monolayer adsorption and heterogeneous surface conditions exist under the experimental conditions used.

TABLE II: COMPARISON OF FTIR BAND POSITIONS OF RAW *ALISMA PLANTAGO AQUATICA* (AP) BEFORE AND AFTER ZINC ION ADSORPTION IN WAVE NUMBER(CM^{-1})

T(K)	Langmuir model			Freundlich model			Dubinin Radushkevich		
	R^2	q_{max}	b	R^2	1/n	K	R^2	q_{max}	E
293	0.99	24.27	0.07	0.99	0.59	2.18	0.88	14.87	0.49
303	0.99	25.12	0.08	0.99	0.59	2.36	0.83	14.92	0.59
313	0.99	25.83	0.09	0.995	0.58	2.74	0.90	16.34	0.65
323	0.995	26.67	0.11	0.997	0.56	3.19	0.85	16.56	0.81

C. Thermodynamic Parameters

The experiments were conducted at different temperatures (20,30,40,50°C).The thermodynamic parameters such as change in standard free energy (ΔG°), enthalpy (ΔH°) and entropy (ΔS°) can be determined by using the following equations:

$$\Delta G = -RT\ln b \quad (16)$$

$$\ln b = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (17)$$

$$\Delta G = \Delta H - T\Delta S \quad (18)$$

where R (8.314 J/mol K) is the gas constant,T(K) the absolute temperature and b (L/mol) is the thermodynamic equilibrium constant. A plot of Gibbs free energy (ΔG) versus temperature was found to be linear. The values of enthalpy and entropy were obtained from the slope and intercept of the plots. Positive values of ΔH indicate that the adsorption of metal ions on the adsorbent is endothermic.The negative values of ΔG reveal the feasibility and spontaneous nature of the process. This results also supports the adsorption capacity of *Alisma Plantago Aquatica* for Zn(II) increases with increasing temperature. The ΔS values are very small which shows the entropic change occurring during adsorption process is negligible. The values of the thermodynamic parameters are tabulated in the Table III.

TABLE III: THERMODYNAMIC PARAMETERS FOR THE ADSORPTION OF ZN(II) ON AP AT VARIOUS TEMPERATURES.

T(K)	b	ΔG	ΔH	ΔS	R^2
	($Lmol^{-1}$)	($kJmol^{-1}$)	($kJmol^{-1}$)	($kJmol^{-1}K^{-1}$)	
293	4570.76	-20.53			
303	4904.25	-21.41	11.364	0.1086	0.9964
313	5800.10	-22.55			
323	6918.30	-23.74			

D. Characterization of the Adsorbent

Fourier transform infrared spectral analysis was carried out in order to identify the different functional groups present in the given samples. The different functional groups which are present in the given samples are OH stretching, CH stretching, C=C stretching, C-O stretching is showed in Fig. 1. FTIR spectrum of given raw AP sample shows a broad and intense peak at 3330.18cm^{-1} which can be attributed to the stretching of O-H group due to inter and intramolecular hydrogen bonding of polymeric compounds such as alcohols or phenols. The peak observed at 2918cm^{-1} was associated with the stretching vibrations of C-H bond of methyl, methylene and methoxy groups .The peaks around

1600-1627cm^{-1} corresponded to the C=C stretching which might be attributed to the presence of aromatic or olefinic or N-H bending bands. The intense peak at 1000-1031cm^{-1} corresponded to the C-O stretching of alcohol or carboxylic acid. FTIR spectrum of the given raw AP loaded with Zn(II) shows that the peaks at 3330,2918,1606,1009 cm^{-1} (before adsorption) had shifted slightly after binding with zinc as shown in the Table IV. . This was due to the participation of these functional groups in the binding of metal ions. It was also noted that shifting of wave number depends on the concentration of the metal present in the given sample agreed by literature survey.

The scanning electron micrograph revealed the surface texture and morphology of the adsorbent is showed in Fig. 2.

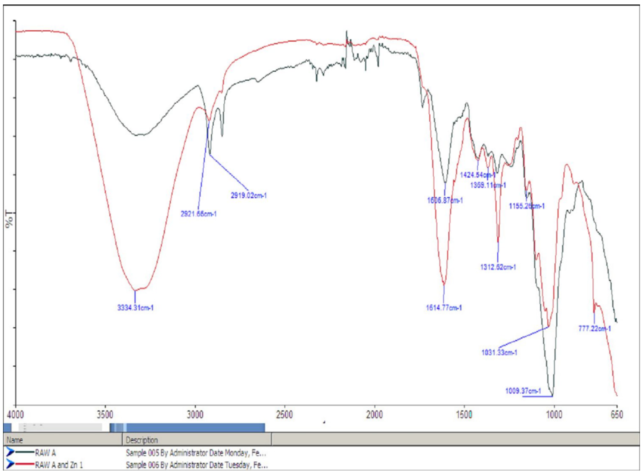

Fig. 1. FTIR spectra of AP before and after adsorption on Zn(II)

TABLE IV: COMPARISON OF FTIR BAND POSITIONS OF RAW ALISMA AP BEFORE AND AFTER METAL IONS ADSORPTION IN WAVE NUMBER(CM^{-1}).

Assignment	Raw AP	After adsorption
O-H stretching	3330.18	3334.31
C-H stretching	2918.70	2921.66
C=C stretching	1606.90	1614.77
C-O stretching	1009.48	1031.33

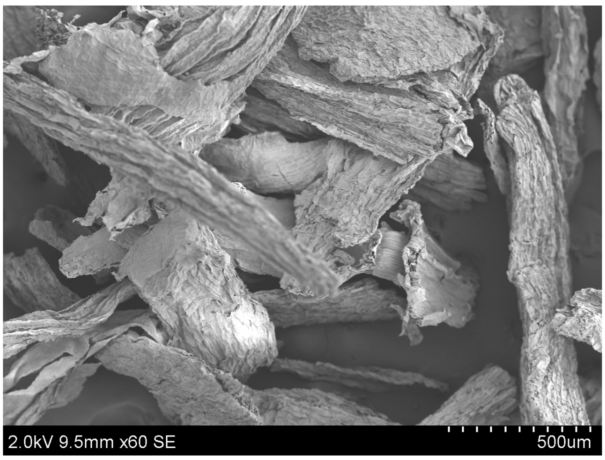

Fig. 2. SEM photograph of AP

E. Continuous Study

A continuous fixed bed study was carried out using *Alisma Plantago Aquatica* as a adsorbent for the removal of zinc. In column operation, rate of adsorption depends on the concentration of metal in the solution being treated. As the adsorbent is continuously in contact with fresh metal bearing solution, the concentration of the solution in contact with a given layer of adsorbent in the column is relatively constant. The performance of packed bed column was analysed using the effluent concentration versus time curves. For adsorption, the plot is usually referred to as the breakthrough curve.

The relative concentration of metal ions in treated effluent was plotted as a function of time in the form of breakthrough curves as illustrated by representative data in Fig. 3 –Fig. 4. As the flow rate increases, the breakthrough curves become steeper and the breakpoint time decreases. The flow rate affects the slope of the linear part of the breakthrough curve. Saturation time is also greatly affected by the flow rate. As flow rate is increased, the time required for breakthrough point of zinc uptake decreased. At lower inlet zinc concentration the treated volume was greater.

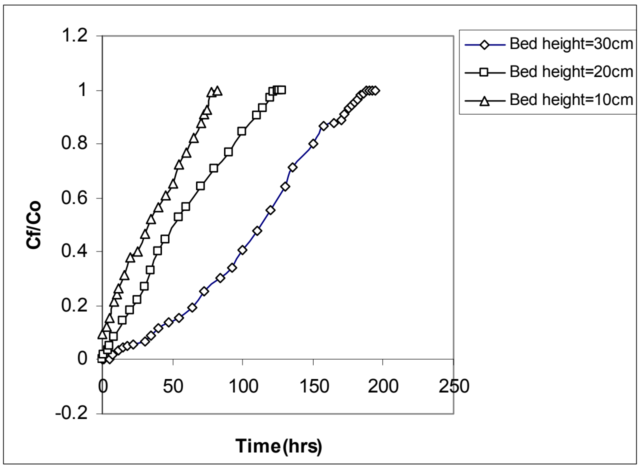

Fig. 3. Breakthrough curves at different bed depths for zinc adsorption. Flow rate 3ml/min, C_o = 10mg/l

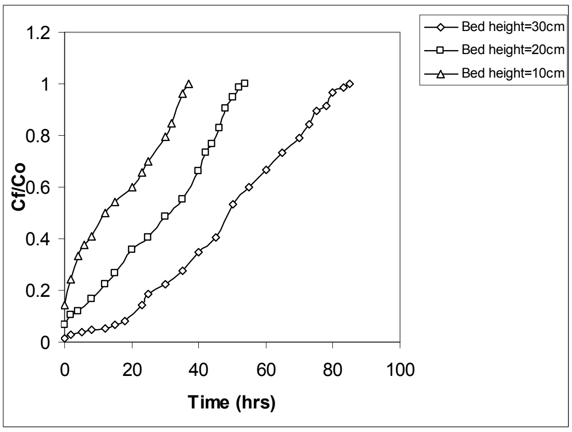

Fig. 4. Breakthrough curves at different bed depths for zinc adsorption. Flow rate 6ml/min, C_o = 10mg/l

1) *Mathematical description*

The performance of packed beds is described through the concept of the breakthrough curve. The time for breakthrough appearance and the shape of the breakthrough curve are very important characteristics for determining the operation and the dynamic response of a adsorption column.

The breakthrough curves show the loading behavior of zinc to be removed from solution in a fixed bed and is usually expressed in terms of adsorbed zinc concentration (C_{ad}) =

inlet zinc concentration (C_o) – outlet zinc (C_f)

The maximum (equilibrium) capacity of the column for given feed concentration is equal to the area under the plot of the adsorbed metal ion concentration C_{ads} versus time or the area behind the breakthrough curve. The amount of metal that remains in the effluent, C_{eq}, is the area under the breakthrough curve as shown in Fig. 3 –Fig. 4

$$C_{\max} = Q \int_{t=0}^{t=t_{total}} C_{ad} dt \tag{19}$$

$$W = QC_o t_{total} \tag{20}$$

$$Y = \frac{C_{\max}}{W} 100 \tag{21}$$

In the first stage of removal studies in the continuous flow fixed column with Alisma Plantago Aquatica, the flow rate was changed from 3ml/min to 6ml/min.and the zinc concentration in the feed was kept constant at 10ppm.From the Table V at the lowest flow rate of 3ml/min, relatively higher uptake capacity values were observed. From the Table V at the lowest flow rate of 3ml/min, relatively higher uptake capacity values were observed. In general the total adsorbed zinc quantity, maximum zinc uptake and zinc removal percentage values decreased with increasing flow rate.

TABLE V: EFFECT OF FLOW RATE AND BED DEPTH ON THE ADSORPTION OF ZN(II) BY *ALISMA PLANTAGO AQUATICA*

Flow rate (ml/min)	Conc. (mg/l)	Bed depth (cm)	Maximum equilibrium capacity(mg)	Metal loading(mg)	Uptake capacity (mg/gm)	Yield (%)
3	10	10 20 30	62.0 101.8 223.0	147.6 230.4 349.2	15.5 12.7 18.6	42.0 44.2 63.8
6	10	10 20 30	51.1 105.0 180.9	133.2 194.4 306.0	12.8 13.1 15.1	38.3 54.0 59.1

2) *Application of the Adams -Bohart and Wolborska models*

The fundamental equations describing the relationship between C/C_o and t in flowing systems were established by Adams -Bohart and Wolborska models. Adams –Bohart model is used for the description of the initial part of the breakthrough curve and is given by the following equations, with parameters k and N_o.

$$\ln \frac{C}{C_o} = kC_o t - kN_o \frac{Z}{U_o} \tag{22}$$

where C is the solute concentration (mg/l); Z the bed depth (cm); k the kinetic constant (l/mg hr); U_o the linear flow rate (cm/hr), defined as the ratio of the flow rate Q (ml/hr) to the cross sectional area A (cm^2) and N_o the saturation concentration (mg/l).

Wolborska model is also used for the description of adsorption dynamics using mass transfer equations for diffusion mechanisms in the range of the low concentration breakthrough curve. The following relationship describes the concentration distribution in the bed for the low concentration region.

$$\ln \frac{C}{C_o} = \frac{\beta C_o}{N_o} t - \frac{\beta Z}{U_o} \tag{23}$$

where β is the coefficient of the external mass transfer (1/hr).The expression of the Wolborska solution is equivalent to the Adams – Bohart relation if the coefficient k is equal to β/N_o.

Both the model expression can be linearized to give a relationship between $\ln(C/C_o)$ and time from which the model parameters can be calculated. It was observed that the model predictions agreed very closely with experimental data giving a linear relationship up to 50%breakthough, for all breakthrough curves(R^2 >0.9). The model parameters, rate constant (k) or mass transfer coefficient (β) and the adsorption capacity (*No*) were calculated from the slope and intercept of the lines respectively. The values of the Adams -Bohart and Wolborska model parameters corresponding to the experimental conditions and determination coefficient (R^2) are given in the Table VI.

The values shown in table was obtained after applying the respective model equations to the experimental data for varying bed depths zinc adsorption column. However Adams -Bohart and Wolborska model predictions of N_o matched well. Although the Adams -Bohart and Wolborska model provides a simple and comprehensive approach to running and evaluating adsorption column tests, its validity is limited to the range of conditions used.

TABLE VI: ADAMS -BOHART AND WOLBORSKA MODEL PARAMETERS CORRESPONDING TO EXPERIMENTAL CONDITIONS

Flow rate (ml/min)	Conc. (mg/l)	Bed depth (cm)	Adams -Bohart		Wolborska		R^2
			N_o (mg/l)	k (l/mg hr)	β (1/hr)	N_o(mg/l)	
3	10	10 20 30	425.32 533.42 641.14	0.00763 0.00303 0.00251	3.245 1.616 1.609	425.30 533.33 641.03	0.964 0.975 0.975
6	10	10 20 30	597.20 695.60 487.62	0.00701 0.00439 0.00725	4.18 3.05 3.53	597.20 695.44 486.89	0.949 0.974 0.983

IV. CONCLUSION

This study indicates that *Alisma Plantago Aquatica* which is low cost, can be used as an efficient biosorbent material for removal of Zn(II) from wastewater. The adsorption isotherms at different temperatures could be well described by the three isotherm models. IR spectrum analysis suggested the different functional groups which are present in the given sample. The thermodynamic study shows that the adsorption of Zn(II) was endothermic in nature. The negative values of ΔG reveal the feasibility and spontaneous nature of the process. The performance of packed column was described through the concept of breakthrough and the values of column parameters predicted as a function of bed depth. Column studies showed good agreement between the experimental data obtained with Adams – Bohart or Wolborska model.

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