EFFECT OF VARIOUS PARAMETERS ON BIOSORPTION OF LEAD (II) BY RAGI HUSK POWDER

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Abstract
The objective of the present investigation is the removal of lead (II) from waste water using Ragi husk powder as adsorbent. All the samples are analyzed by the atomic absorption spectrophotometer. Various parameters like agitation time, adsorbent dosage, adsorbent size, pH, concentration and temperature are studied. The equilibrium agitation time is achieved at 50 minute. The maximum percentage removal of lead (II) is achieved at pH=9. The adsorption kinetic studies follow second order. The adsorption isotherm follows the Langmuir followed by Freundlich. The maximum metal uptake is 24.39 mg/g. The present used adsorbent is available naturally and abundantly in our environment. And also biosorption is one of the low cost processes for the removal of lead (II) from waste water using Ragi husk powder.
Keywords: Lead, Adsorption, wastewater, Adsorption Isotherms and Adsorption Kinetics

1. Introduction

Rapid industrialization and growing population are the major cause of inclusion of heavy metals into the nearby water bodies all over the world. Heavy metal contamination of water bodies has becoming serious threat on humans, aquatic system and animals, because of their toxic nature event at lower concentrations also damages nerves, liver, kidney and bone. Heavy metals discharged by various industries are the major pollutants in marine, ground, industrial and also some extent in treated waste waters. Among the heavy metals, lead is one considered as major pollutant found in industrial wastewater and it poses severe threat to the environment due to its toxic nature to aquatic and terrestrial bodies [1].

A conventional method used for removal of heavy metals includes ion exchange, membrane separation, chemical precipitation, solvent extraction, and adsorption [2]. While several merits exist for these processes, primary limitations of these techniques are that they are either expensive or being ineffective at lower concentrations. An alternative process to be considered for the removal of Pb (II) from wastewater streams is the bio-sorption which has been gained increased credibility during last two decades and also recognized as a low cost process due to low cost of naturally available agricultural waste materials [3]. Various investigators have used the various adsorbents like a natural zeolite [4], CalotropisProcera roots [5], Dried leaves of water hyacinth [6] etc for the removal of lead from waste water.

The objective of present study is the use of ragi husk powder as an effective and inexpensive material for the removal of lead from waste water in a batch mode. Adsorption isotherms like Langmuir isotherm, Freundlich isotherm, Tempkin isotherms and adsorption kinetics like first order, second order and intra particle mechanism were studied.

2. Materials and methods

2.1 Preparation of synthetic lead solution

The lead stock solution has been prepared from Lead Nitrate, $Pb(No_3)_2$. 1.6144 grams of 99% $Pb(No_3)_2$ has been dissolved in distilled water of 1.0 L volumetric flask up to the mark to obtain 1000 ppm (mg/L) of lead stock solution.

Synthetic samples of different concentrations of lead have been prepared from this stock solution by appropriate dilution. Similarlysolution with different metal concentrations such as (20,40,60,80 and 100 mg/L) has been prepared.

2.2 Preparation of Ragi husk powder

Ragi husk powder was prepared and used as adsorbent powder fromRagi husk which was collected from vizianagaram, Andhra Pradesh, India; materials were washed and dried. After drying, size reduction was carried out using roll crusher. The material obtained through various size reduction devices was screened through BSS meshes. Finally the products obtained were stored in storage device for further use. All the materials were used as such and no pretreatment was given to the materials.

2.3 Batch experimental procedure

Batch mode adsorption studies for individual metal ions were carried out to investigate the effect of different parameters such as initial metal ion concentration, adsorbent dosage, adsorbent size, agitation time, temperature and pH. The range of different parameters studied in the present work has been given in Table 1. Solution having adsorbate and adsorbent was taken in 250 mL capacity conical flasks and agitated at 180 rpm in a mechanical shaker at known time intervals as shown in Table 1. The adsorbate was decanted and separated from the adsorbent by filtration technique.

Final residual metal concentration after adsorption was measured by Atomic absorption Spectrophotometer (PinAAcle, 500, Perkin Elmer Pvt Ltd). To estimate the percentage removal of lead from waste water, the following equation was used.

$$\text{Removal of lead} = \frac{Initial - Final\ metal\ ion\ concentration}{Initial\ metal\ ion\ concentration} \times 100 \quad (1)$$

Table 1. Various parameters and its ranges investigated in present experimentation

Parameter	Range of parameters
Agitation time (t), min	5, 10, 15, 20, 25, 30, 40, 50, 60, 90 and 120
Adsorbent dosage(w), g	0.05, 0.1, 0.15, 0.2, 0.25, 0.3, 0.35, 0.4, 0.45, 0.5 and 0.6.
Initial chromium (VI)concentration (C_0), mg/L	20, 40, 60, 80 and 100
pH	3, 4, 5, 6, 7, 8, 9 and 10
Temperature, K	273, 283, 293, 303.313, 323
Adsorbent particle size, µm	63, 89, 125

3. Results and discussion

3.1 Effect of contact time at various mixed adsorbent dosages

The agitation time profile for adsorption of Pb (II) metal for solution of 20 mg/L for different adsorbent dosages is depicted in Figure 1. The data shows that the agitation time at equilibrium is achieved at 50 minute at optimum pH value of 9 for the percentage removal of lead. After equilibrium time, no further change in percentage removal of lead metal is noticed. Figure shows that percentage removal of lead increases with increase in adsorbent dosage.

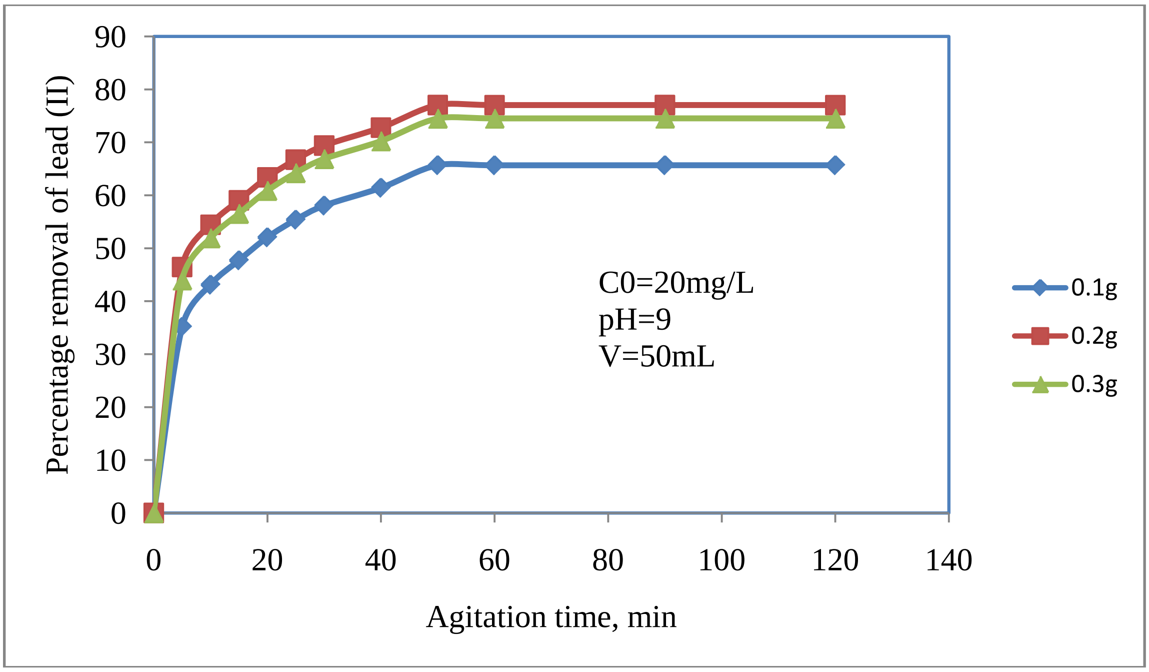

Figure 1.Effect of agitation time on adsorption of lead by the adsorbent.

3.2 Effect of pH

The literature reveals that pH is the most important physical factor influencing the process of adsorption, the degree of ionization and the species of the adsorbate. Figure 2 depicts the effect of pH on adsorption of lead at different initial concentrations. Three was increase in adsorption of lead with increase in pH values from 3 to 9, after words it decreases when pH is greater than 9. The maximum percentage removal of lead was noticed at pH 9.

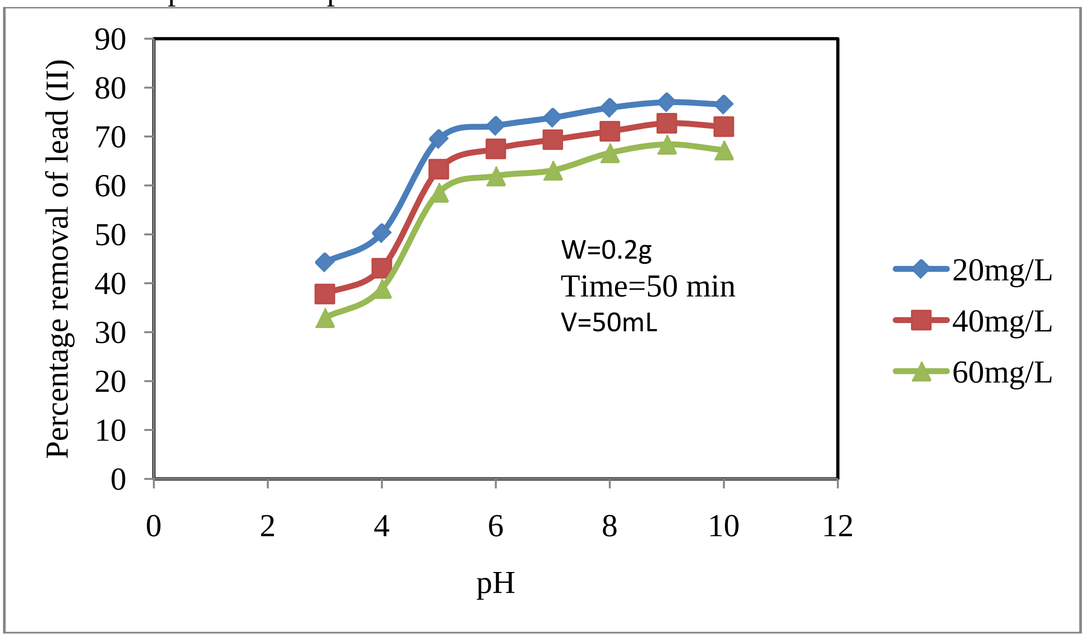

Figure 2. Effect of pH on adsorption of lead at different initial concentrations

3.3 Effect of adsorbent dosage and size in aqueous solution

The variations in percentage removal of lead (II) and also metal uptake with the adsorbent dosage is depicted in Figure 3. The percentage removal of lead (II) increases from 47.83% to 77.01% and also metal uptake decreases from 9.566 to 1.183 mg/g with an increase in the adsorbent dosage from 0.05 g to 0.2 g, Afterwardsfurther increase in adsorbent dosage from 0.2 g to 0.6 g in 50mL solution, it result in decreases of percentage removal of lead. This may be due to unavailability of binding sites and also due to the blockage of binding site with the excess adsorbent dosage. The percentage removal of lead(II) from waste water with adsorbent size is reported in Table 2. The percentage removal of lead (II) increases from 75.01 (3.751 mg/g) to 77.01% (3.851 mg/g) with decrease in the adsorbent size from 125 to 63 µm.The maximum adsorption is achieved at 89 µm.

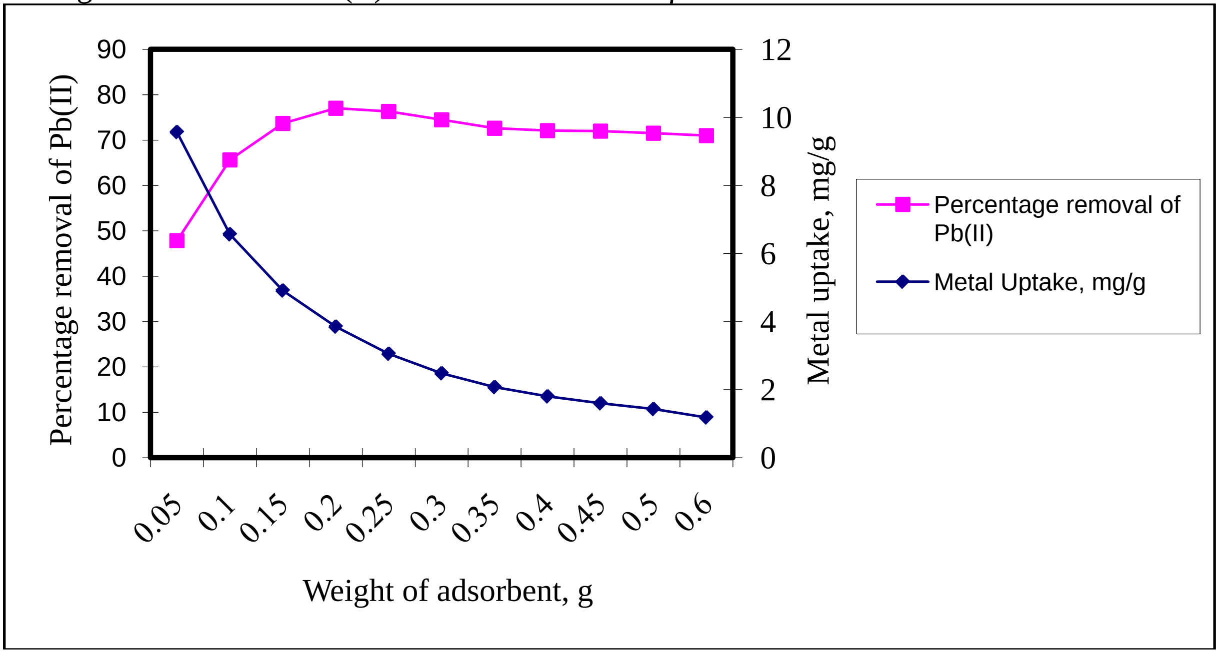

Figure 3. Effect of adsorbent dosage on percentage removal of lead and metal uptake

Table 2. Effect of adsorbent particle size, C_0=20 mg/L; Temp=303 K; V=50mL; t=50min; pH=9.

S.No.	Adsorbent average particle size, µm	Final concentration, mg/L	Percentage removal of Cr(IV)	Metal uptake (mg/g)
1	63	4.998	75.01	3.751
2	89	4.598	77.01	3.851
3	125	4.786	76.07	3.804

3.4 Effect of Temperature

The effect of temperature on adsorption of lead at different initial concentrations is depicted in Figure 4. The maximum percentage removal of lead (II) for various initial concentrations is reported at temperature 323 K. It is confirmed that adsorption of lead (II) increases with increase in temperature values for all concentrations; it reveals that the present adsorption process is endothermic in nature. Further, low initial metal ion concentrations of the solution shows favorable adsorption in comparison with higher metal ion concentration solutions. The percentage removal of lead is increased from 56.32% to 80.02% for a temperature range of 283-323 K. Maximum adsorption of 80.02% was observed at 323 K. This increase in binding might be due to increase in surface activity and increased kinetic energy of the lead (II) metal ions.

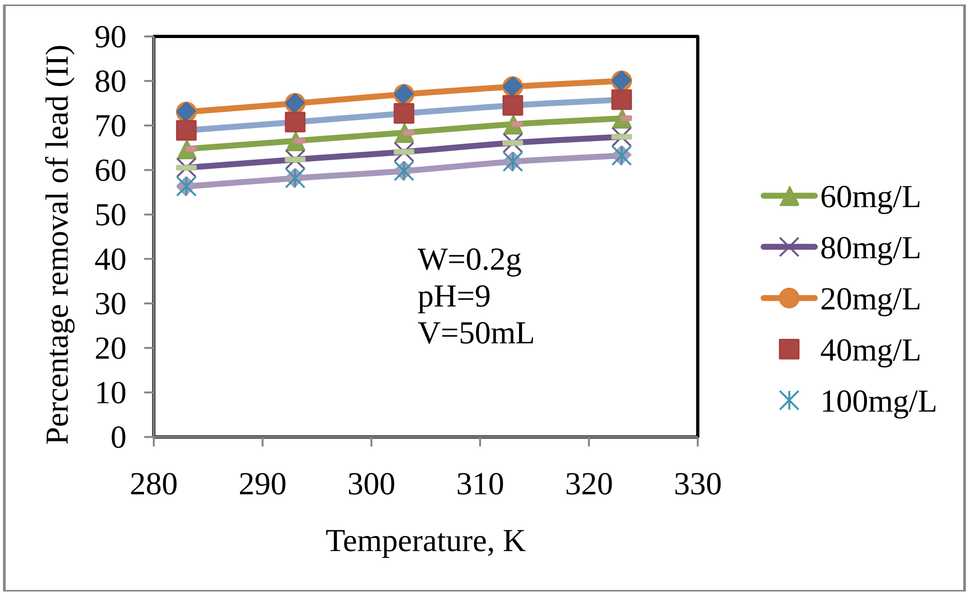

Figure 4.Effect of temperature on percentage removal of lead.

3.5 Adsorption Isotherms

An applicability of Langmuir, Freundlich, Tempkin isotherms for Ragi husk powder was tested to characterize the interaction of the metal ions with the Ragi husk powder. This provides a relationship between the concentration of metal ions in the solution and the amount of metal ions adsorbed to the solid phase when the two phases are at equilibrium.

3.5.1 Langmuir model

The applicability of Langmuir isotherm [7] was tested to estimate the maximum metal uptake which occurs on a homogeneous surface by monolayer sorption without interaction between adsorbed molecules. The model of this is presented as

$$q_e = \frac{q_{\max} b C_e}{1 + b C_e} \qquad (2)$$

q_e is the metal uptake (mg/g), C_e is the equilibrium metal ion concentration (mg/L). q_{max} is the maximum metal uptake, and b is the ratio of sorption rates. The linear form of Langmuir isotherm is represented as follows

$$\frac{C_e}{q_e} = \frac{C_e}{q_{\max}} + \frac{1}{q_{\max} b} \qquad (3)$$

Figure 5 depicts the linearity for Langmuir isotherm for the adsorption of lead on Ragi husk adsorbent powder. Their equations along with the q_{max} and b calculated from the slope and the intercept are depicted in Table 3. It is noticed that there was strong binding of lead onto the surface of Ragi husk powder. The separation factor, R_L determined from Table 4 at Temperature 303 K and pH 9 is in between 0.2012-0.5574 for various concentrations shows favorable adsorption ($0<R_L<1$).

Table 3. Parameters of the Langmuir isotherms for removal of lead (II)

S.No.	Temperature, K	Model Equations	q_m	b	R_L	R^2
1	283	$\frac{C_e}{q_e} = 0.042C_e + 1.269$	23.81	0.0331	<1	0.999
2	293	$\frac{C_e}{q_e} = 0.041C_e + 1.152$	24.39	0.0355	<1	0.998
3	303	$\frac{C_e}{q_e} = 0.041C_e + 1.031$	24.39	0.0397	<1	0.998

4	313	$\frac{C_e}{q_e} = 0.04C_e + 0.940$	25.00	0.0426	<1	0.997
5	323	$\frac{C_e}{q_e} = 0.04C_e + 0.872$	25.00	0.0459	<1	0.997

Table 4. $R_L = \frac{1}{1+bc_i}$ values at pH=9; T=303K.

Initial concentration, C_i (mg/L)	**R_L**
20	0.5574
40	0.3864
60	0.2957
80	0.2395
100	0.2012

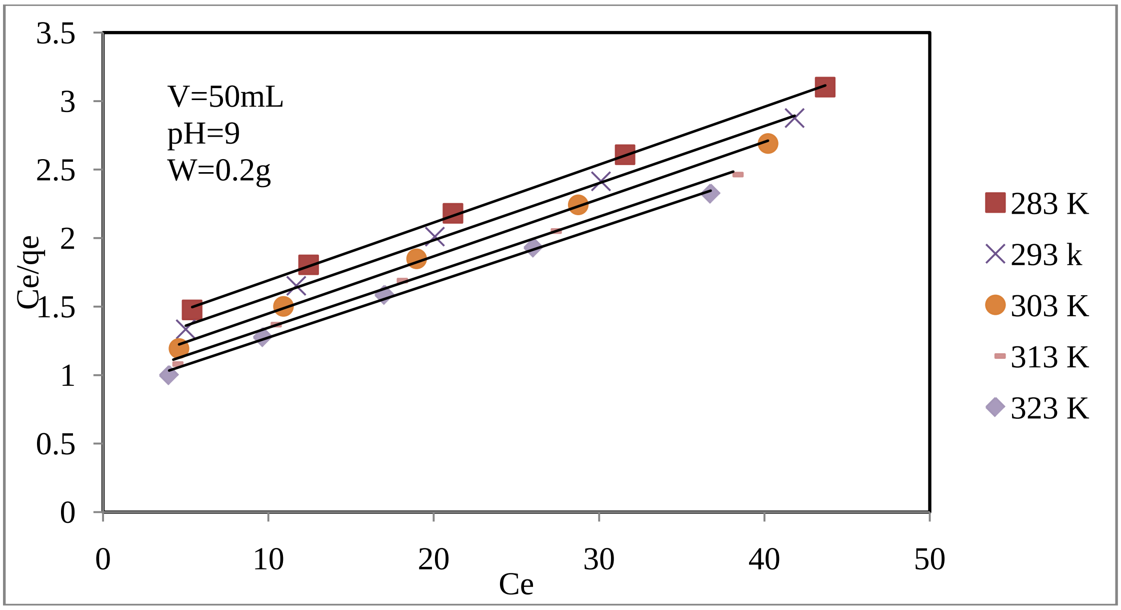

Figure 5.The Langmuir isotherm for adsorption of lead.

3.5.2 Freundlich model

The applicability of Freundlich isotherm [8] was tested for nonlinear multilayer adsorption model with heterogeneous energetic distribution active sites and reversible adsorption, followed by interaction between adsorbed molecules. The general of this model is represented by:

$$q_e = K_f C_e^{\frac{1}{n}} \qquad (4)$$

K_f and n are the adsorption capacity and adsorption intensity respectively. q_e is the equilibrium metal uptake and C_e is concentration of metal at equilibrium. The equation representing the Freundlich model is presented linearly as.

$$\log q_e = \log K_f + \frac{1}{n}\log C_e \qquad (5)$$

The values of $K_f$and n obtained from intercept and slope of a plot of log q_e versus log C_e is shown in Table 5. Figure 6 depicts good linearity with high regression coefficient for Freundlich isotherm for the adsorption of lead on Ragi husk powder and Linearity of the relationship reveals strong binding of lead on the adsorbent. As the slope of isotherm (n) is greater than 1, it favors for the adsorption of lead metal ions on adsorbent.

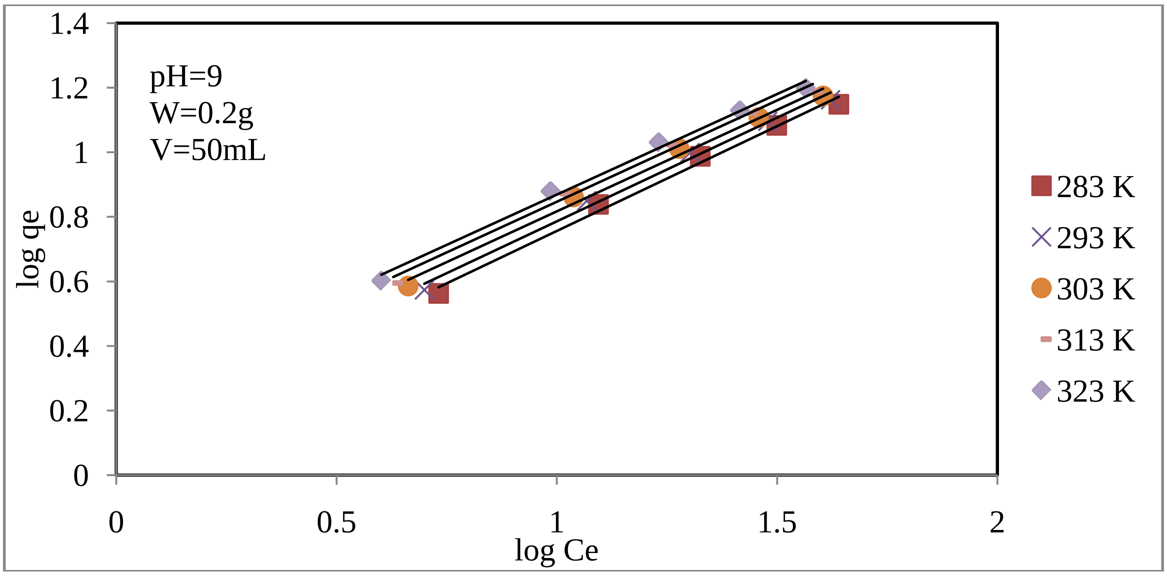

Figure 6. The Freundlich isotherm for adsorption of lead

Table 5. Parameters of the Freundlich isotherms for removal of lead(II)

S.No.	Temperature, K	Equations	K_f	n	R^2
1	283	$\log q_e = 0.648 \log C_e + 0.107$	1.2794	1.5432	0.992
2	293	$\log q_e = 0.642 \log C_e + 0.143$	1.3899	1.5576	0.992
3	303	$\log q_e = 0.628 \log C_e + 0.188$	1.5417	1.5923	0.992
4	313	$\log q_e = 0.627 \log C_e + 0.219$	1.6557	1.5948	0.992
5	323	$\log q_e = 0.622 \log C_e + 0.246$	1.7619	1.6077	0.993

3.5.3 Tempkin model

The applicability of Tempkin isotherm model [9] was tested for direct/indirect the adsorbent-adsorbate interactions. Both the nonlinear and linear form of Tempkin equations are given below.

$$q_e = \frac{RT}{b_T} \ln(A_T C_e) \quad (6)$$

$$q_e = B_T \ln A_T + B_T \ln C_e \quad (7)$$

B_T= (RT/b_T). T is the temperature (K) and *R* is the universal ideal gas constant. Constant b_T represents the heat of adsorption. A_T is the equilibrium binding constant at the maximum binding energy. A plot of q_e versus ln C_e at a fixed temperature will give Tempkin isotherm constants, A_T and b_T.

Figure 7 depicts the linearity for Tempkin isotherm for the adsorption of lead on Ragi husk powder. Their equations with regression coefficients are given in Table 6 and Linearity relationship of figure reveals that strong binding of lead on the adsorbent.

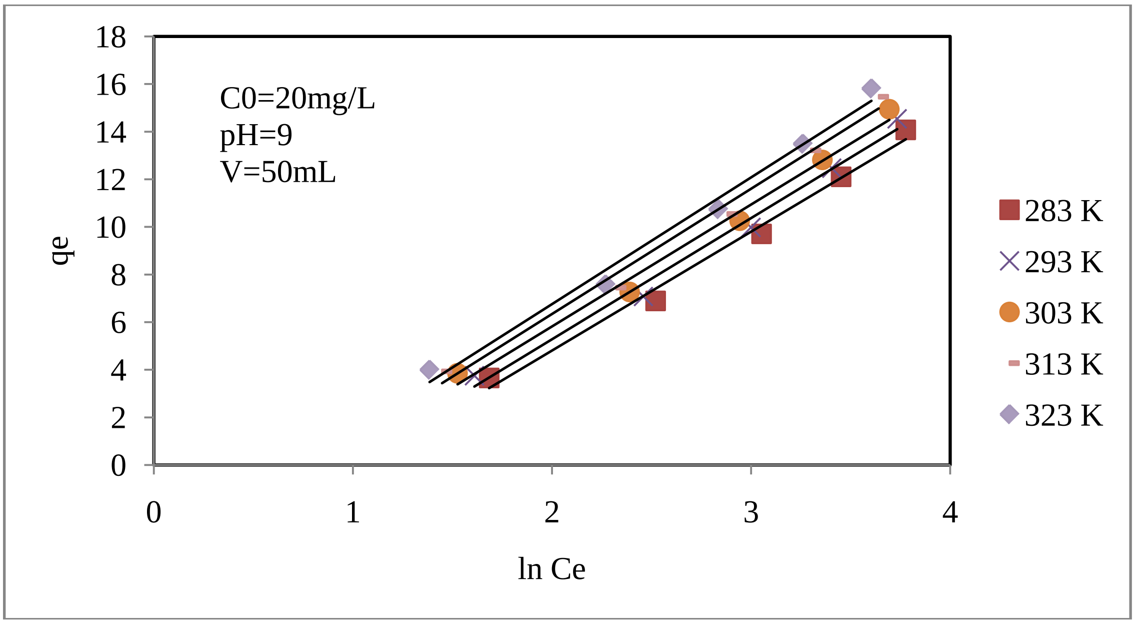

Figure 7.The Tempkin isotherm for adsorption of lead.

Table 6. Parameters of the Tempkin isotherm for removal of lead (II)

S.No.	Temperature, K	Equations	b_T	A_T	R^2
1	283	$q_e = 4.995 \ln C_e - 5.186$	471.043	0.3540	0.989
2	293	$q_e = 5.09 \ln C_e - 4.899$	478.397	0.3821	0.988
3	303	$q_e = 5.122 \ln C_e - 4.423$	491.827	0.4217	0.988
4	313	$q_e = 5.263 \ln C_e - 4.183$	494.449	0.4517	0.987
5	323	$q_e = 5.324 \ln C_e - 3.892$	504.399	0.4814	0.987

3.6 Studies on kinetics of adsorption

The adsorption kinetic models are used to find out the metal uptake,reaction rate along with reaction pathways and time to reach equilibrium. Two adsorption kinetic models such as pseudo first order and pseudo second order kinetic models at various parameters have been tested. Based on regression coefficient (R^2 value), the closeness of experimental data to the model predicted values are decided. A relatively high R^2 value (close or equal to one) is preferred for better adsorption of lead on to theRagi husk powder.

3.6.1 Pseudo-first order kinetic model

The pseudo first order kinetic model is presented as

$$\frac{dq}{dt} = k_1 (q_e - q_t) \tag{8}$$

q_e and q_t are the metal uptake at equilibrium and time t, respectively (mg/g) and k_1 is the rate constant of pseudo first order adsorption (min^{-1}). Equation (8) is arranged as follows

$$\log(q_e - q_t) = \log q_e - \left(\frac{k_1}{2.303}\right)t \tag{9}$$

The plot of $\log(q_e\text{-}q_t)$ vs t is linear from which k_1 can be determined from the slope. Figure 8 depicts the first order kinetics for adsorption of lead on Ragi husk powder. The model equations with regression coefficients are shown in Table 7. From the graph, the q_e is estimated for different process parameters and the estimated value is checked with the experimental value.

From Table 7, it is seen that q_{ecal} and q_{eexp} are not the same. Therefore, first order kinetics may not represent the first order kinetic model. The above model is not fit for lead removal on Ragi husk powder.

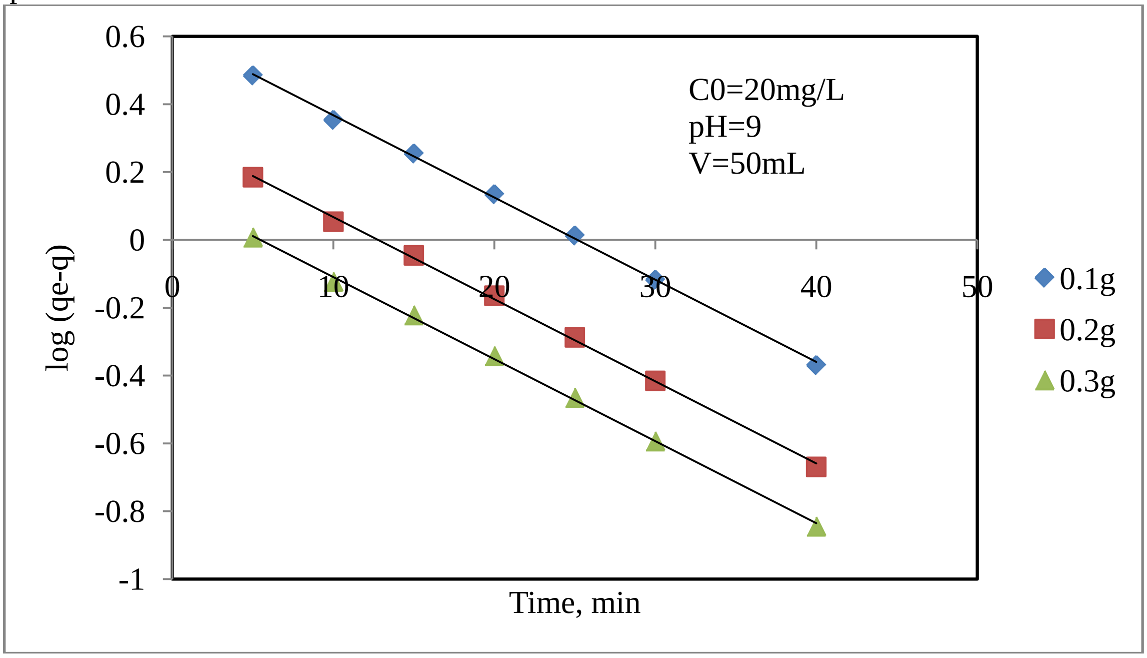

Figure 8. Testing the first order kinetics for adsorption of lead

Table 7. Kinetic data of Lagergren pseudo first order coefficients for removal of lead (II)

Kinetics	**Parameters** **Initial concentration= 20mg/L** **pH=9, Temperature=303K** **adsorbent dosage, g**	**Model equations**	**q_{ecal}**	**q_{eexp}**	**R^2**	**Rate constants, K_1, min^{-1}**
1^{st} order	0.1	$\log(q_e - q) = -0.024t - 0.609$	4.064	6.569	0.999	0.0553
	0.2	$\log(q_e - q) = -0.024t + 0.309$	2.037	3.853	0.998	0.0553
	0.3	$\log(q_e - q) = -0.024t + 0.132$	1.3552	2.484	0.999	0.0553

3.6.2 Pseudo–second order kinetic model

The pseudo-second-order kinetic model rate equation is expressed as

$$\frac{dq}{dt} = k_2(q_e - q_t)^2 \qquad (10)$$

k_2 is the rate constant (g mg^{-1}min^{-1}). The linear form of Equation (10) after integration is given by

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^{\ 2}} + \frac{t}{q_e} \qquad (11)$$

k_2 is obtained from the intercept of the plot of t/q_t vs t. Figure 9 depicts the second order kinetic model for adsorption of lead on Ragi husk powder. The model equations with correlation coefficients are shown in Table 8, it is seen that q_{ecal} and q_{eexp} are almost the same. Therefore, the second order kinetic model is fit for lead removal by Ragi husk powder.

Table 8. Kinetic data of Lagergren pseudo second order coefficients for removal of lead (II)

Kinetics	**Parameters** **Initial concentration=20 mg/L.** **pH=2, Temperature=303K** **Adsorbent dosage, g**	**Model equations**	**q_{ecal}**	**q_{eexp}**	**R^2**	**Rate constants, K_1, g mg^{-1} min^{-1}**
2nd order	0.1	$\frac{t}{q} = 0.136t + 0.987$	7.353	6.569	0.994	0.0187
	0.2	$\frac{t}{q} = 0.239t + 1.336$	4.184	3.853	0.996	0.0427
	0.3	$\frac{t}{q} = 0.369t + 2.168$	2.710	2.484	0.995	0.0628

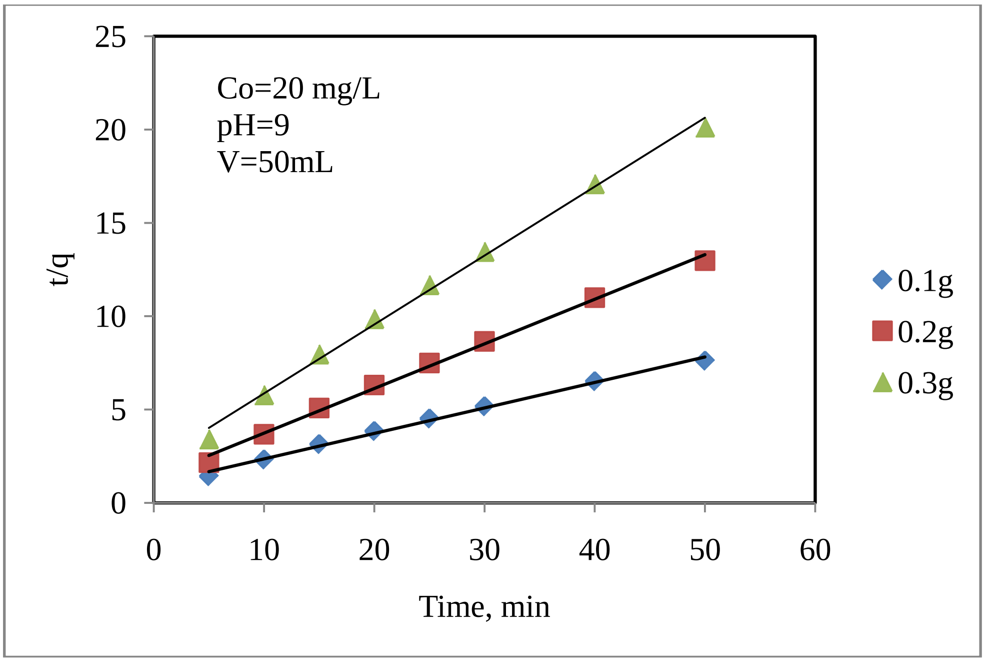

Figure 9.Testing the second order kinetics for adsorption of lead.

3.6.3 Intra-particle diffusion model

The combination of four consecutive steps [10] are used for lead removal from waste water on adsorbent; diffusion in the bulk solution, then diffusion across the thin film surrounding the adsorbent particles, followed by intra-particle diffusion and adsorption within the particles. According to Weber and Moris [11] if the rate limiting step is the intra-particle diffusion, then amount of adsorbed at any time is directly proportional to the square root of contact time t and may pass through the origin which is shown mathematically in Equation (12)

$$q_t = K_{diff} t^{0.5} + C \qquad (12)$$

q_t, t and K_{diff} are the metal uptake, contact time and the intra-particle diffusion coefficient respectively. A plot of q_t against $t^{0.5}$ will give a straight line with a positive intercept for intra particle diffusion. The value of K_{diff} will be calculated from slope. The higher value of K_{diff} indicates the enhancement of the rate of adsorption.

Figure 10 depicts the linearity for intra particle diffusion model based on the regression coefficients. The R^2 values (refer to Table 9) are close to unity, conforming that the rate-limiting step is actually the intra-particle diffusion process. The values of K_{diff} calculated from slope provide information about thickness of the boundary layer, i.e. the resistance to the external mass transfer. The larger slope indicates higher external resistance.

Table 9. Parameters of intra particle diffusion model for removal of lead (II).

S.No.	**Adsorbent dosage, g**	**Model equations**	**R^2**	**K_{diff}**	**Constant, C**
1.	0.1	$q_t = 0.619t^{0.5} + 2.322$	0.985	0.619	2.322
2.	0.2	$q_t = 0.310t^{0.5} + 1.725$	0.985	0.310	1.725
3.	0.3	$q_t = 0.206t^{0.5} + 1.068$	0.985	0.206	1.068

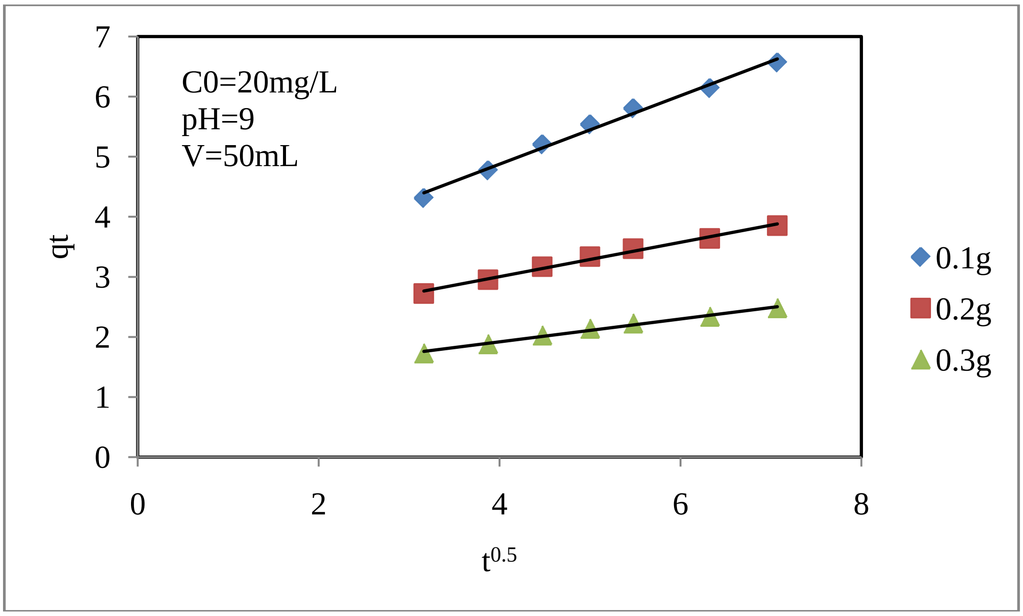

Figure 10. Testing the intra particle diffusion model for adsorption of lead

3.7. Thermodynamics of adsorption

Adsorption is the temperature dependent and also associated with three thermodynamic parameters like change in enthalpy (ΔH), change in entropy (ΔS) and change in Gibb's free energy (ΔG) [12] .

The Van't Hoff equation is given by

$$K_D = \frac{C_{Ae}}{C_e} \qquad (13)$$

$$\Delta G = -RT \ln K_D \qquad (14)$$

K_D is the equilibrium constant and C_{Ae} and C_e(both in mg/L) are the equilibrium concentrations for solute on the adsorbent and in the solution, respectively. The K_D values calculated from equation (13) are used in equation (14) to find out the ΔG, ΔH and ΔS. Then K_D is expressed below as function of ΔH (KJ/mol) and ΔS (KJ/mol K) and absolute temperature.

$$\ln K_D = -\frac{\Delta H}{RT} + \frac{\Delta S}{R} \quad (15)$$

The values of ΔH and ΔS are calculated for the slope and intercept respectively by plotting ln K_D vs (1/T).

The experimental data was plotted in Figure 11 which indicates the linearity for Van't Hoff equation. Their equations with correlation coefficients are shown in Table 10. From values of ΔH, ΔS and ΔG, the thermodynamic parameters such as change in Gibbs free energy (ΔG), change in enthalpy (ΔH), and change in entropy ΔS for the adsorption of lead on Ragi husk powder indicates the feasibility, spontaneity, irreversibility and endothermic nature.

Table 10. Thermodynamic parameters for removal of lead (II).

Initial Conc, mg/L	**Model Equations**	**ΔH, KJ/mole**	**ΔS, J/mole K**	**ΔG, KJ/mole**				
				283	**293**	**303**	**313**	**323**
20	$\ln K_D = -0.906(\frac{1}{T}) + 4.19$ R^2=0.998	7.533	34.91	-2.346	-2.674	-3.045	-3.404	-3.726
40	$\ln K_D = -0.805(\frac{1}{T}) + 3.64$ R^2=0.998	6.693	30.263	-1.873	-2.154	-2.468	-2.792	-3.067
60	$\ln K_D = -0.741(\frac{1}{T}) + 3.22$ R^2=0.998	6.161	26.804	-1.428	-1.675	-1.944	-2.241	-2.486
80	$\ln K_D = -0.698(\frac{1}{T}) + 2.89$ R^2=0.997	5.803	24.044	-1.006	-1.229	-1.457	-1.736	-1.956
100	$\ln K_D = -0.668(\frac{1}{T}) + 2.61$ R^2=0.996	5.554	21.725	-0.059	-0.081	-0.099	-1.261	-1.458

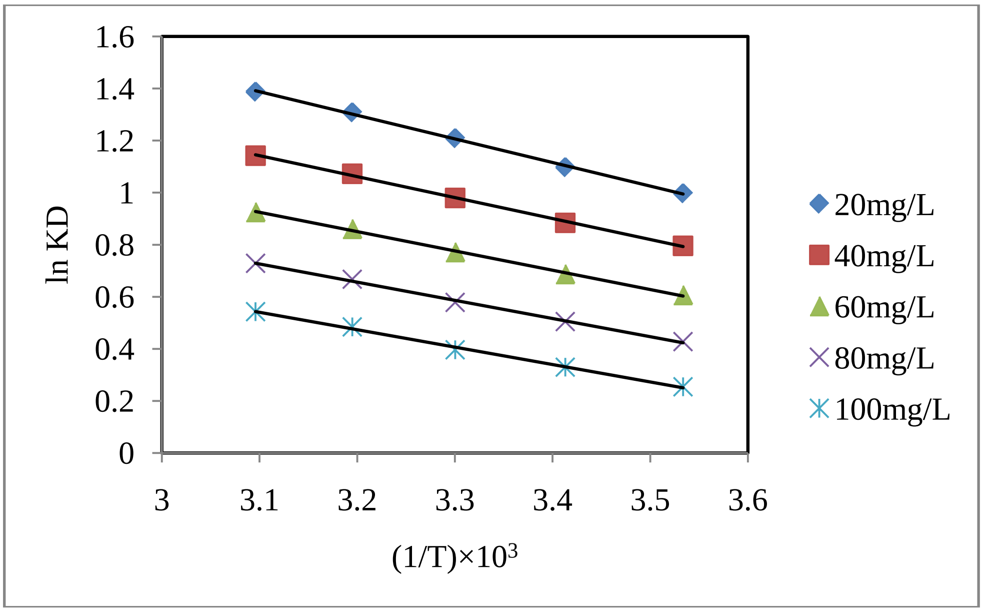

Figure 11.Finding out the Von'thoff equation.

4. CONCLUSIONS

Ragi husk powder has the potential for use as cheap naturally available bio-sorbent for the removal of lead from waste water, up to 77.01% for an initial concentration of lead at 20 mg/L and temperature of 303 K. The Pb (II) adsorption performance by Ragi husk powder is strongly affected by parameters such as contact time, Pb (II) concentration, pH, adsorbent dosage, adsorbent particle size and temperature.

- Percentage adsorption of lead by Ragi husk adsorbent is reached the equilibrium agitation time at 50 minutes for all initial concentration of lead solution.
- With an increase in pH from 3 to 10, the percentage adsorption of Pb (II) is increased upto pH value of 9 after that it decreases. The optimum pH is taken at pH of 9.
- With the increase inthe adsorbent dosage from 0.05 to 0.6 g (for 50 mL of solution of 20 mg/L), the percentage adsorption of lead is increased from 47.83 to 77.01%.
- The percentage adsorption of lead on Ragi husk powder increases with increase in the temperature from 283 to 323 K for all initial concentrations of lead.
- The adsorption isotherms follows the Langmuir, Freundlich models for Pb (II) adsorption onto Ragi husk powder
- The adsorption kinetic model follows the pseudo second-order kinetics
- The thermodynamic parameters such as change in Gibb's free energy (ΔG), change in enthalpy (ΔH) and change in entropy (ΔS) reveals the feasibility, irreversibility, spontaneity and endothermic nature and an increased randomness at the surface of the Ragihusk powder.

The present study has been performed in the batch process as this gives a platform for the designing of continuous flow systems. It is suggested that the use Ragi huskpowder for the removal of lead from waste water is effective and efficient and low cost process.

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