



Adsorption Studies of Lead (II) Ions from a Synthetic Media using Jackfruit (*Artocarpus heterophyllus* L.) Rags: Kinetics, Equilibrium and Thermodynamic Studies

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Abstract- Water is vital to humans, animals and the entire ecosystem. However, its quality is deteriorating every dawn due to increased industrial advancements in our contemporary society leading to careless discharge of both organic and inorganic pollutants to the environment. This is not limited to metal ions which are eventually disposed to water bodies. Efforts to their remediation has yielded insignificant results as the used decontamination conventional techniques are costly. The use of adsorption using locally available adsorbents from agricultural wastes have increased research interest. Jackfruit rags (JR), a waste of Jackfruit was utilized as an adsorbent in lead (II) ions de-contamination from water. Powdered adsorbent was characterized using FT-IR which showed the presence of hydroxyl (-OH) and carboxylate (-COOH), carbonyl (-C=O) and ether (-C-O-C-) as important adsorption sites for lead (II) ions adsorption. The influence of contact time, pH, adsorbent dose, temperature and initial lead (II) ions concentration was investigated by batch adsorption technique. The optimal pH, time, dosage, temperature and initial lead (II) concentration was found to be: 5, 30 minutes, 0.08 g, 25 °C, 30 mg L⁻¹ respectively. Isotherm studies showed that Freundlich model best described the lead (II) ions adsorption with adsorption capacity of 4.6716 mg/g which described a physisorption process. Kinetic parameters suggested the pseudo-second-order adsorption models best described the adsorption process. Thermodynamic functions of ΔG° , ΔH° and ΔS° showed that lead (II) ions adsorption onto JR adsorbent was spontaneous, exothermic, non-entropy driven and physisorption in nature. The findings of the study showed that Jackfruit rags can be applied as an alternative, cheap and eco-friendly adsorbent in heavy metal ions removal from drinking water both at household level and industrial level.

Keywords: Heavy metals, Jackfruit rags, Adsorption, kinetics, isotherms, thermodynamics

I. INTRODUCTION

Safe water is vital for life [1]. Existing water sources such as lakes, seas and groundwater sources are becoming polluted every dawn by discharge of vast contaminants among them being toxic heavy metal ions majorly due to municipal and industrial modern engagements [2]. Heavy metal water pollution has become of global environmental concern [3]. Characteristically, once in the water bodies, they are toxic, non-biodegradable and persistent [4]. They are therefore bioaccumulated and biomagnified in the food chain to ascending trophic levels and finally to human beings to levels exceeding set maximum limits leading to health effects [5]. The associated health problems are such as cancer, kidney failure, skin damage, liver dysfunction stomach damage amongst others [6,7]. Therefore, reducing these heavy metal pollutants to permissible levels [8,9,10,11] from water bodies has drawn global attention from the scientists and government research bodies.

A variety of decontamination techniques: membrane filtration, precipitation, ion exchange, adsorption amongst others have been explored for heavy metal ions removal in drinking water [12,13,14]. Among them, adsorption technique have remained the most effective technique due to its easy in operation, cost-effectiveness, easy in design and environmentally friendly [15]. Cellulose, being an abundant biopolymer is reported as widely used adsorbent for their de-contamination [16]. Recently, the use of agricultural based wastes as low-cost adsorbents for water purification have increased research interest [17].

Jackfruit (*Artocarpus heterophyllus* L.) is one of the largest ever known fruit worldwide [18] with about 35 % edible flesh (yellowish bulbs) with the rest: rags, seeds, peels and the core (65 %) discarded as a waste [19,20]. Due to their non-seasonal nature, its consumption increases the accumulation of biowastes disposal to both terrestrial and aquatic environment creates an environmental problem of foul odour and taste [21]. Utilization of biowaste as low-

cost adsorbents in their de-contamination will provide a solution not only to the above problem but also in water purification. Various studies are reported on the use of some of the biowastes in their removal [15, 22, 23, 24, 25, 26]. To our best of authors' knowledge, no information have been reported on the use of Jackfruit rags as an adsorbent. Therefore, this study assesses the use of Jackfruit rags adsorbent in studying the kinetics, equilibrium and thermodynamic adsorption studies of lead (II) ions from water.

II. METHODOLOGY

Materials

The chemicals used were of AR grade. The solutions were prepared using deionized water at a room

temperature (25 ± 1 °C). Pb (NO₃)₂, HNO₃, KOH) and C₂H₃O₂Na were commercially purchased from Sigma Aldrich (Kobian, Nairobi Kenya).

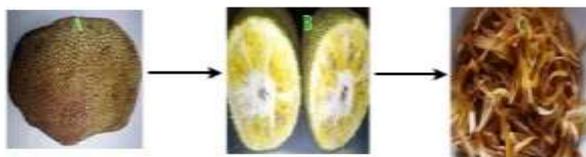


Fig-1: Jackfruit (A); Transverse section of Jackfruit (B); Jackfruit rags (C)

Adsorbent preparation

Jackfruits were collected from Maragua market, Murang'a, Kenya. They were then transported to the laboratories, washed to remove any dirt, cut open and fruit sample (rags) were obtained. The sample was chopped into pieces and oven dried at 105 °C for 24 hours to a constant weight to eliminate total moisture content. The adsorbent was ground into powder using a manual grinding machine (Retsch SR 200). The resulting powder was sieved by a fine-mesh sieve (150 to 250 mm) and kept in a desiccator labelled Jackfruit rags (JR) adsorbent awaiting subsequent adsorption studies.

Metal ion solutions preparation

1000 mg L⁻¹ stock solutions of Pb²⁺ metal solutions were made in C₂H₃O₂Na buffer media. Working solutions were then prepared from stock solution by dilution. 0.1N HNO₃ or 0.1 N KOH solutions were used for pH adjustments of working solutions. Fresh dilutions were used in each adsorption experiments.

Instrumentation

The Jackfruit rags adsorbent was characterized using a Shimadzu Fourier Transform Infrared (FTIR-IR Tracer-100 made in Japan) spectrophotometer to identify the functional groups in JR responsible for metal ions adsorption²³. The concentration of Pb²⁺ ions in the solution was determined by Shimadzu (AAS-AA 6200). Reciprocating mechanical shaker (SSL₂ Harrogate, UK)

was used for batch adsorption. pH meter of HANNA model adjusted pH of Pb²⁺ metal solutions.

Batch adsorption

The effect of contact time, pH, dosage and initial lead (II) ions concentration were optimized by batch method using 120 mL plastic screw cap bottles by keeping other parameters constant while varying the one under investigation. Influence of pH (1-9), contact time (0-60 minutes), dosage (0.01 – 0.08 g), temperature (25-80 °C) and initial lead (II) concentration (10 -100 mg L⁻¹) were investigated in 20 mL aqueous solution at a constant agitation speed of 150 revolutions per minute. The amount of lead (II) ions adsorbed by JR was calculated by the equation:

$$q_e = \frac{C_i - C_e}{M} V \quad [1]$$

Where q_e is the amount of adsorbed lead (II) at equilibrium, C_i is initial lead (II) concentration and C_e is the final lead (II) concentration at equilibrium (mg L⁻¹), V is the volume of the lead (II) solution and M is the amount of dose [23].

The % removal of Pb²⁺ ions by JR was calculated by the equation:

$$\% \text{ removal} = \frac{C_i - C_e}{C_i} \times 100 \quad [2]$$

Where C_i is the initial lead (II) concentration and C_e is the final lead (II) concentration [17].

III. RESULTS AND DISCUSSIONS

FTIR Characterization

The functional surface groups in JR adsorbent were detected by FT-IR as shown by spectrum in figure 2:

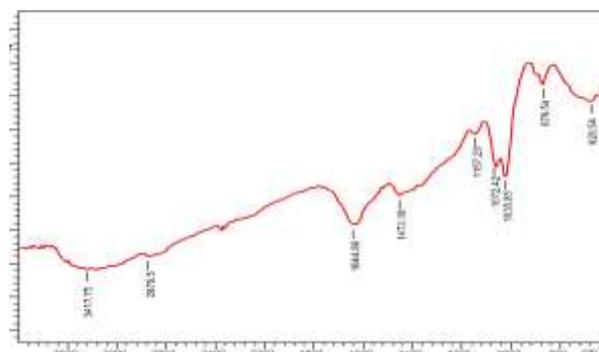


Fig-2: FT-IR spectrum of Jackfruit rags (JR) adsorbent

FT-IR spectrum results in figure 1 showed a broad band at 3417.75 cm⁻¹ indicated free and intermolecular bonded -OH groups in alcohols or phenols or carboxylic functional groups in polymeric compounds [27]. A weak peak at 2879.50 cm⁻¹ could be ascribed to symmetrically and asymmetrically sp³ C-H stretching vibrations in aliphatic polymeric compounds. The peak at 1644.18 cm⁻¹ could be

a characteristic vibration of $-C=O$ stretching from aldehydes and ketones [28]. Peak band at 1473.18 cm^{-1} can be associated with the stretching and flexion vibrations of methylene ($-CH_2$) and methoxy (CH_3-O-) and methyl (CH_3-) groups [23]. Peaks at 1157.29 cm^{-1} and 1035.85 cm^{-1} may be ascribed to C-O-C stretching vibrations of esters and ethers in the pyranose skeletal ring of polymeric compounds. The bands at 620.54 cm^{-1} [29] and 879.54 cm^{-1} is attributed to bending vibrations of aromatic compounds. $-OH$, $-C=O$) and $-COO-H$ functional groups are therefore important sorption sites for uptake of lead (II) ions.

OPTIMIZATION

Influence of pH

The influence of pH on adsorption of lead (II) ions was investigated by varying pH values (1-10) and the results are presented by figure 3:

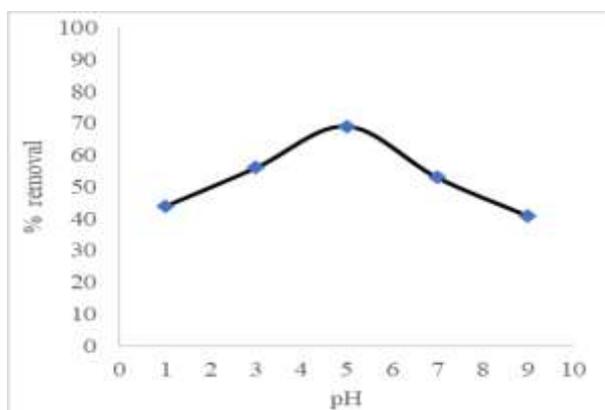


Fig-3: Influence of pH on adsorption of Pb (II) ions onto JR adsorbent

The influence of pH on the removal of lead (II) ions is shown in the figure 2. The optimum pH is 5 with a maximum removal percentage of 69.81 %. The percentage removal increases as pH is increased to optimum then it decreases. At low pH values, H_3O^+ ions concentration is high, which protonates carboxylate groups making the surface more positive. [4,15,23]. This makes the $-COOH^+$ groups lose affinity for metal ions [30] by inducing electrostatic repulsive forces between the JR surface and lead (II) ions. This leads to competition between H_3O^+ ions and lead (II) ions in solution thereby decreasing the removal efficiency of the lead (II) ions [31]. At pH values above optimal, soluble lead (II) ions are solvated forming soluble hydroxylated complexes that compete for active sites on the adsorbent [4]. This reduces the amount of lead (II) ion the solution to be adsorbed at the adsorbent surface²⁹. This is summarized by the following general reactions [4].



The results concur with those reported by [32,33].

Influence of contact time

The varied time intervals (10, 20, 30, 40, 50 and 60 minutes) was used to investigate influence of contact time and results are presented in figure 4:

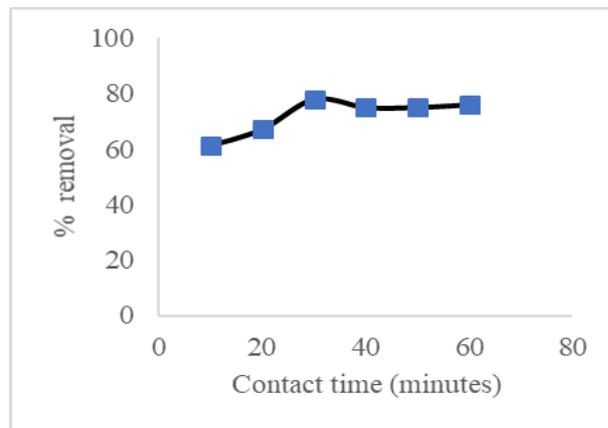


Fig-4: Influence of contact time on adsorption of Pb (II) ions onto JR adsorbent

Removal of lead (II) ions was influenced by the residence time as shown in figure 3. Removal of lead (II) ions was rapid within the first 30 minutes with removal percentage of 78.15 %. Thereafter, there was a plateau which indicated equilibrium state. At initial time, there are large number of vacant active sites and increased concentration gradient [34] which increased the adsorption process to optimum [23]. Beyond this, the rate levelled off. At equilibrium, the surface sites are exhausted and tend to worn out [35] and there exist a repulsive force between the lead (II) ions adsorbed and the ones in the solution making even the few remaining available sites difficult to be filled with lead (II) ions [36,37]. The results are coherent with those reported by [38, 39, 40].

Influence of dosage

The influence of JR dosage on lead (II) ions adsorption was evaluated by changing the dose from 0.01g-0.15g. Results are presented by figure 5:

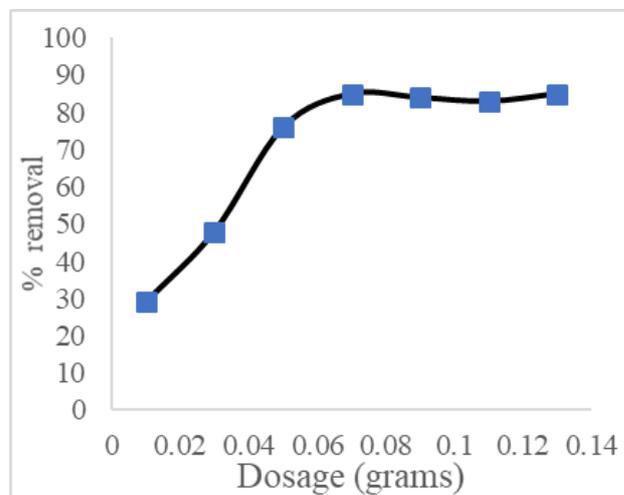


Fig-5: Effect of adsorbent dose on adsorption of Pb (II) ions onto JR adsorbent

Results in figure 4 showed that lead (II) ions uptake was influenced by adsorbent dose. The % removal of lead (II) ions increased with increase in dosage (0.01 g) to optimal (0.08 g) with percentage uptake increase from 29.15 % to maximum of 85.62 % beyond which a plateau was observed. Increase in dosage increased the exposure of more available active sites for adsorption of lead ions leading to an increase in percentage removal [41]. A plateau state beyond optimal was due to burying of the active sites due to overcrowding and this reduced the adsorbent-adsorbate interaction making it difficult for the active sites to adsorb the metal ions in the solution [23,34]. 0.08 g was taken as the optimal dose for subsequent doses. These findings compare with those reported by [42,43] in the study of lead (II) ions adsorption on fly ash based-geopolymer.

Influence of temperature

The influence of temperature (15-65 °C) was investigated and results are presented in figure 6:

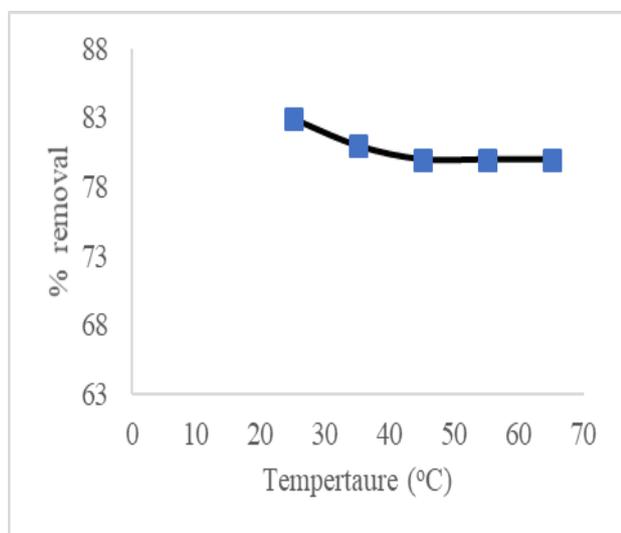


Fig-6: Effect of temperature on adsorption of Pb (II) ions on JR adsorbent

Temperature increase (25 °C to 65 °C) decreased the removal % of lead (II) ions. This indicated that adsorption was maximum at a room temperature (25 °C) with a maximum percentage removal of 83.61 %. This is due to a reduced adsorbent surface activity which showed that removal of lead (II) ions was predominated by physical adsorption and exothermic in nature [44]. Therefore, effect of initial concentration was determined at 25 °C. The results compare with those reported by [45,46].

Influence of initial concentration

The effect of initial concentration (10-100 mg L⁻¹) at pH=5, contact time= 30 minutes, dosage = 0.08 g and temperature= 25 °C was evaluated and results are presented in figure 7:

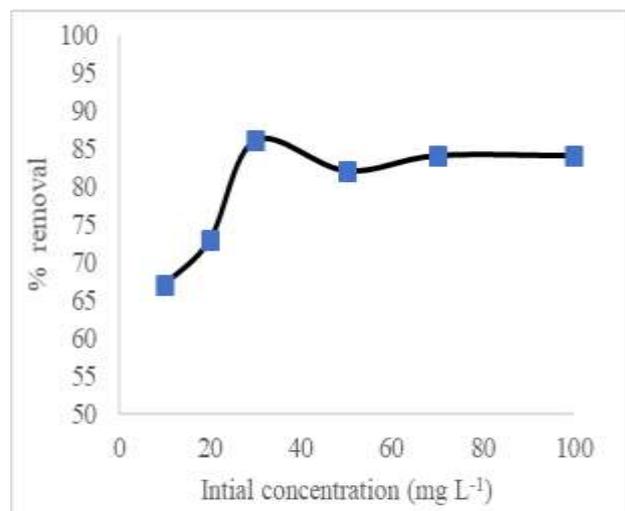


Fig-7: Effect of initial concentration on adsorption of Pb (II) ions on JR adsorbent

The % of lead (II) removal increased substantially with increased initial concentration from 10 mg L⁻¹ to 30 mg L⁻¹ beyond which a plateau state was observed. The uptake increased from 67.97 % to 86.28 %. The trend can be explained by the fact that at low initial concentration values, there are available adsorbent sites to adsorb the less available ions in the metal solution but as the concentration is increased with unchanging amount of adsorbent sites led to increased in their percentage removal to optimal concentration [47] of 30 mg L⁻¹. At concentration beyond optimal, the number of exchangeable adsorbent active sites are already used up due to higher Pb²⁺/ adsorbent sites ratio and this rendered no significant change in lead (II) ions uptake [48]. The results are coherent with those reported by [23] in their study on the lead (II) ions removal onto ethylenediamine modified Jackfruit seeds from aqueous solution.

ADSORPTION ISOTHERM STUDIES

The experimental data as a function of initial concentration was analysed using Langmuir and Freundlich isotherm models. The linearized form of Langmuir isotherm equation is given by the equation:

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{K_L q_m} \quad [6]$$

Where q_e (mg/g) is the adsorbed lead (II) ions at equilibrium, q_m (mg/g) is the maximum amount of lead (II) adsorbed by JR, C_e (mg/l) is the concentration of lead (II) ions adsorbed at equilibrium. K_L (L/mg) is a constant [49]. A straight line obtained from a plot of $\frac{C_e}{q_e}$ against C_e is used to obtain q_m , b and K_L from slope and intercept. The linearized form of Freundlich isotherm equation is given by the equation:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad [7]$$

Where K_F is Freundlich adsorption capacity constant and n is adsorption intensity constant. A linear graph obtained by plotting $\ln q_e$ against $\ln C_e$ is used to determine K_F and n from intercept and slope respectively [23]. Langmuir and Freundlich parameters are presented by table 1:

Table 1: Isotherm parameters for adsorption of lead (II) ions using JR adsorbent

Langmuir isotherm			Freundlich isotherm		
q_{max} (mg/g)	K_L (L/mg)	R^2	K_f (mg/g)	n	R^2
-	-	0.3296	4.6716	1.8936	0.9047

The adsorption of lead (II) ions onto JR adsorbent gave correlation coefficient (R^2) value of 0.9047 showing that the experimental data fitted well in Freundlich isotherm model. The model describes a multi-site physical sorption due to weak Van der waal forces [50, 51, 52]. The value of n is >1 and this shows that adsorption of lead (II) onto JR adsorbent is favourable at even high concentrations [51, 53]. The results compare with those reported by in their study on removal of lead (II) ions onto attapulgit/poly(acrylic acid) nanocomposite microgels [53].

ADSORPTION KINETIC STUDIES

The time data obtained for the adsorption of lead (II) ions onto JR adsorbent were analyzed by pseudo-first-order and pseudo-second-order kinetic models. Linear pseudo-first-order equation is expressed as:

$$\log(q_e - q_t) = \log q_e - \left(\frac{k_1}{2.303}\right)t \quad [8]$$

Where k_1 (mg/g/min) is the pseudo-first-order rate constant [49]. Linear pseudo-second-order equation is expressed as:

$$\frac{t}{q_t} = \frac{t}{q_e} + \frac{1}{k_2 q_e^2} \quad [9]$$

Where k_2 (mg/g/min) is the pseudo-second-order rate constant [54]. The kinetic parameters and constants are tabulated in table 2:

Table 2: Kinetic parameters for adsorption of lead (II) ions using JR adsorbent

Pseudo-first-order				Pseudo-second-order			
q_{exp} (mg/g)	q_{cal} (mg/g)	k_1 (mg/g/min)	R^2	q_{cal} (mg/g)	k_2 (mg/g/min)	R^2	
6.24	1.9762	4.4×10^{-2}	0.6549	6.3857	5.74×10^{-2}	0.9968	

The kinetic parameters for adsorption of lead (II) ions onto JR adsorbent is given by table 2. The regression analysis (R^2) of the experimental time data using pseudo-first-order was lower (0.6549) that that of pseudo-second order (0.9968). this showed that the adsorption process could not be explained by this model. adsorption of lead (II) ions fitted well pseudo-second-order kinetic model with $R^2 > 0.99$. This confirmed the closeness of q_{exp} and q_{cal} . The model assume that chemisorption is the rate determining step in the adsorption of lead (II) ions onto JR adsorbent [15, 55, 56]. The results are in agreement with those reported by [57, 58, 59].

ADSORPTION THERMODYNAMIC STUDIES

Thermodynamic parameters play a crucial role in describing adsorption behavior of metal ions in a temperature controlled system [60]. The data obtained for

lead (II) ions adsorption as a function of temperature for JR adsorbent was analyzed using Van't Hoff equation(10,11 & 12) [61] respectively.

$$K_C = \frac{C_{Ae}}{C_e} \quad [10]$$

$$\Delta G^\circ = -RT \ln K_C \quad [11]$$

$$\log K_C = \frac{\Delta S^\circ}{2.303 R} - \frac{\Delta H^\circ}{2.303 RT} \quad [12]$$

Where K_C is the distribution thermodynamic constant; C_e is the concentration of lead (II) ions at equilibrium (mg L⁻¹); C_{Ae} is the solid-phase adsorption capacity at equilibrium (mg g⁻¹); ΔG° , ΔS° and ΔH° are changes in standard Gibb's free energy (KJ/mol), standard Entropy change (KJ/mol) and standard enthalpy (J/mol/K) change respectively, R is gas constant (8.314 J/mol/K) and T is temperature (K). ΔG° values were calculated using equation 11. A plot of $\log K_C$ against $1/T$ yield a straight line with a slope and intercept being used to determine ΔH° and ΔS° respectively. The thermodynamic parameters are presented by the table 3:

Table 3: Thermodynamic parameters for adsorption of lead (II) ions using JR adsorbent

ΔG° (kJ mol ⁻¹) at different temperatures (K)								
$\Delta H^\circ / R$	$\Delta S^\circ / R$	298 K	308 K	318 K	328 K	338 K	ΔH° kJ /mol	ΔS° J/mol /K
418.52	-1.13	-0.82	-0.51	-0.34	-0.35	-0.37	-3.48	-9.38

The results in table 3 showed that Gibb's free energy (ΔG°) values were all negative in all temperatures. The results revealed that adsorption of lead (II) ions onto JR adsorbent was spontaneous and feasible [62, 63, 64]. Also, high negative ΔG° values at lower temperatures showed that adsorption of lead (II) ions was more favoured at lower temperatures [62]. Negative value of ΔH° showed that adsorption of lead (II) ions onto JR adsorbent as exothermic in nature [65,66,67]. ΔS° value was negative and this showed a decreased randomness of JR-lead (II) solution interface during the adsorption process [65]. The results are in agreement with those reported by [44,67,68] in their study on the removal of lead (II) ions using cashew nut shell adsorbent.

IV. CONCLUSION

In this study, it is evident that JR adsorbent had important functional groups at 3417.75 cm⁻¹ (-OH), 1644.18 cm⁻¹ (-C=O) and 1035.85 cm⁻¹ (-C-O-C-) which successfully adsorbed lead (II) ions. Adsorption efficiency was examined at different experimental parameters (pH, contact time, dosage, temperature and initial concentration) and they greatly influenced the adsorption process. The adsorption capacity of JR adsorbent on lead (II) ions removal as a function of initial concentration was analyzed by Langmuir and Freundlich isotherm models and that lead

(II) ions adsorption was described well by Freundlich model with adsorption capacity of 4.6716 mg/g. Kinetic time data on adsorption of lead (II) ions followed pseudo-second-order with $R^2 > 0.99$. Thermodynamic parameters showed that the adsorption process was spontaneous and exothermic.

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