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Performance evaluation of agro-based adsorbents for the removal of cadmium from wastewater

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ABSTRACT

An effort has been made in the present research to evaluate the agro-based adsorbents such as coconut shell, walnut shell, and almond shell for the removal of cadmium from electroplating industrial effluent. Adsorption experiments were conducted to evaluate the performance. The highest removal efficiency for Cd(II) is 83.7% at pH 6.5 for coconut shell-activated carbon. The percentage removal of cadmium increases with the decrease in metal concentration. The extent of removal depends on the metal ion concentration, adsorbent dose, contact time, and particle size. Coconut shell is found to be more effective with respect to removal efficiency. Cadmium adsorption follows second-order rate equation for coconut shell. The isotherm data obtained more closely follow the Freundilch adsorption isotherm for coconut shell, while walnut shell and almond shell follow the Langmuir adsorption isotherm better. The coconut shell has the highest potential to remove cadmium ion from electroplating wastewater.

Keywords: Cadmium; Wastewater; Agro based; Adsorption

1. Introduction

Wastewater pollution due to lethal heavy metals has been a major cause of concern to scientists, engineers, and health policy makers. Many disasters happen because of heavy metal contamination in aquatic environment [1–3]. Public awareness for pollution caused by heavy metals has been worldwide now. These metals have widespread application in industries and enter the environment wherever they are produced, used, or discarded [4–7]. All these metals become seriously toxic as ions or compound being soluble in water and readily absorbable by living organisms [8,9]. Cd(II) may be introduced in aquatic environments through untreated wastewater discharge from various industrial processes such as mining, phosphate fertilizers, pigments, alloy industries, electroplating, cadmium and nickel batteries, and sewage sludge [10–14].

Cadmium is highly toxic and categorized as carcinogenic to human beings by International Agency for Research on Cancer [15]. Cadmium mainly accumulates in the liver and kidney [16]. Other body organs exposed to cadmium are bones, heart, pancreas, testes, and hematopoietic system. These organs are affected following chronic exposure to cadmium, their functions are impaired, and anemia occurs. It can also cause some other chronic and acute disorders like skeletal deformity, pulmonary problems, kidney stone formation, behavior disturbance, and cognitive impairment [16–18]. Itai-Itai disease is well known to be caused by cadmium toxicity. So maximum permissible limit of cadmium in drinking water is 0.003 mg/L [19].

Various adsorbents have been used for the treatment of many types of wastewater, depending on the type of pollutants that exists in water and wastewater. Natural adsorbents

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due to their abundance, low cost, and high removal efficiency have been used by many researchers for the removal of various pollutants from aqueous solutions. Many researchers have studied different adsorbents for the removal of heavy metals from wastewater by use of adsorption process [16–18,20,21]. In Iran, coconut, walnut, and almond shell are available as the sources of low-cost adsorbent or activated carbon which can be effectively used in the industrial wastewater treatment for pollutant [22–24]. This is because these materials are considered as waste products from the country's economy of palm oil production. Therefore, it can help to create a large-scale recycling mechanism where utilization of waste products would be achieved to the most possible extent, thus reducing the impact of pollution on the environment and helping to rationally consume natural resources.

The experimental study evaluates and compares the potentials of agro-based waste materials such as coconut shell-activated carbon (CSAC), walnut shell-activated carbon (WSAC), and almond shell-activated carbon (ASAC) for the removal of cadmium from the electroplating wastewater.

2. Materials and methods

The wastewater samples were brought from the electroplating industry, located at Shah Jamal, 6 km from Aligarh. Various agro-based waste materials such as CSAC, WSAC, and ASAC have been used as absorbents for the removal of cadmium from the electroplating wastewater. The adsorbents used in the present study are widely available in Iran and other counties.

2.1. The preparation of adsorbate CSAC, WSAC, and ASAC

After drying, CSAC, WSAC, and ASAC were grinded and then sieved to obtain an average particle size of 0.3, 0.6, and 1.181 mm, respectively. Adsorbents were washed several times with distilled water in order to remove the lignin, lighter materials, and other impurities [25,26]. The adsorbents were then dipped in 0.1 mol/L NaOH for a duration of 6 h and washed several times with distilled water to remove the color and lignin content and then dried [26]. The adsorbents were again washed separately with double-distilled water and dipped into 0.1N H_2SO_4 for a period of 6 h to remove its alkalinity. The acid-treated adsorbents were washed thoroughly with double-distilled water. Finally, these were dried in an oven at 110°C for 3 h and stored in desiccators.

2.2. Batch adsorption experiments

The adsorption experiments were conducted in a batch system that was composed of some 100-mL Erlenmeyer flasks in a thermostatic shaker (30°C, 1500 rpm). Each flask was filled with 50 mL of solution. The following parameters were used in the series of adsorption experiments: pH from 2.50 to 9 (initial cadmium concentration = 52 mg/L, temperature = 30°C, particle size = 0.3 mm, and agitation speed = 150 rpm), cadmium concentrations from 5 to 52 mg/L for different adsorbents (agitation speed = 150 rpm, temperature = 30°C ± 1°C, contact time = 2.5 h, size of adsorbent = 0.3 mm), particle size 0.3–1.18 mm (initial concentration of Cd = 52 mg/L; pH = 6.5; temperature = 30°C ± 1°C; adsorbents: CSAC, WSAC, and ASAC), contact time from 5 min to 7 h (adsorbents: CSAC, WSAC, and ASAC; temperature 30°C ± 1°C; pH 6.5; adsorbent dose 15 g/L).

2.3. Determination method for cadmium

A digestion solution for tissue was prepared with 6 mL of 65% nitric acid (HNO_3) (Carlo Erba) and 2 mL of 30% peroxide hydrogen (H_2O_2-Carlo Erba) over a 50-min operation cycle at 200°C. After mineralization, the vessels were opened if a temperature <25°C was reached, then the content was decanted in falcon tubes, and ultra-pure water (Merck, Germany) was added to the samples up to 30 mL; for quantification of metals, an ICP-MS Elan-DRC-e (Perkin–Elmer, USA) was used. Cadmium ion concentration was measured by using inductively coupled plasma - optical emission spectrometry [27].

2.4. Equilibrium adsorption modeling

Isotherm models, Langmuir and Freundlich, were applied to determine the relationship between equilibrium capacity (q_e) and equilibrium concentration (C_e). Adsorption kinetic models were used to predict the rate of adsorption and adsorption mechanisms. To describe cadmium adsorption on the CSAC, WSAC, and ASAC adsorbents, four kinetic models (pseudo-first order and pseudo-second order) were used according to Mohammadi et al. and Jafari et al. [28,29].

3. Results and discussion

The experimental data obtained during the adsorption studies show the effects of adsorbent dose and contact time, influence of pH, effects of various initial Cd(II) concentrations, effects of variation of contact time and different particle sizes, and effects of contact time and temperature. Adsorption isotherms are obtained to determine the feasibility of the system. Kinetic studies have been investigated to understand the mechanism of adsorption. Thermodynamic data indicate the feasibility of the process. Column studies have been performed to compare these with results from the batch studies.

3.1. Effect of different adsorbent doses

The adsorbent dose was varied from 0 to 20 mg/L at fixed initial cadmium concentration of 52 mg/L. The adsorbent dose result shows a higher Cadmium removal occurs with corresponding increase in the dose of coconut shell up to a certain level, beyond which the removal of adsorbent remains constant. It is evident that a dose of 12.5 g/L is sufficient to remove 83.7% Cd(II) for 0.3 mm particle size, whereas it removes 54.5% for 1.181 mm particle size. The increase in the removal percentage with simultaneous increases in adsorbent dose and low particle size is due to the increase in the surface area and hence more number of active sites are available for adsorption (Fig. 1).

The observations indicate that an increase in the removal of cadmium occurs with increase in the dose of walnut shell up to a certain level, beyond which the removal of cadmium

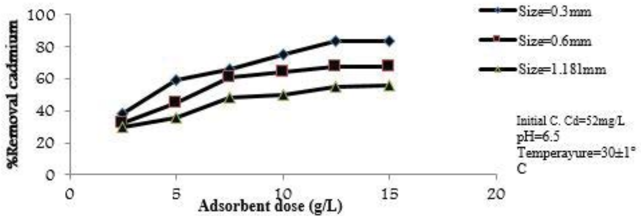

Fig. 1. Variation of CSAC doses with different particle sizes on removal of Cd(II).

remains constant. It is evident that a dose of 15 g/L is sufficient to remove 80.6% Cd(II) for 0.3 mm particle size (Fig. 2).

Fig. 3 indicates that 0.3 mm ASAC uptakes 5–17.5 g/L of absorbent dose gradually and a maximum dose of 17.5 g/L is sufficient to remove 73% cadmium in wastewater having an initial concentration of 52 mg/L.

Similar study conducted by Madhava Rao (2006) on removal of copper and cadmium from the aqueous solutions by activated carbon derived from *Ceiba pentandra* hulls showed that removal of cadmium increases in the concentration of the activated carbon due to the greater availability of the exchangeable sites or surface area at higher concentrations of the adsorbent [30].

3.2. Effect of initial Cd(II) concentration

It has been observed in Fig. 4 that the percentage removal of cadmium decreased with the increase in concentration of cadmium in the wastewater. This is due to an increase in the number of cadmium ion for the fixed amount of adsorbent. The amount of metal ion adsorbed per adsorbent increased with the increase in metal ion concentration. This is due to the full utilization of surface adsorption available.

Cadmium solutions with different initial concentrations in the range of 5–52 mg/L were investigated. Result showed the maximum removal of 83.7%–100% is achieved for the range of concentrations for 0.3 mm particle size. The removal efficiency of cadmium decreased when cadmium concentration is increased. The removal efficiency is recorded at 83.7% at a concentration of 52 mg/L (Fig. 5).

The effects of initial Cd(II) concentration on removal of cadmium by WSAC were studied over the range of cadmium concentration of 5–52 mg/L. The maximum removal of 80.5%–98.6% is achieved for 0.3 mm particle size. The removal efficiency of cadmium decreased when cadmium concentration is increased. Removal efficiency is recorded at 80.6% at a concentration of 52 mg/L (Fig. 6).

The adsorption of Cd decreases from 98% to 72.5% for 0.3 mm particle size when the initial metal concentration was increased from 5 to 52 mg/L at an adsorbent dose of 17.5 g/L for 2.5 h contact time at pH 6.5 (Fig. 7).

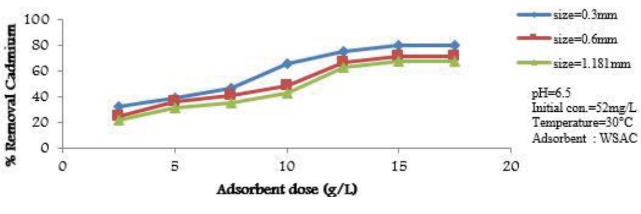

Fig. 2. Variation of WSAC doses with different particle sizes on removal of Cd(II).

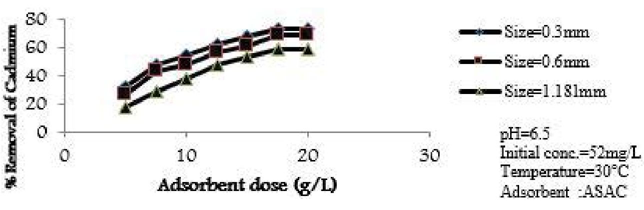

Fig. 3. Variation of ASAC doses with different particle sizes on removal of Cd(II).

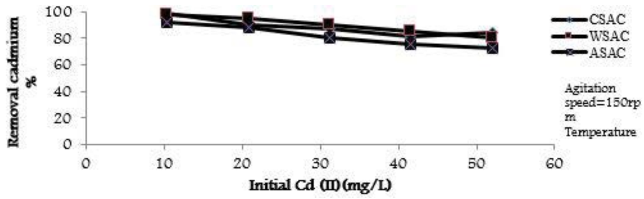

Fig. 4. Removal efficiency with initial Cd(II) concentration by different adsorbents.

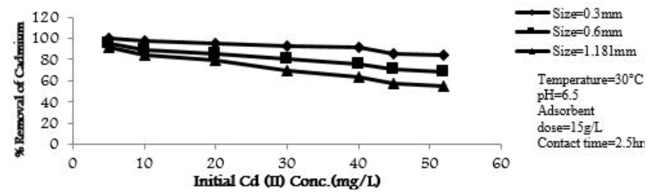

Fig. 5. Variation of initial Cd(II) concentration on removal of cadmium by CSAC.

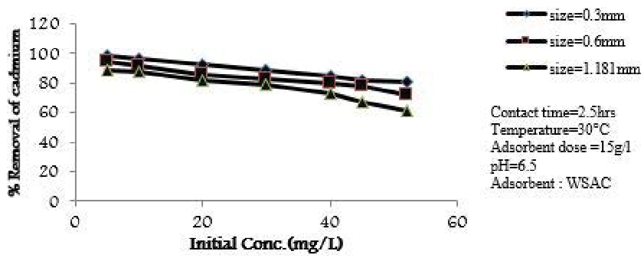

Fig. 6. Variation of initial Cd(II) concentrations on removal of cadmium by WSAC.

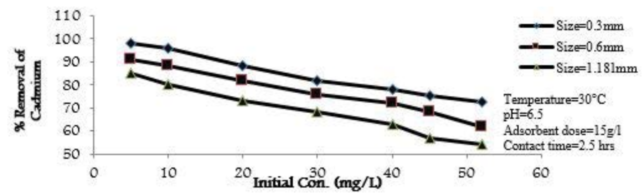

Fig. 7. Variation of initial Cd(II) concentrations on removal of cadmium by almond shell.

3.3. Effect of pH on adsorption of Cd(II)

The effect of pH on the adsorption of Cd(II) for the different adsorbents is shown in Fig. 8. The maximum removal efficiencies for Cd(II) by adsorbents CSAC, WSAC, and ASAC are 83.7% at pH 6.5, 80.6% at pH 7, and 72.5% at pH 6, respectively (Fig. 8).

With increasing pH, the negative charge on the surface of adsorbent increases thereby enhancing the metal adsorption [30]. A similar behavior of increase in metal adsorption with increasing pH was observed [31].

A similar study done by Osasona on activated carbon from spent brewery barley husks for cadmium ion adsorption from aqueous solution showed that the percentage removal of Cd increased from 75% to 99.75% when the pH was raised from 1.5 to 6 [32].

3.4. Effect of contact time

The maximum removal was observed as 83% within the 2.5 h for 0.3 mm particle size. It can be seen from the figure that the cadmium adsorption capacity at equilibrium increases with the decrease of adsorbent sizes. Therefore, increasing particle size increases the time needed to reach equilibrium (Fig. 9).

The adsorption of cadmium increased with increasing contact time and becomes almost constant after 2.5 h for 0.3 mm size particles (Fig. 10).

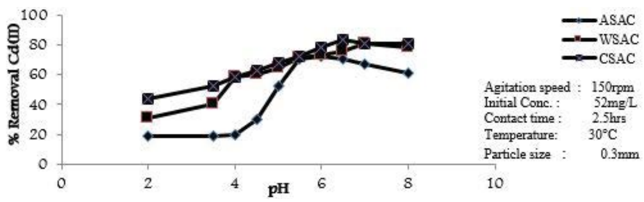

Fig. 8. Variation of pH on removal of cadmium by different adsorbents.

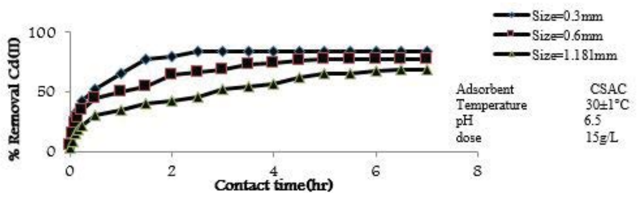

Fig. 9. Variation of contact time and different particle sizes on removal of cadmium by CSAC.

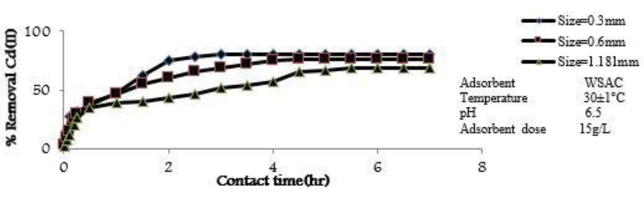

Fig. 10. Variation of contact time and different particle sizes on removal of cadmium by WSAC.

It has been observed in Fig. 11 that adsorption capacity reaches equilibrium of all the adsorbents. The smaller size of the adsorbent attained the higher adsorption level, which may not be discrete to the fact that smaller particles provide large surface areas.

3.5. Adsorption isotherm

Batch experiments were analyzed using the Langmuir and Freundlich models [33,34]. The results show that CSAC had remarkable potential for the removal of Cd(II) ion from wastewater. In view of the values of the linear regression coefficient, Langmuir model fits very well to the sorption data in the studied concentration range. The R_1 value 0.356 lies between 0 and 1 indicating a highly favorable adsorption; results are shown in Table 1. Langmuir adsorption isotherm for almond shell indicated that the Langmuir model fitted very well to the sorption data obtained. The results also indicated that WSAC and ASAC have the highest potential for the removal of Cd(II) ion from wastewater and aqueous solution as indicated by the values of the linear regression coefficients of 0.9416 and 0.9706, respectively.

3.6. Adsorption isotherm study

The value of n in Freundlich equation was found to be 1.198 for CSAC. Since n lies between 1 and 10, this indicates physical adsorption of Cd(II) onto CSAC. The values of regression coefficients (R^2 = 0.94) are regarded as a measure of goodness of fit of the experimental data to the isotherm models. From Table 1, it is evident that both models fit the data reasonably well, but the best was obtained with the Langmuir isotherm model (R^2 = 0.9677). The regression coefficient equals R^2 = 0.4732. The Freundlich model does not fit well to the sorption data in the concentration range studied for WSAC. A similar result was reported by Sigdel et al. (2017). Adsorption of cadmium and benzene onto the three selected sorbents was a better fit to the Langmuir model than the Freundlich model, which demonstrated homogeneous sorption on a uniform binding site of the adsorbent [35].

Also Osasona conducted a study on activated carbon from spent brewery barley husks for cadmium ion adsorption from aqueous solution which revealed that the data were better described by Langmuir model because it presented a higher correlation coefficient and lower error values than Freundlich model [32].

The Freundlich model was applied to the adsorption data for ASAC. The results indicated that ASAC had remarkable potential for the removal of Cd(II) ion from wastewater as indicated by the value of the linear regression coefficient (R^2 = 0.9589) over the concentration range studied.

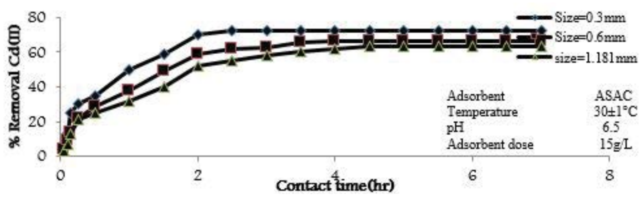

Fig. 11. Variation of contact time and different particle sizes on removal of cadmium by ASAC.

Table 1
Values of Langmuir and Freundlich isotherm constants

Temperature	Langmuir constants				Freundlich constants			Recommended isotherms
	a	*b*	R^2	R_1	*K*	1/*n*	R^2	Langmuir isotherm
30°C	0.0098	0.0348	0.967	0.356	0.462	0.834	0.94	$\frac{1}{x/m} = 28.7 + \frac{2923.7}{c}$

3.7. Kinetics of adsorption studies

The study of kinetics of adsorption is an important parameter in wastewater treatment as it determines the solute uptake rate, which in turn controls the residence time of adsorbate uptake at the solid-solution interface. It was observed that the uptake of Cd(II) increases with the lapse of time. However, the adsorption of Cd(II) was fast for the first 2.5 h after which the rate slowed down as equilibrium approached. In order to determine the rate constant for adsorption, the first-order kinetic model has been used Fig. 12.

In order to define the adsorption kinetics of cadmium ion, the kinetic parameters for the adsorption processes were undertaken for the contact times ranging from 0.5 to 3.0 h and first-order, second-order, and intraparticle diffusion models were applied to the experimental data.

The kinetics in most cases follows the second-order rate equation. The obtained correlation coefficient $R^2 > 0.98$ showed that Cd(II) on WSAC followed the pseudo-second-order equation Fig. 13.

The plot of log (q_e-q) vs. t gives straight line for the first-order adsorption kinetics, and the rate constant k_{ad} is computed from the plot. For studying the adsorption kinetics of Cd(II) on adsorbent ASAC, first-order kinetics and second-order kinetics were applied to the batch experimental data as shown in Fig. 14.

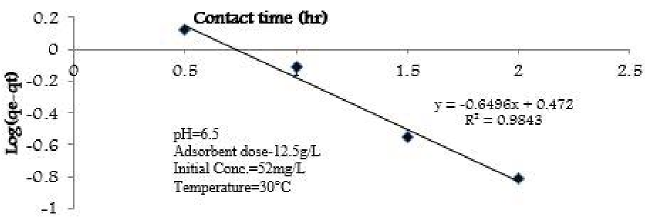

Fig. 12. Lagergren plot for walnut shell.

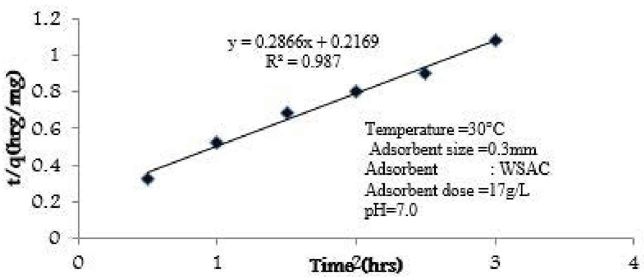

Fig. 13. Pseudo-second-order adsorption for walnut shell.

In order to define the adsorption kinetics of cadmium, the kinetic first-order and second-order equations were applied to the experimental data. The second-order equation appeared to the better fitting model than the first order because it has higher R^2 value Fig. 15.

3.8. Thermodynamic parameters; column studies

The experiments were performed at different temperatures (20°C, 30°C, and 40°C). The thermodynamic parameters such as change in standard free energy ($\Delta G°$), enthalpy ($\Delta H°$), and entropy ($\Delta S°$) can be evaluated using the equations Table 2. R is (8.314 J/mol K) being the gas constant, T (°K) the absolute temperature, and b (L/mol) the thermodynamic equilibrium constant. The obtained values of enthalpy and

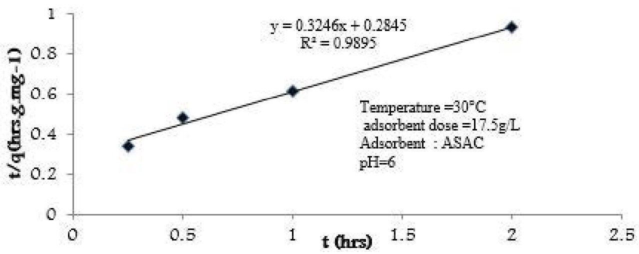

Fig. 14. Pseudo-second-order adsorption for almond shell.

Table 2
Thermodynamic parameters

Temperature (K)	Langmuir constant	Ln *b*	1/*T*(K^{-1})
293	0.0318	–1.497	3.412 x 10^{-3}
303	0.0348	–1.458	3.300 x 10^{-3}
313	0.0419	–1.377	3.194 x 10^{-3}

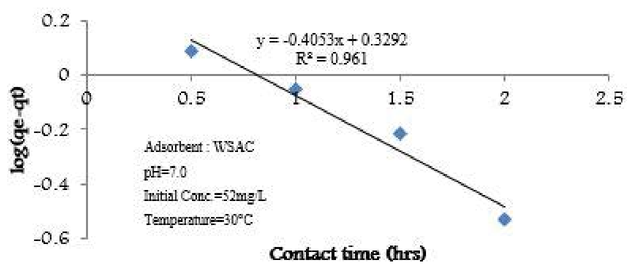

Fig. 15. Lagergren plot for WSAC.

entropy were calculated from the slope and intercept of the plot as shown in Fig. 16. Positive values of Δ*H* indicate that the adsorption of cadmium on the adsorbent is endothermic.

The apparent heat change (Δ*H*) related to Langmuir constant *b* follows the Van't Hoff equation. The enthalpy change of sorption as calculated from the slope of Ln *b* Vs 1/*T* is found to be 4.559 kJ/mol. The positive Δ*H* value confirms the endothermic nature of the sorption process and suggests the possibility of strong binding between sorbate and sorbent. The free energy (Δ*G*) and Δ*S* are given in Table 3.

The negative value of Δ*G* indicates the process to be feasible and spontaneous and positive values of entropy reflected the affinity of the adsorbent material. Positive values of Δ*H* indicate that the adsorption of metal ions on the adsorbent is endothermic (–Δ*H*/*R* = –261, Δ*H* = 2.169 kJ/mol and Δ*G* = –8.083 kJ/mol for Cd). The negative values of Δ*G* reveal the feasibility and spontaneous nature of the process. This result also shows that the adsorption capacity of WSAC for cadmium increases with increasing temperature. The Δ*S* values are very small which means that the entropy change occurring during the adsorption process is negligible [36].

The enthalpy increased with increasing sorption for ASAC from the slope of Ln b versus 1/*T* (Fig. 17) –Δ*H*/*R* = –436.36 are found Δ*H* = 3.627 kJ/mol. The positive Δ*H* value confirms the endothermic nature of the sorption process and suggests the possibility of strong binding between sorbate and sorbent.

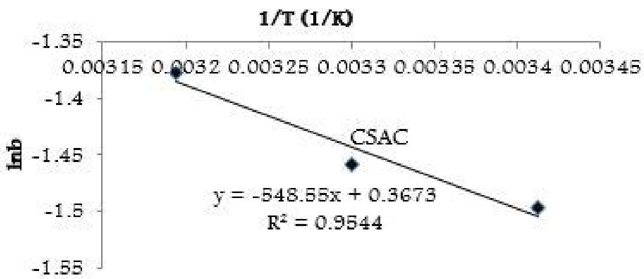

Fig. 16. Vant Hoff plot for CSAC.

Table 3
Thermodynamic parameters at different temperatures

Temperature (K)	–Δ*G* kJ/mol	Δ*S* J/mol
303	8.460	42.90

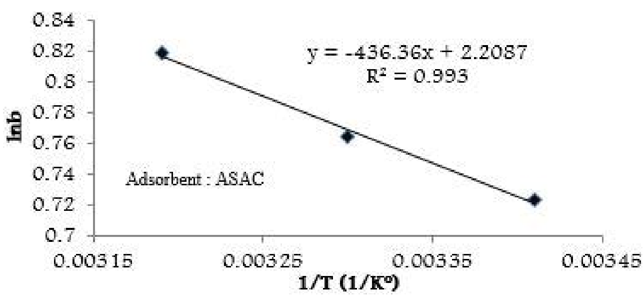

Fig. 17. Vant Hoff plot for ASAC.

4. Conclusion

The following conclusion could be drawn on the basis of the present study:

- The investigation shows that the agricultural waste adsorbents (coconut shell, almond shell, walnut) can be used as effective adsorbents for the removal of cadmium from electroplating and metal finishing wastewater. It was concluded that adsorption behavior is dependent on the nature of adsorbent and the different agro-based materials do not behave identically.
- pH is an important factor in adsorption processes since it causes electrostatic changes in the solution. The cadmium uptake capacity of all the adsorbents investigated is found to be highly pH dependent, and the best results were obtained in the pH range of 6–7.0.
- The highest removal efficiencies for Cd(II) of 83.7% at pH 6.5, 80.6% at pH 7, and 72.5% at pH 6 were obtained for CSAC, WSAC, and ASAC, respectively.
- The initial stage of Cd removal is found to be very fast and the later stage of Cd removal is much slower up to the saturation limit. The percentage removal of cadmium increases with decrease in metal concentration. The extent of removal depends on the metal concentration, adsorbent dose, contact time, and particle size. Coconut shell is found to be most effective with respect to removal efficiency.
- Cadmium adsorption follows second-order rate equation for coconut shell.
- The isotherm data obtained more closely follow the Freundilch adsorption isotherm for coconut shell, while for walnut shell follow the Langmuir adsorption isotherm better.
- With the increase in the amount of adsorbent, more cadmium ions can be removed from cadmium wastewater and removal of cadmium ion decreases with the increase in cadmium concentration in industrial wastewater.
- The agro-waste adsorbents studied do not hydrolize and BOD of leaches does not change. The adsorbents are therefore stable materials under process conditions.
- These adsorbents require only alkali/acid treatment to increase their efficiency.
- The powder of coconut shell has the potential to remove cadmium ion from electroplating wastewater.
- The results show that initial pH highly affected the uptake capacity of the adsorbent. For Cd(II) ions, the best value of pH for adsorption was 6.5.

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Conflict of Interest

The authors of this article declare that they have no conflict of interests.

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