

## Research Article

# Removal of Pb from Water by Adsorption on Apple Pomace: Equilibrium, Kinetics, and Thermodynamics Studies

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The adsorption-influencing factors such as pH, dose, and time were optimized by batch adsorption study. A 0.8 g dose, 4.0 pH, and 80 min of contact time were optimized for maximum adsorption of Pb on AP. The adsorption isotherms (Langmuir and Freundlich) were well fitted to the data obtained with values of  $q_{\max}$  (16.39 mg/g;  $r^2 = 0.985$ ) and  $K$  (16.14 mg/g;  $r^2 = 0.998$ ), respectively. The kinetics study showed that lead adsorption follows the pseudo-second-order kinetics with correlation coefficient ( $r^2$ ) of 0.999 for all of the concentration range. FTIR spectra also showed that the major functional groups like polyphenols (–OH) and carbonyl (–CO) were responsible for Pb binding on AP. The thermodynamic parameters as  $\Delta G$ ,  $\Delta H$  (33.54 J/mol), and  $\Delta S$  (1.08 J/mol/K) were also studied and indicate that the reaction is feasible, endothermic, and spontaneous in nature.

## 1. Introduction

Due to the industrialization, especially in the developing countries, the emission of the heavy metals as lead, cadmium, chromium, nickel, arsenic, and mercury are highly concerned to public and aquatic health. Lead is released with the effluent from the paint, batteries, and automobiles manufacturing units. Lead is one of the toxic metals and largely affects the central, peripheral nervous system. Besides this the other toxic effects of the lead are visual disturbances, convulsions, loss of appetite, antisocial behaviors, constipation, anemia, tenderness, nausea, vomiting, severe abdominal pain, anemia, and gradual paralysis in the muscles [1]. There are several methods for removing heavy metals from aqueous solutions, such as chemical precipitation, membrane filtration, ion exchange, reverse osmosis, and adsorption [2]. However, the methods for the removal of metal are expensive, difficult, incomplete, and generate large amount of solid waste.

Among these the adsorption process is the most demanding technique which is easily accessible and economically feasible for the removal of water contaminants [3, 4]. It is the most suitable process for the removal of metals due to low cost, being easily obtained, and minimizing the volume

of chemical and biological sludge. Adsorption of metals involves several mechanisms that differ qualitatively and quantitatively, according to the species used, the origin of the biomass, and its processing procedure [5]. The literature was reviewed and it was found that different kinds of the adsorbent material as activated carbon [6], pine cone [7] grape bagasse [8], pine needles [9], peels of banana are used for removal of particular metals from water.

In India, out of 5000 tons of apple pomace (AP) 3000 tons are produced in Himachal Pradesh [10]. Its huge production in apple juice industry becomes a challenge of its utilization as well as its disposal. Presently, the apple pomace produced after the extraction juice from its manufacturing unit is disposed off in the field for natural decomposition. After period of time the waste undergoes anaerobic decomposition during rain and cause environmental pollution by releasing significant amount of methane. Globally, about 3–19% emission of total anthropogenic methane was contributed by waste dumping site [11]. This is also creating the problem for the public as well as the environment. About 25–30% of apple pomace is left of the total processed fruit, which is rich in polyphenols, polysaccharides, pectins, cellulose hemicellulose, and lignin [10]. Since these contains the functional groups, –COO, –CO, –NH<sub>2</sub>, and –OH they are

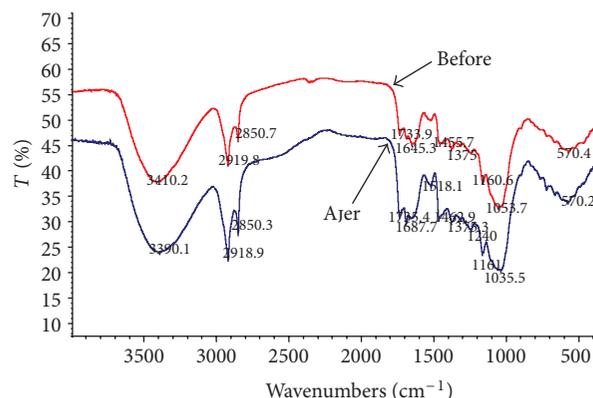


FIGURE 1: FTIR spectra of AP before and after adsorption of Pb ions.

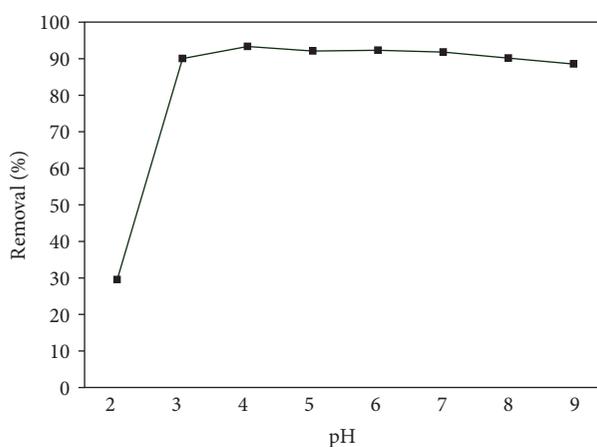


FIGURE 2: Effect of pH on adsorption of Pb ions onto AP (dose of 0.8 g, metal concentration of 50 mg/L).

highly responsible for the metals binding capacity [12]. The literature reported that the polyphenols are highly efficient for the removal of lead than the others metals [13].

Since Pb ion had large binding capacity with polyphenols and AP is rich source of polyphenols, by viewing this fact, the present study investigated for removal of Pb from water by the surface adsorption method which is inexpensive, feasible, and environmental friendly. The adsorption model as Langmuir and Freundlich satisfied the data with regression coefficient ( $r^2$ ),  $q_{max}$ , and  $K$  for Pb. Spontaneity of adsorption process with respect to temperature and the behavior of adsorption with the passage of time were studied using thermodynamic and kinetic studies. The adsorption parameters as pH, dose, and time were studied which affect the adsorption.

## 2. Materials and Methods

**2.1. Chemicals.** All the chemicals used in the present work were of analytical grade. The filter papers were obtained from Qualigens (125 mm), 615A, Germany. The stock solution of Pb (1000 mg/L) was prepared by dissolving appropriate amount of lead nitrate  $Pb(NO_3)_2$  (Sd Fine Chemicals Ltd. Mumbai, India). The desired concentrations of lead solutions

were prepared by appropriate dilution of the stock solution for adsorption studies.

**2.2. Apple Pomace.** The AP was collected from Himachal Pradesh Horticultural Produce Marketing and Processing Corporation (HPMC), processing unit Parwanoo, District Solan, Himachal Pradesh, India. The AP was dried at room temperature, crushed in an electric grinder to make fine particle size and sieved through 0.5 mm pore size. The sieved biosorbent was stored in a container for further adsorption study.

**2.3. Batch Studies.** The optimization of batch adsorption parameters, that is, dose, pH, and time for AP, was performed in a synthetic solution of Pb metal by varying a single parameter at a time with respect to that of the others constant. After adsorption, the flask was removed and filtered through Wattmann filter paper and the filtrate analyzed for residual metal. The effect of AP dose and favourable pH was investigated between 0.1 to 1.2 g and 2 to 9, respectively, in 50 mL synthetic solutions of lead ions. 0.1 N of HCl and NaOH were used for pH adjustment. The pH was measured by Cyberscan PC510 (Eutech, Singapore). The

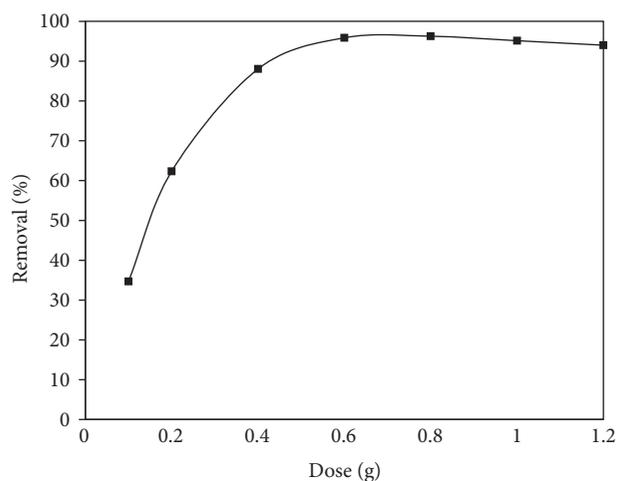


FIGURE 3: Effect of adsorbent dose (0.1–1.2 g) on adsorption of Pb ions onto AP (pH of 4.0, metal concentration of 50 mg/L).

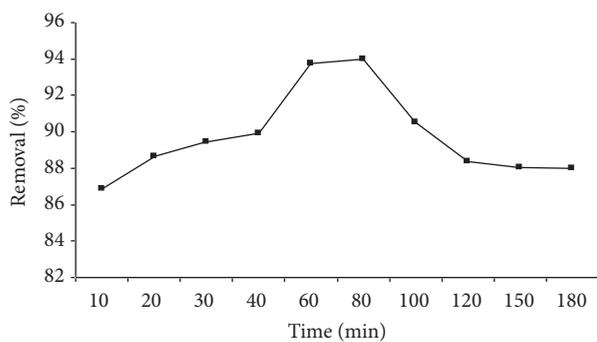


FIGURE 4: Effect of time on Pb adsorption onto AP (dose = 0.8 g, pH = 4, metal concentration of 50 mg/L).

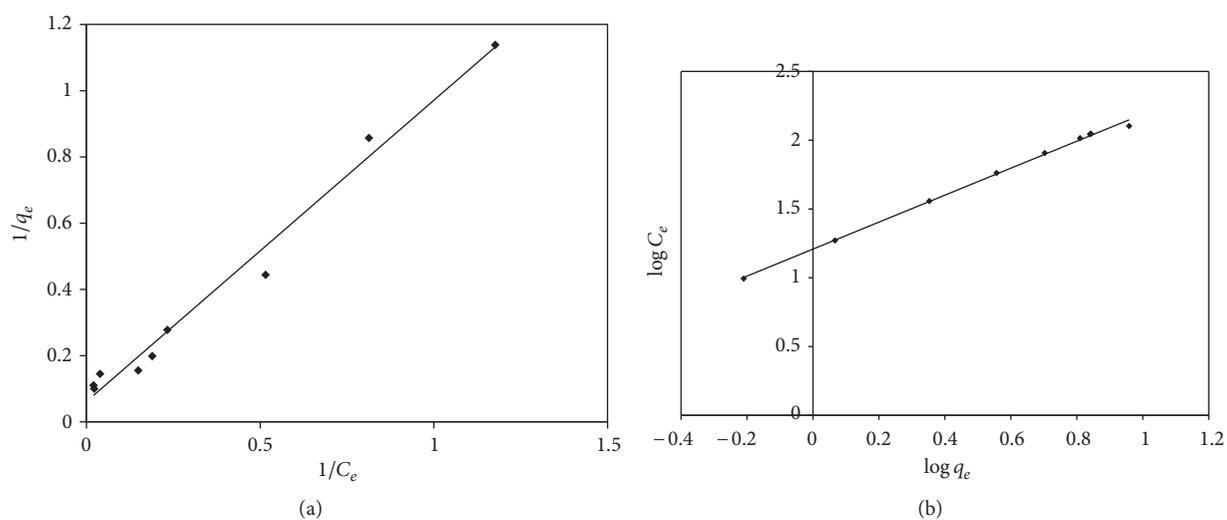


FIGURE 5: (a) Graphical representation of Langmuir isotherm. (b) Graphical representation of Freundlich isotherm (dose = 0.8 g, pH = 4, time = 80 min, metal concentration = 10–200 mg/L).

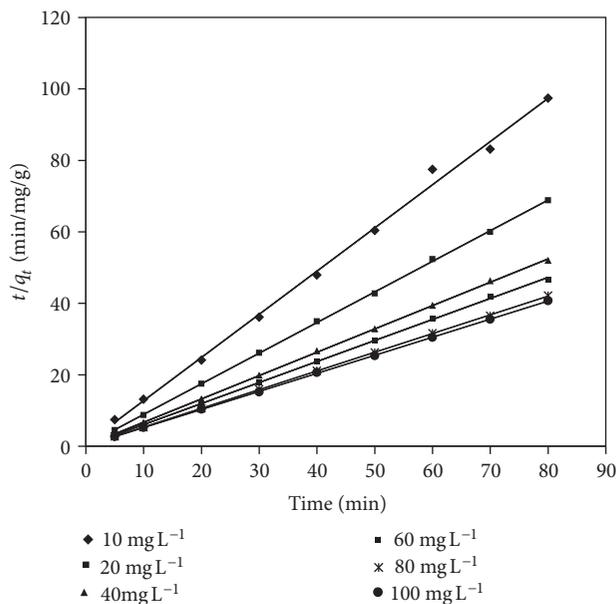


FIGURE 6: Pseudo-second order of kinetics for Pb adsorption on AP (dose = 0.8 g, pH = 4, time = 5–80 min, concentration = 10–100 mg/L in 50 mL of synthetic water).

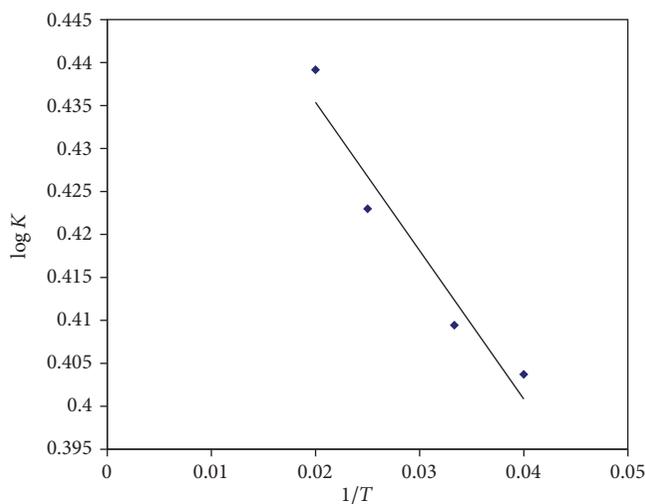


FIGURE 7: Plot of  $\log K$  versus  $1/T$  for determination of  $\Delta H$  and  $\Delta S$  values (dose = 0.8 g, temp = 25–60°C, pH = 4, time = 80 min metal concentration of 50 mg/L).

TABLE 1: FTIR spectral characterization of AP before and after Pb adsorption.

IR peaks	Assignment			Functional groups
	Before adsorption	After adsorption	Differences	
1	3410.2	3390.1	-20.1	-OH group in bonded form
2	2919.8	2918.9	-0.9	Aliphatic -C-H stretching
3	2850.7	2850.3	-0.4	-C-H stretching
4	1733.9	1735.4	1.5	-C=O, stretching of ester group
5	1645.3	Disappear	Unknown	-C=O stretching of carbonyl group
6	1455.7	1462.9	7.2	Carboxyl groups
7	1375.0	1375.3	0.3	-C-O-C- stretching of ethers groups
8	1160.6	1161.0	0.4	-C-O stretching
9	1053.7	1035.5	-18.2	-C=O group
10	570.4	570.2	-0.2	-C-C- groups

TABLE 2: Various biosorbent available for Pb adsorption from water.

Adsorbent	Langmuir $q_{\max}$ (mg/g)	Freundlich $K$	References
Natural spider silk	1.17	1.39	[14]
Peels of banana	2.18	2.04	[15]
<i>Polygonum orientale</i> activated carbon	98.41	41.85	[16]
Coconut shell activated carbon	18.1	15.25	[17]
Sericite	4.697	2.056	[18]
<i>Oryza sativa</i> husk	8.69	—	[19]
Walnut shell	31.23	9.14	[20]
Tobacco stems	5.57	0.94	[21]
Retorted shale	36.15	7.75	[22]
Almond shells	8.13	4.58	[23]
Rice husk ash	12.61	3.948	[24]
Bagasse fly ash	2.50	2.57	[25]
Apple pomace	16.39	16.14	Present study

TABLE 3: Pseudo-second order of kinetics for Pb adsorption onto AP.

Concentration (mg/L)	Equation	$r^2$	$q_e$ (mg/g)	$K$
10	$Y = 1.2089x + 0.6502$	0.9968	0.82	2.28
20	$Y = 0.8574x + 0.2923$	0.9998	1.167	2.51
40	$Y = 0.6530x + 0.1515$	0.9995	1.530	2.81
60	$Y = 0.588x + 0.1803$	0.9995	1.699	1.92
80	$Y = 0.5237x + 0.1157$	0.9997	1.909	2.37
100	$Y = 0.5059x + 0.1571$	0.9994	1.977	1.63

contact time and concentration varied between 10–180 min and 10–100 mg/L for Pb adsorption onto AP. Once the pre-set contact time reached, the solutions were filtered. The filtrate was analyzed for residual metal ion concentration by using Atomic Absorption Spectrophotometer (Shimadzu model AA 6300, Tokyo, Japan). The temperature was maintained for thermodynamics study by incubator Innova 44, New Brunswick scientific, New Jersey, USA, at optimized condition.

**2.4. FTIR Spectral and Surface Area Analysis.** The AP was characterized by FTIR (Fourier transform infrared spectrophotometer) spectral analysis to know the major functional group responsible for the Pb binding on AP. FTIR was done by using FTIR, Thermo Scientific, Nicolet 6700, Madison, USA, with KBr (spectroscopic grade). The IR spectra were recorded in the range of 400 to 4000  $\text{cm}^{-1}$ . Surface areas of AP before and after adsorption were obtained by  $\text{N}_2$  adsorption using Micromeritics ASAP-2000. Surface areas were calculated by applying the BET method.

**2.5. Adsorption Isotherm and Kinetics Study.** Nine concentration ranged from 10–200 mg/L of Pb ions were prepared from the stock solution of 1000 mg/L by appropriate dilution. The optimized parameters as dose (0.8 g), pH (4), and time (80 min) remain constant for adsorption isotherm study. The kinetic study was done to observe the behavior of adsorption with the passage of time to establish equilibrium. The concentration of Pb ions from 10–100 mg/L in 50 mL was

used in all optimized condition and each flask was removed after regular interval of time from 5–80 min.

**2.6. Thermodynamics Study.** Effect of temperature on Pb adsorption was studied from 25–50°C in incubator shaker at initial concentration (50 mg/L) of Pb ion. Thermodynamics study was done to confirm the feasibility and spontaneity of adsorption process by using the Van't Hoff equation [26, 27]. By using the Van't Hoff equation a plot of  $\log K$  versus  $1/T$  is drawn to found the slope and intercept which is  $\Delta H$  and  $\Delta S$ , respectively.

### 3. Results and Discussion

**3.1. Characterization of AP.** The different functional groups responsible for the Pb binding on AP were observed by FTIR spectra and data are given in Figure 1 and Table 1. The more number of peaks represent the adsorptive nature of AP. The peaks were observed at 3410.2, 2919.8, 2850.7, 1733.9, 1645.3, 1455.7, 1375, 1160.6, 1053.7, and 570.4  $\text{cm}^{-1}$ . The broad band at the region of the 3200–3400  $\text{cm}^{-1}$  is due to the presence of polyphenols (–OH) groups. The peaks at 2850.7 to 2919.8  $\text{cm}^{-1}$  represent the –C–H stretching of aliphatic carbon chain. The peaks at 1733.9, 1645.3, 1455.7, and 1375  $\text{cm}^{-1}$  showed the presence of the –C=O of ester, carbonyl (–CO) and –C–O–C– group of ether. The last three peaks at 1160.6, 1053.7, and 570.4  $\text{cm}^{-1}$  were observed due to stretching of –CO, –C=O and –C–C– groups, respectively.

The spectral analysis of AP before and after adsorption of Pb ions showed that the peaks either decreases in intensity or disappear might involve in metals adsorption [28]. The peaks at 3410.2, 2919.8, 2850.7, 1645.3, 1053.7, and 570.4  $\text{cm}^{-1}$  are because of polyphenols ( $-\text{OH}$ ) groups, aliphatic  $-\text{C}-\text{H}$  stretching, carbonyl group ( $-\text{CO}$ ),  $-\text{C}=\text{O}$ , and  $-\text{C}-\text{C}-$  groups, respectively, and were responsible for the Pb binding. The surface area of AP before and after adsorption was found to 0.7129  $\text{m}^2/\text{g}$  and 0.4834  $\text{m}^2/\text{g}$ , respectively. It indicated that the adsorption of Pb was successfully carried out onto AP.

**3.2. Effect of pH.** pH is an important variable in the ion exchange governed adsorption process than the other physicochemical parameters. In order to observe the effect of pH on adsorption of Pb, AP contacted with Pb solution at different pH (2–9). The effect of pH on Pb adsorption on AP is summarized in Figure 2. At lower pH, the adsorption was found low due to sorbet lyphobic behavior [29]. After pH 2, adsorption increased sharply up to pH 4 and thereafter no significant change was observed for greater pH. As the pH increases, the lower the number of  $\text{H}^+$  ion and greater number of negatively charge metal binding sites are available for metal adsorption [30]. The maximum adsorption of 93% was observed at pH 4.0 for Pb ions and no significant difference observed by increasing pH. This may be due to the established equilibrium between metal and hydrogen ions. Similar finding was observed for Pb adsorption on chitosan [31]. No pH values over 9.0 were studied due to the precipitation of metals ion occurs.

**3.3. Effect of Adsorbent Dose.** The adsorbent dose of AP was investigated from 0.1–1.2 g/50 mL of Pb ions solution (Figure 3). The result showed that as the dose ( $g$ ) of adsorbent increased, the % removal also increases; this is due to the more availability of the adsorbent surface for complex formation with metal ions in water. The maximum removal of Pb ions was observed at 0.8 g dose of AP from water. The steady state was obtained by further increased in the dose of adsorbent towards Pb metal ion adsorption. Further decrease in adsorption may be due to the adsorbent gets aggregated and provides less effective surface area for metal binding.

**3.4. Effect of Time.** Time plays important role in adsorption of metal ions on adsorbent surface. The effect of time on Pb adsorption onto AP was studied in the range of 10–180 min (Figure 4). The result revealed that as the time increased the rate of adsorption increased upto 80 min and sudden decreased in adsorption rate was observed beyond 80 min from 94 to 90%. The maximum removal of 94% was found at 80 min. So the optimized time for the maximum removal of Pb was 80 min.

**3.5. Adsorption Isotherm.** The Langmuir and Freundlich isotherms (Figures 5(a) and 5(b)) were obtained to establish the equilibrium data in concentration ranged from 10–200 mg/L at optimized condition of pH, time, and dose. The data obtained from the study of adsorption at different concentration applied to Langmuir and Freundlich isotherm by using well known adsorption isotherm equation [15].

Both the adsorption isotherms were well fitted with  $q_{\text{max}}$  and correlation coefficient ( $r^2$ ) for the adsorption of Pb metal ions on AP. In case of Langmuir isotherm maximum adsorption capacity ( $q_{\text{max}}$ ) and constant  $b$  for adsorption of Pb on AP were found to be 16.39 mg/g, and 0.67 L/g with correlation coefficient of 0.985, respectively. On the other hand value of Freundlich constant ( $K$ ) and  $n$  was found to be 16.14 mg/g, and 1.02 with correlation coefficient of 0.998, respectively. The value of  $n$  less than one indicate chemical adsorption while the value greater than one tells about the physical process [32]. Since the  $n$  value was found above one, that is, 1.02 indicated that adsorption on AP surface was carried out by physical process. The  $q_{\text{max}}$  values of other researchers studied for Pb removal was compared with present study (Table 2).

**3.6. Kinetic Study.** In order to find the effect of time, the concentration ranged from 10–100 mg/L were studied in 250 mL of volumetric flasks at pH of 4, dose 0.8 g and time from 5–80 min. The pseudo-second order kinetics model has been used to evaluate the experimental kinetics data of AP [33, 34]. Linear plot was between  $t/q_t$  versus  $t$ , whose intercept and slope gives the value of  $K$  and  $q_e$  (mg/g), respectively, (Figure 6). The pseudo second order equation of all selected concentration from 10–100 mg/L with their  $q_e$ ,  $r^2$  and  $K$  values were given in Table 3. The correlation coefficient of ( $r^2$ ) for the second order of kinetics was found to be 0.999 which showed that reaction rate follows pseudo-second order kinetics.

**3.7. Effect of Temperature.** In thermodynamics study, in the isolated system, energy neither be created nor destroyed and the change in entropy is the only energy source. In environment concept of the energy and entropy must calculate for the occurrence of spontaneity of the adsorption reaction. From Van't Hoff equation a plot of  $\log K$  versus  $1/T$  (Figure 7) was drawn to found the slope and intercept for  $\Delta H$  and  $\Delta S$ , respectively. The value of  $\Delta H$  and  $\Delta S$  was found to be 33.51 J/mol and 1.08 J/mol/K, respectively, which indicated that the adsorption was endothermic and increased entropy. The value of  $\Delta G$  was found to be  $-62.79$ ,  $-64.69$ ,  $-68.94$ , and  $-73.84$  J/mol at temperature of 293, 303, 313 and 323 K. As the temperature increases the negative value of  $\Delta G$  also increased which indicate the feasibility and spontaneity of Pb adsorption onto AP. The endothermic nature of reaction was also supported by increase in adsorption when temperature increases.

## 4. Conclusions

The present study was aimed to evaluate the industrial waste AP for the Pb adsorption from synthetic water. The result revealed that 0.8 g of dose, 4 pH, and contact time of 80 min were optimized for maximum removal of Pb from water. This study also follows the Langmuir and Freundlich isotherm with  $q_{\text{max}}$  value of 16.39 mg/g and  $K$  of 16.14 mg/g, respectively. In case of thermodynamics study the negative value of  $\Delta G$ , positive value of  $\Delta H$  (33.54 J/mol) and  $\Delta S$  (1.08 J/mol/K)

were indicated that the reaction is feasible, endothermic and spontaneous in nature. The kinetics study showed that the adsorption of Pb onto AP follows the pseudo second order of kinetics ( $r^2 = 0.999$ ). The result revealed that the AP may prove to be good, economical, cheap, and environment friendly adsorbent for the Pb removal from water.

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