CHAPTER -2

Adsorption Studies of Pb (II) on Arjun Tree Fruit (Terminalia arjuna)
2.1. INTRODUCTION

Owing to the rapid industrialization and urbanization, environmental degradation is accelerating day by day due to the contaminants being released in air, soil and water [Zhou et al., 2009]. The most significant pollutants found in wastewater are heavy metals. They are persistent in nature and highly toxic even at ultra trace levels [Ozdesa et al., 2009]. Since they are non-biodegradable they tend to accumulate in the tissues and vital organs of living organism thereby causing various diseases and long term disorders [Loutseti et al., 2009]. Among the variety of heavy metals found in wastewater Pb(II) is considered to be one of the most poisonous contaminant. This metal is known to cause a number of adverse effects on human health as it can accumulate in the kidney, liver, brain, bones, and muscles. Chronic toxicity occurs at blood levels of 0.4–0.6 mg.L⁻¹ [Flora et al., 2012]. The maximum permissible limit assigned by World Health Organization (WHO) for Pb(II) in drinking water is 0.05 mg.L⁻¹ [Rao et al., 2011]. As reported by health organizations, Pb(II) can alter numerous metabolic body processes and induce impairment and dysfunction in the blood and cardiovascular system in human adults and particularly in children [Moreira et al., 2001]. The condition of children in developing countries is quite miserable with more than 15 million of them are suffering from permanent neurological damage due to Pb(II) poisoning [HEI special report 15, 2004].

Keeping in mind the hazardous effects of Pb(II) it seems to be the need of hour to remove Pb(II) from water and wastewater. Some of the techniques commonly employed to remove Pb(II) from aqueous solutions include membrane filtration, ultra-filtration and reverse osmosis, chemical precipitation of Pb(II) as the hydroxide and sulfide and ion-exchange [Gzara et al., 2001; Chen et al., 2009; Kongsuwan et al., 2009; Deng et al., 2013; Galindo et al., 2013; Sarada et al., 2013]. All these methods suffer from one or the other drawback. Some of the drawbacks include high operational cost, production of a chemical byproduct that requires further treatment, low removal efficiency, difficult implementation etc. Adsorption is a quite attractive process for the removal of Pb(II) from aqueous solutions. Activated carbon has long been used as effective adsorbent but owing to its high cost and difficulty in regeneration some naturally occurring biomass or by-products obtained from agricultural or industrial processes are preferably used in adsorption process as they are available in abundance, economical, easily prepared and eco-friendly. The most
significant feature of this naturally occurring biomass is that they do not release any toxic or objectionable substance when added into water or wastewater for treatment. Various studies on Pb(II) removal from aqueous solution using natural adsorbents include activated carbon prepared from biomass materials [Goel et al., 2005], rice husks [Elham et al., 2010; Singha et al., 2012], tree fern [Ho et al., 2004], maize husks [Zvinowanda et al., 2010], sawdust [Ahmad et al., 2009; Ofomaja et al., 2010], sugarcane bagasse [Martin-Lara et al., 2010], coriander seed [Rao et al., 2012], NOC (neem oil cake) [Rao et al., 2007], modified water hyacinth [Nabila et al., 2014], castor leaf powder [Amanda et al., 2013], green alga [Sari et al., 2008], laminaria japonica [Ghimi et al., 2008] etc.

The objective of the present work is to explore the adsorption efficiency of *Terminalia arjuna* fruit for the removal of Pb(II) from aqueous solution. *Terminalia arjuna*, commonly known as arjuna or arjun belongs to the family combretaceae. It is a large evergreen tree and the height reaches upto 60-80 feet. It is mainly found in the greater parts of Indian peninsula along rivers and sub-Himalyan tract, chota nagpur, orissa, west Bengal, Punjab, deccan and konkan. Flowering begins in April and extends to May with the fruit ripening the following February-May, nearly a year after the appearance of the flowers. The diameter of fruit is 1-1.5 inch with 5-7 longitudinal lobes [Parakh, 2010]. *Terminalia arjuna* is a widely used medicinal plant. The bark of the plant is quite useful and finds its application in the treatment of heart diseases, diabetes, tumour, skin disorder, inflammation, asthma, etc [Parakh, 2010]. The bark, leaves and fruit possesses glycosides, large quantities of flavonoids, tannins and minerals [Nema et al., 2012]. Phenolic contents of these parts are also quite high (72.0–167.2 mg.kg\(^{-1}\)) [Bajpai et al., 2005]. In comparison to other commonly used medicinal plants, bark of *Terminalia arjuna* contains a very high level of flavonoids. Some of the flavonoids obtained from its bark include pelargonidin, arjunolone, flavones, kempferol, quercetin, and bicalein [Nema et al., 2012]. The fruit of *Terminalia arjuna* is found to possess large number of phytochemicals namely: Arjunone, methyl oleanolate, gallic acid, hentriacontane, arachidic stearate, ellagic acid, arjunic acid, β-sitisterol, friedelin, myristil oleate [Parakh, 2010].
2.2. MATERIAL AND METHODS

2.2.1. Preparation of adsorbent

Terminalia arjuna nuts used in this study were collected locally from university campus of Aligarh, U.P., India in the month of May. Collected nuts were washed several times with distilled water in order to remove adhering dirt and dust particles and then dried in an open air oven at 60°C for 48 h. The dried sample was grounded and sieved through 0.300-0.150 mm screen size using standard ASTM sieves. The sieved sample was again washed repeatedly with double distilled water (DDW) until the wash water contained no colour. The biomass was finally dried at 60°C for 24 h in oven and was stored in an air tight bottle for further study.

2.2.2. Chemicals and reagents

All chemicals and reagents used were of analytical grade (A.R) and were obtained from Merck, Germany. They were used as such without further purification. Stock solution of Pb(II) (1000 mg.L\(^{-1}\)) was prepared by dissolving appropriate amount of lead nitrate salt in required volume of doubled distilled water (DDW). Working standard solutions of Pb(II) of desired concentrations were obtained by diluting the stock solution.

2.2.3. Characterization of adsorbent

To investigate the physical surface morphology of the adsorbent before and after Pb (II) adsorption, scanning electron microscopy (SEM) of gold coated samples were carried out on a carbon tape in a JSM-6510LV scanning electron microscope (JEOL, Japan) at an accelerating voltage of 20 kV and magnification of 7500. The type of binding groups present on the adsorbent were examined by Fourier transform infrared (FTIR) analysis with pellets prepared by mixing spectroscopic grade potassium bromide (KBr) and the adsorbent in the ratio of 4:1. The spectra were recorded in the range of 400–4000 wave number (cm\(^{-1}\)) in the diffuse reflectance mode at a resolution of 4 cm\(^{-1}\) in KBr pellets. The determination of elemental composition of the adsorbent before and after Pb(II) adsorption was carried out using the Oxford Instruments INCA X-sight energy dispersive X-ray (EDAX) spectrometer equipped SEM. The zero surface charge characteristic of the adsorbent was determined by solid addition method [Lataye et al., 2006].
2.2.4. Batch adsorption studies

The adsorption properties of TAFP were investigated as a function of adsorbent dose, initial pH, initial metal ion concentration, contact time and temperature. Thermodynamics and kinetics studies were carried out by batch process. 0.2 g of adsorbent was weighed and placed in a series of 250 mL standard conical flasks containing 20 mL of Pb(II) solution of desired concentration. The mixture was then shaken in a temperature controlled incubator shaker for different contact times (5 min to 24 h). The conical flasks were taken out from shaker at a predetermined time interval and the mixture was filtered using whatmann filter paper No.1. The final concentration of Pb(II) in filtrate was then determined by atomic absorption spectrophotometer (AAS). The % adsorption of Pb(II) and the adsorption capacity, $q_e$ (mg.g$^{-1}$) of the adsorbent at equilibrium were determined using the following relationships.

\[
\% \text{ Adsorption} = \left(\frac{C_o - C_e}{C_o}\right) \times 100
\]

\[
\text{Adsorption capacity, } q_e = \left(\frac{C_o - C_e}{W}\right) \times V
\]

Where, $C_o$ & $C_e$ are the initial and final Pb(II) concentration respectively; $V$ is the volume of the solution (L) and $W$ is the mass of the adsorbent (g).

2.2.4.1. Effect of contact time

A series of 250 mL conical flasks, each containing 0.2 g adsorbent and 20 mL solution of Pb(II) of concentration 50 mg.L$^{-1}$, were shaken in a temperature controlled incubator shaker. The flasks were taken out from shaker at a predetermined time interval and were filtered using whatmann filter paper No.1. The concentration of Pb(II) in each filtrate was then determined by AAS and the amount of Pb(II) adsorbed in each case was determined as described above.

2.2.4.2. Effect of pH

The effect of pH was studied in the pH range of 1–10. The experiments were carried out in batch mode as follows: 40 mL of Pb(II) solution of concentration 50
mg.L\(^{-1}\) was taken in a beaker. The pH adjustment of solution was done by adding either 0.1N HNO\(_3\) or 0.1N NaOH. The concentration of Pb(II) in this solution was then determined (initial concentration). 20 mL of this solution was taken in conical flask and treated with 0.2 g adsorbent. The mixture was shaken in a shaker incubator at the speed of 110 rpm. After equilibrium, the mixture was filtered and the final concentration of Pb(II) in filtrate was determined by AAS. The final or equilibrium pH (pH\(_f\)) was also recorded by using pH-meter. In order to investigate the effect of electrolyte (KNO\(_3\)), same procedure was followed as described above except that Pb(II) solution of 50 mg.L\(^{-1}\) concentration was prepared in 0.1N KNO\(_3\) solution instead of DDW.

### 2.2.4.3. Point of zero charge (PZC)

The zero surface charge characteristics of the adsorbent were determined by solid addition method [Lataye et al., 2006]. The experiment was carried out as follows: 15 mL of 0.1 N KNO\(_3\) solution was placed in each of a series of 100 mL conical flasks. The initial pHs (pH\(_i\)) of the solutions were roughly adjusted between 1 to 10 by adding either 0.1 N HNO\(_3\) or 0.1 N NaOH solutions. The total volume of the solution in each flask was made up to 20 mL by adding KNO\(_3\) solution of the same strength. The initial pH values (pH\(_i\)) of the solutions were accurately measured by using pH-meter. Thereafter 0.2 g of adsorbent was transferred into each flask and the mixture was allowed to equilibrate for 24 h with intermittent manual shaking. After equilibrium, solutions were filtered and the final pH (pH\(_f\)) of the supernatant liquid was recorded. The point of zero charge curves was obtained by plotting difference between the initial pH (pH\(_i\)) and final pH (pH\(_f\)) i.e. ΔpH values against pH\(_i\). The point of intersection of the resulting curve with the abscissa, at which ΔpH = 0, gave the pH\(_{\text{PZC}}\) value.

### 2.2.4.4. Effect of adsorbent dose

To study the effect of adsorbent dose on the adsorption of Pb(II), a series of 250 mL conical flasks each containing a fixed volume of Pb(II) solution (20 mL) of known concentration (50 mg.L\(^{-1}\)) were treated at 30°C with varying amount of adsorbent (0.1–1.0 g). The flasks were kept in a shaker incubator and shaken at the speed of 110 rpm the solutions were then filtered after 24 h. The amount of Pb(II) in the filtrate was then determined by AAS.
2.2.5. Breakthrough studies

0.5 g of adsorbent was accurately weighed and transferred into a glass column (0.6 cm internal diameter) with glass wool support. 250 mL of Pb(II) solution with initial concentration (C₀) of 50 mg·L⁻¹ was then passed through the column with a flow rate of 1 mL·min⁻¹. First 100 mL of the effluent was collected in 10 mL fractions and the remaining volume was collected in 50 mL fractions. The amount of Pb(II) (Cₑ) in each fraction was then determined by AAS. The breakthrough curve was obtained by plotting Cₑ/C₀ versus volume of the effluent.

2.2.6. Desorption studies

The exhausted column obtained after the determination of breakthrough capacity was used to study the desorption behaviour. The column was washed several times with DDW in order to remove traces of Pb(II) ions remained unadsorbed. 0.1N HCl was used as desorbing solution for the desorption of Pb(II) ions from adsorbent. The solution was passed through the column at a flow rate of 1 mL·min⁻¹ and the effluent was collected in 10 mL fractions. Each fraction was then analyzed by AAS for determining the amount of Pb(II) desorbed.
2.3. RESULTS AND DISCUSSIONS

The adsorption efficiency of *Terminalia arjuna* fruit powder (TAFP) for various heavy metal ions was investigated and it was observed that the adsorbent showed highest affinity towards Pb(II) followed by Zn(II), Ni(II), Cd(II), Cr(VI) and Cu(II) (Fig. 2.1). The monolayer adsorption capacity of Pb(II) investigated in the present study is higher than many other non conventional adsorbents reported earlier as summarized in Table 2.1.

![Graph showing percent adsorption of heavy metal ions from aqueous solution onto TAFP](image)

**Fig 2.1:** Percent adsorption of heavy metal ions from aqueous solution onto TAFP (Conditions; adsorbent dose = 0.2 g, temperature = 30° C)
Table 2.1: Comparison of the adsorption capacity of various adsorbents towards Pb(II) ions as reported in literature

<table>
<thead>
<tr>
<th>Adsorbents</th>
<th>Adsorption capacity (mg.g⁻¹)</th>
<th>References</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ball clay</td>
<td>03.52</td>
<td>Rao et al., 2011b</td>
</tr>
<tr>
<td>Barley straw</td>
<td>23.20</td>
<td>Ho et al., 2004</td>
</tr>
<tr>
<td>Coir</td>
<td>18.90</td>
<td>Conrad et al., 2007</td>
</tr>
<tr>
<td>Chemically modified oil palm fruit fibre</td>
<td>05.58</td>
<td>Abia et al., 2008</td>
</tr>
<tr>
<td>Coral</td>
<td>01.14</td>
<td>Ahmad et al., 2012</td>
</tr>
<tr>
<td>Egg shell</td>
<td>04.74</td>
<td>Ahmad et al., 2012</td>
</tr>
<tr>
<td>Hazelnut shells</td>
<td>16.23</td>
<td>Iqbal et al., 2009</td>
</tr>
<tr>
<td>Imperata cylindrica leaf powder</td>
<td>13.50</td>
<td>Hanafiah et al., 2006</td>
</tr>
<tr>
<td>Oil palm shell</td>
<td>03.39</td>
<td>Chong et al., 2013</td>
</tr>
<tr>
<td>Olive cake</td>
<td>19.53</td>
<td>Iqbal et al., 2009</td>
</tr>
<tr>
<td>Orange peels</td>
<td>01.22</td>
<td>Xuan et al., 2006</td>
</tr>
<tr>
<td>Pinus sylvestris</td>
<td>09.71</td>
<td>Ghasemi et al., 2014</td>
</tr>
<tr>
<td>Rice husk</td>
<td>11.40</td>
<td>Iqbal et al., 2009</td>
</tr>
<tr>
<td>Terminalia arjuna fruit powder</td>
<td>27.39</td>
<td>Present study</td>
</tr>
</tbody>
</table>
2.3.1. Characterization of adsorbent

2.3.1.1. Scanning electron microscope (SEM) analysis

SEM was carried out to examine the surface morphology of TAFP before and after adsorption of Pb(II). Scanning electron micrographs are depicted in Fig. 2.2. In both the cases surface of the adsorbent appeared to be irregular and porous. The surface of adsorbent before adsorption of Pb(II) seems to be less dense while after adsorption it became denser, showing adherence of Pb(II) on the surface.

2.3.1.2. EDX analysis

EDX spectra of TAFP before and after adsorption of Pb(II) are illustrated in Fig. 2.3. Spectra before adsorption clearly indicated the peak for the presence of carbon and oxygen as major constituents. EDX spectra after adsorption showed a peak due to Pb(II). The weight % of different constituents obtained from EDX analysis before and after Pb(II) adsorption are reported in Table 2.2. Moreover, before and after adsorption no characteristic peaks were observed for any impurities.

Table 2.2: EDX analysis

<table>
<thead>
<tr>
<th>Elements</th>
<th>Before adsorption</th>
<th>After adsorption</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Weight%</td>
<td>Atomic%</td>
</tr>
<tr>
<td>C</td>
<td>45.46</td>
<td>52.61</td>
</tr>
<tr>
<td>O</td>
<td>54.54</td>
<td>47.39</td>
</tr>
<tr>
<td>Pb</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Total</td>
<td>100.00</td>
<td>100.00</td>
</tr>
</tbody>
</table>

2.3.1.3. Fourier transform infrared (FTIR) spectroscopy

FT-IR analysis was carried out to identify characteristic functional groups present in TAFP and the spectra of the adsorbent before and after adsorption of Pb(II) are shown in Fig. 2.4 (a) and 2.4(b). The broad and strong absorption peak at 3446 cm\(^{-1}\) indicated the presence of OH group [Rao et al., 2011a]. The sharp and intense peak at 1642 cm\(^{-1}\) was aroused due to stretching vibrations of C=C [Haque et al., 2008], while the band at 1429 cm\(^{-1}\) indicated the presence of \(-\text{COO}^-\) group [Rao et
al., 2011b]. The broad peak at 1122 cm\(^{-1}\) was due to C–O stretching vibrations [Wang et al., 2013]. The broad and short peak observed at 601 cm\(^{-1}\) might be due to M–O (metal-oxide) bond as metal oxides generally give absorption bands below 1000 cm\(^{-1}\) arising from inter-atomic vibrations [Anandan et al., 2011; Wang et al., 2013]. FTIR spectra of the adsorbent after adsorption of Pb(II) (Fig. 2.4(b)) showed shift in some of the absorption bands towards lower frequencies. Shifting of bands to lower frequencies indicates bond weakening while a shift to higher frequencies indicates an increase in bond strength [Dwivedi et al., 2011; Wang et al., 2013]. Shifting of the peaks was observed in the region close to 1642 cm\(^{-1}\), 1122 cm\(^{-1}\) and 601 cm\(^{-1}\). These peaks were shifted to 1629 cm\(^{-1}\), 1068 cm\(^{-1}\) and 587 cm\(^{-1}\) respectively. The disappearance of peak at 1429 cm\(^{-1}\) indicated the strong interaction of Pb(II) with COO group.
Fig 2.2: SEM micrograph of TAFP (a) before and (b) after adsorption of Pb(II) onto TAFP (Magnification: 5000 in both)
Fig 2.3: EDX spectra of TAFP (a) before and (b) after adsorption of Pb(II) onto TAFP.
Fig 2.4: FTIR spectra of TAFP (a) before and (b) after adsorption of Pb(II) onto TAFP
2.3.2. Effect of initial Pb(II) concentration and contact time

The effect of initial concentration of Pb(II) onto adsorption was studied in the concentration range of 10-100 mg.L\(^{-1}\) (Fig. 2.5). A usual phenomenon of increase in adsorption capacity with increase in initial Pb(II) concentration was observed which may be attributed to the increased concentration gradient between the bulk solution and adsorbent surface thereby lowering the resistance to mass transfer of Pb(II) from aqueous to solid phase [Aksu et al., 2001; Rao et al., 2007]. The maximum adsorption of Pb(II) at equilibrium was found to be 0.95, 1.92, 4.91, 5.90, 7.85, and 9.80 mg.g\(^{-1}\) at initial Pb(II) concentrations of 10, 20, 50, 60, 80, and 100 mg.g\(^{-1}\) respectively. The contact time required to reach equilibrium was found to be dependent on initial Pb(II) concentration up to 80 mg.L\(^{-1}\) and then became independent of concentration (Fig. 2.5). This was due to the fact that empty adsorbent sites adsorbed Pb(II) ions rapidly at lower concentration but at higher concentration adsorption of Pb(II) ions occurred by diffusion (slower step) into the inner sites of the adsorbent. The equilibrium time of 5-30 min investigated in the present work is much shorter than many other adsorbents reported earlier for Pb(II) adsorption (Table 2.3).
Table 2.3: Comparison of equilibrium time for the adsorption of Pb(II) onto various adsorbents

<table>
<thead>
<tr>
<th>Adsorbents</th>
<th>Equilibrium time (min)</th>
<th>Conc.(mg.L(^{-1}))</th>
<th>References</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coriander seed</td>
<td>60-120</td>
<td>50-70</td>
<td>Rao et al., 2012a</td>
</tr>
<tr>
<td>Green alga</td>
<td>60</td>
<td>10</td>
<td>Sari et al., 2008</td>
</tr>
<tr>
<td>Sugarcane bagasse</td>
<td>120</td>
<td>100</td>
<td>Hamza et al., 2013</td>
</tr>
<tr>
<td>Mentha piperita treated carbon</td>
<td>180</td>
<td>50</td>
<td>Ahmad et al., 2012</td>
</tr>
<tr>
<td>Penicillium oxalicum</td>
<td>120</td>
<td>100</td>
<td>Svecova et al., 2006</td>
</tr>
<tr>
<td>Spirodela polyrhiza(L)schleiden biomass</td>
<td>72</td>
<td>100</td>
<td>Meitei et al., 2013</td>
</tr>
<tr>
<td><em>Terminalia arjuna</em> fruit powder</td>
<td>5-30</td>
<td>10-100</td>
<td>Present study</td>
</tr>
</tbody>
</table>
Fig 2.5: Effect of contact time on the adsorption of Pb(II) onto TAFP at different concentration (conditions; pH = 4; adsorbent = 0.2g; temp = 30°C)
2.3.3. Effect of pH

The effect of pH on the adsorption of Pb(II) was investigated in the pH range of 1–10. The results are shown in Fig. 2.6. The % adsorption of Pb(II) was minimum at pH 2 and increased with increasing pH, reached maximum (96%) at pH 4 but beyond pH 4 the % adsorption decreased slowly up to pH 10. The effect of pH on the adsorption of Pb(II) can be explained on the basis of the initial pH (pHi) of the solution, final or equilibrium pH (pHf), surface charge of the adsorbent and the speciation of the metal ions in the solution [Pourreza et al., 2014]. The adsorbent surface (MOH) would be completely covered by H⁺ ions (MOH₂⁺) at lower pH because of the presence of excess of H⁺ ions but at higher pH hydroxide ions react with hydrous oxide to produce deprotonated oxide (MO⁻) as shown in the following reactions [Inan et al., 2010; Kumar et al., 2013].

\[
\begin{align*}
\text{MOH} + \text{H}^+ & \rightarrow \text{MOH}_2^+ \\
\text{MOH} + \text{H}^- & \rightarrow \text{MO}^- + \text{H}_2\text{O} \\
\text{MOH}^{2-} + 2\text{OH}^- & \rightarrow \text{MO}^- 
\end{align*}
\]

Therefore the adsorbent surface became positively charged at lower pH and negatively charged at higher pH. Fig. 2.6 showed that when initial pH (pHi) was adjusted to 1, pHf remained almost constant and the adsorption of Pb(II) was least (10%) because of the electrostatic repulsion between positively charged adsorbent surface and positively charged Pb²⁺ ions since adsorbent surface was protonated due to the presence of excess H⁺ ions [Zou et al., 2006; Rout et al., 2013]. When pH_i was adjusted to 2, the adsorption of Pb(II) increased to 42%, possibly due to a little less competition of Pb²⁺ with H⁺ ions. However, when pH_i was adjusted to 4, the pH_f value increased to 6 at equilibrium and at the same time, the adsorption of Pb(II) increased to maximum (96%) possibly as a result of the adsorption of Pb²⁺ ions along with H⁺ ions. When pH_i was further increased to 6, the pH_f remained unaltered (pH_f = 6.5) but adsorption of Pb(II) decreased slightly (95%). A similar trend continued when pH_i was further increased and adsorption of Pb(II) decreased to 84% and pH_f also reduced to a value of 7.5 at pH_i = 10 (Fig. 2.6). The decrease in the equilibrium pH (pH_f) and adsorption of Pb(II) at pH > 6 can be explained on the basis of Pb(II) speciation at different pH values. Pb(II) is present in the forms of Pb³⁺ (pH 2-4);
Pb(OH)\(^+\) (pH 4-6); Pb(OH)\(_2\) (pH 6-10); and Pb(OH)\(^3\) (pH 10-12) [Tan et al., 2008; Pourreza et al., 2014]. It can therefore be concluded that Pb(II) was adsorbed as Pb\(^{2+}\) ions up to pH 4 and above this pH Pb(II) was adsorbed in the form of various hydroxide Pb(II) species [Yongjie et al., 2009, Rao et al., 2012a] in the form of micro precipitation. Further, the formation of hydroxide species of Pb(II) started above pH 6 hence pH\(_i\) decreased due to excess H\(^+\) ions remaining in the solution.

\[
Pb^{2+} + H^+ + OH^- \rightarrow Pb(OH)^+ + H^+ \tag{6}
\]

\[
Pb^{2+} + 2H_2O \rightarrow Pb(OH)\_2 + 2H^+ \tag{7}
\]

The effect of electrolyte (KNO\(_3\)) on the adsorption of Pb(II) was also studied and it was found that adsorption was decreased in presence of 0.1 N KNO\(_3\) because of the competitive effect of K\(^+\) ions. The effect of electrolyte was more pronounced below pH 4 (Fig. 2.6) but at pH 4 and above, the adsorption of Pb(II) was not affected perhaps due to the fact that below pH 4 both H\(^+\) and K\(^+\) ions screen the electrostatic attraction between negatively charged adsorbent surface and positively charged Pb(II) ions but above pH 4 the effect of H\(^+\) ions (extremely low concentration) was reduced to much extent and hence adsorption of Pb(II) was not much influenced in presence of electrolyte alone. The pH\(_{pzc}\) (point of zero charge) of the adsorbent surface was found to be 4 (Fig. 2.7), indicating that adsorbent surface was positive at pH< 4; neutral at pH = 4 and negatively charged at pH > 4 [Rout et al., 2013].
Fig 2.6: Effect of pH and electrolyte on the adsorption of Pb(II) onto TAFP (conditions; adsorbent = 0.2g; Pb(II) = 50 mg.L⁻¹ temp = 30°C)

Fig 2.7: Point of zero charge
2.3.4. Effect of adsorbent dose

The effect of adsorbent dose on adsorption capacity and percent adsorption of Pb(II) by varying the dose of TAFP is shown in the Fig. 2.8. Percent adsorption increased while adsorption capacity, \( q_e \) (mg.g\(^{-1}\)) decreased when adsorbent dose increased from 0.1-1.0 g. This increase in the percent adsorption of Pb(II) might be due to the fact that on increasing the adsorbent dose, number of sites available for adsorption also increased [Baniamerian et al., 2009; Wang et al., 2013]. The maximum adsorption of Pb(II) was about 99.4% at 1g dose. The decrease in adsorption capacity with increasing adsorbent dose might be due to the fact that some of the adsorption sites were remain unsaturated during the adsorption process [Rao et al., 2007] or some of the adsorbent particles interact with each other to form aggregates which reduced the total surface area of the adsorbent [Yongjie et al., 2009; Rao et al., 2012b].
Fig 2.8: Effect of doses on the adsorption of Pb(II) onto TAFP

(Conditions; Pb(II) = 50 mg.L\(^{-1}\); pH = 4; temp = 30\(^{0}\)C)
2.3.5. Adsorption Isotherms

The equilibrium adsorption isotherm is fundamentally very crucial in designing adsorption systems. In general, an adsorption isotherm is an invaluable curve describing the phenomenon governing the retention (or release) or mobility of a substance from the aqueous phase to a solid-phase at a constant temperature and pH [Allen et al., 2004; Limousin et al., 2007; Foo et al., 2010]. The Langmuir, Freundlich, Temkin and Dubinin–Radushkevich (D–R) models were used to fit the experimental data for the adsorption of Pb(II) at 30, 40 and 50°C. The results obtained are reported in Table 2.4. In order to evaluate the fitness of the data, the values of the correlation coefficient ($R^2$), were evaluated for each model.

Langmuir adsorption isotherm describes quantitatively the formation of monolayer on the surface of the adsorbent. The model assumes uniform energies of adsorption onto the surface and no transmigration of adsorbate in the plane of the surface [Dada et al., 2012]. The linear form of the Langmuir model may be written as:

$$\frac{1}{q_e} = \frac{1}{q_m} \times \frac{1}{b} \times \frac{1}{C_e} + \frac{1}{q_m}$$

where $C_e$ is the equilibrium concentration of Pb(II) in the solution (mg.L$^{-1}$), $q_e$ is the amount of Pb(II) adsorbed per unit weight of adsorbent (mg.g$^{-1}$), $q_m$ is the amount of Pb(II) required to form a monolayer (mg.g$^{-1}$), or the maximum monolayer adsorption capacity, and $b$ is a constant related to the energy of adsorption (L.mg$^{-1}$). The values of $b$ and $q_m$ can be calculated from the slope and intercept of the linear plots of $1/q_e$ versus $1/C_e$ at different temperatures (Fig. 2.9). The coefficient $b$ in Langmuir equation is a measure of the stability of the complex formed between metal ions and adsorbent under specified experimental conditions [Ayranci et al., 2005; Rao et al., 2007]. The data obtained from the model were best fitted at 30°C as indicated by high correlation coefficient value ($R^2 = 0.999$) (Table 2.4).

Freundlich adsorption isotherm is commonly used to describe the adsorption characteristics for the heterogeneous surface [Hutson et al., 2000; Dada et al., 2012]. The linear form of the Freundlich model can be written as:
log \( q_e \) = log \( K_F \) + \( \frac{1}{n} \) log \( C_e \)  \hspace{1cm} (9)

Where, \( K_F \) and \( n \) are the Freundlich constants. The constant \( K_F (\text{mg g}^{-1})(\text{L mg}^{-1})^n \) is an approximate indicator of adsorption capacity, while \( 1/n \) is a function of the strength of adsorption in the adsorption process [Voudrias et al., 2002]. The data obtained from this model (Fig. 2.10) indicated that the values of \( K_F \) and \( n \) increased as the temperature increased from 30 to 50°C. The values of \( n \) lies between 1 and 10, indicated favourable adsorption [Goldberg, 2005]. The data also revealed that Freundlich isotherm was best obeyed at 30°C \( (R^2 = 0.999) \n
The Temkin isotherm contains a factor that explicitly taking into the account of adsorbent–adsorbate interactions. By ignoring the extremely low and large value of concentrations, the model assumes that heat of adsorption (function of temperature) of all molecules in the layer would decrease linearly rather than logarithmic with coverage [Dada et al., 2012]. The linear form of the Temkin equation can be written as:

\[
q_e = \left( \frac{RT}{b} \right) \ln A + \left( \frac{RT}{b} \right) \ln C_e
\]

Or,

\[
q_e = B \ln A + B \ln C_e
\]

\hspace{1cm} (10)

Where, \((RT/b) = B\), \( R \) is the universal gas constant \((\text{J mol}^{-1} \cdot \text{K}^{-1})\), \( T \) is the absolute temperature \((\text{K})\) and \( b \) is a constant. The quantities \( A \) \((\text{g L}^{-1})\) and \( B \) \((\text{J mol}^{-1})\) are Temkin constants related to the adsorption potential and the heat of adsorption, respectively. The values of \( A \) and \( B \) can be calculated from the slope and intercept of the plot of \( q_e \) versus \( \ln C_e \) (Fig. 2.11). The data obtained from the Temkin model (Table 4) indicated that this model was best fitted at 30 °C \( (R^2 = 0.991) \).

Dubinin–Radushkevich isotherm is generally applied to express the adsorption mechanism with a Gaussian energy distribution onto a heterogeneous surface [Dabrowski et al., 2001; Gunay et al., 2007, Dada et al., 2012]. The linear form of this equation is represented as:
\[ \ln q_e = \ln q_m - \beta \varepsilon^2 \]  

(12)

Where, \( \varepsilon \) is the Polanyi potential (J.mol\(^{-1}\)), \( q_m \) is the monolayer capacity (mol.g\(^{-1}\)), \( C_e \) is the equilibrium concentration (mol.L\(^{-1}\)) and \( \beta \) is a constant related to the adsorption energy (mol.K\(^{-1}\).mol\(^{-2}\)). The Polanyi potential \( \varepsilon \) and the mean free energy of adsorption \( E \) (kJ.mol\(^{-1}\)) can be calculated from the following equations;

\[ \varepsilon = RT \ln \left( 1 + \frac{1}{C_e} \right) \]  

(13)

And

\[ E = \frac{1}{(2\beta)^{1/2}} \]  

(14)

The D-R constants \( q_m \) and \( \beta \) were evaluated from the linear plots of \( \ln q_e \) versus \( \varepsilon^2 \) (Fig. 2.12). The constant \( \beta \) gives an idea about the mean free energy \( E \) (kJ.mol\(^{-1}\)) of adsorption. The values of \( E \) (kJ.mol\(^{-1}\)) obtained lies between 8 and 16 kJ mol\(^{-1}\) showed that the adsorption was chemical in nature [Rao et al., 2010]. The data listed in Table 4 indicated that all the above models gave a good fit to the experimental data obtained at 30 °C, with the \( R^2 \) values for all the models being greater than 0.99.
Table 2.4: Adsorption isotherm parameters at different temperatures

<table>
<thead>
<tr>
<th>Isotherm</th>
<th>Parameters</th>
<th>30°C</th>
<th>40°C</th>
<th>50°C</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Langmuir</strong></td>
<td>B (L mg⁻¹)</td>
<td>0.2743</td>
<td>1.0556</td>
<td>2.3115</td>
</tr>
<tr>
<td></td>
<td>qₘ (mg g⁻¹)</td>
<td>27.390</td>
<td>13.850</td>
<td>11.820</td>
</tr>
<tr>
<td></td>
<td>R²</td>
<td>0.9993</td>
<td>0.8795</td>
<td>0.9506</td>
</tr>
<tr>
<td><strong>Freundlich</strong></td>
<td>Kᵥ (mg g⁻¹)</td>
<td>5.8425</td>
<td>7.0372</td>
<td>8.3291</td>
</tr>
<tr>
<td></td>
<td>(L mg⁻¹)</td>
<td>1.3450</td>
<td>1.698</td>
<td>2.2230</td>
</tr>
<tr>
<td></td>
<td>R²</td>
<td>0.9994</td>
<td>0.9031</td>
<td>0.9766</td>
</tr>
<tr>
<td><strong>Temkin</strong></td>
<td>A (L g⁻¹)</td>
<td>3.105</td>
<td>5.873</td>
<td>14.546</td>
</tr>
<tr>
<td></td>
<td>B (J mol⁻¹)</td>
<td>5.264</td>
<td>4.108</td>
<td>3.1620</td>
</tr>
<tr>
<td></td>
<td>R²</td>
<td>0.9917</td>
<td>0.8481</td>
<td>0.9397</td>
</tr>
<tr>
<td><strong>D-R</strong></td>
<td>qₘ (mol g⁻¹)</td>
<td>2.967×10⁻³</td>
<td>1.324×10⁻³</td>
<td>0.562×10⁻³</td>
</tr>
<tr>
<td></td>
<td>β (mol k⁻² J⁻²)</td>
<td>4.896×10⁻⁹</td>
<td>3.608×10⁻⁹</td>
<td>2.441×10⁻⁹</td>
</tr>
<tr>
<td></td>
<td>E (kJ mol⁻¹)</td>
<td>10.106</td>
<td>11.772</td>
<td>14.312</td>
</tr>
<tr>
<td></td>
<td>R²</td>
<td>0.9996</td>
<td>0.9021</td>
<td>0.9704</td>
</tr>
</tbody>
</table>
Fig 2.9: Langmuir isotherm for the adsorption of Pb(II) onto TAFP at different temperature

Fig 2.10: Freundlich isotherm for the adsorption of Pb(II) onto TAFP at different temperature
Fig 2.11: Temkin isotherm for the adsorption of Pb(II) onto TAFP at different temperature

Fig 2.12: D-R isotherm for the adsorption of Pb(II) onto TAFP at different temperature
2.3.6. Adsorption thermodynamics

Thermodynamic studies were conducted because they can provide information on inherent energetic changes. The effect of temperature on the adsorption of Pb(II) ions was studied over the temperature range of 30–50°C. The thermodynamic parameters such as enthalpy change (ΔH°) and entropy change (ΔS°) were obtained from the Van’t Hoff equation [Rao et al., 2007].

\[
\log K_c = \frac{\Delta S^0}{2.303R} - \frac{\Delta H^0}{2.303RT}
\]  

(15)

ΔS° and ΔH° were calculated from the slope and intercept of linear plot of log Kc versus 1/T (Fig. 2.13). Equilibrium constant (Kc) was calculated from the following relationship [Rao et al., 2007].

\[
K_c = \frac{C_{Ac}}{C_e}
\]  

(16)

Where, C_{Ac} and C_e are the equilibrium concentration of Pb(II) ions on the adsorbent and in the solution, respectively. Standard Gibb’s free energy change (ΔG°) was then calculated from the equation.

\[
\Delta G^0 = -RT \ln K_c
\]  

(17)

Where, T is the absolute temperature (K) and R is the gas constant (J.mol^{-1}.K^{-1}). The values obtained for the various thermodynamic parameters are listed in Table 2.5. The positive value of ΔH° suggested endothermic nature of adsorption of Pb(II) on TAFP. The values of ΔG° were found to be negative at all temperatures which indicated that the process was spontaneous and spontaneity increased with increase in temperature as indicated by decreasing ΔG° values. The positive value of ΔS° suggested increasing randomness at the solid/liquid interface during the adsorption process.
Table 2.5: Thermodynamic parameters for the adsorption of Pb(II) on TAFP at different temperatures

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>$K_c$ (kJ mol$^{-1}$)</th>
<th>$\Delta G^\circ$ (kJ mol$^{-1}$)</th>
<th>$\Delta H^\circ$ (kJ mol$^{-1}$)</th>
<th>$\Delta S^\circ$ (kJ mol$^{-1}$ K$^{-1}$)</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>0.4067</td>
<td>-0.927</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>0.6150</td>
<td>-1.0376</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>40</td>
<td>0.9900</td>
<td>-1.1958</td>
<td>3.902</td>
<td>0.163</td>
<td>0.9977</td>
</tr>
<tr>
<td>50</td>
<td>1.6567</td>
<td>-1.3722</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Fig 2.13: Van’t Hoff plot
2.3.7. Adsorption kinetics

The evaluation of kinetic models is an important aspect for designing and optimization of water and wastewater treatment process. Adsorption kinetic models correlate the adsorbate uptake rate from bulk concentration of the adsorbate [Subha et al., 2009; Azarudeen et al., 2013]. To investigate the mechanism of adsorption and rate controlling steps, the kinetic data were analyzed using Pseudo-first order, Pseudo-second order and intra-particle diffusion models. A pseudo-first-order kinetic model as expressed by Lagergen [Li et al., 2013] can be written as:

\[
\log (q_e - q_t) = \log q_e - \left(\frac{K_1}{2.303}\right) x t
\]  

(18)

Where \( q_e \) and \( q_t \) are the amount of metal adsorbed (mg g\(^{-1}\)) at equilibrium and at time \( t \), respectively and \( K_1 \) is the pseudo-first order equilibrium rate constant (min\(^{-1}\)). A plot of \( \log(q_e - q_t) \) versus \( t \) gave straight line over a wide range of initial Pb(II) concentrations (Fig. 2.14). However values of \( q_e \) calculated (\( q_{e(cal)} \)) from this model differed appreciably from the values of \( q_e \) obtained experimentally (\( q_{e(exp)} \)), indicating that the adsorption process did not follow the pseudo first order rate equation (Table 6).

Pseudo-second-order adsorption rate equation [Ho et al., 1998] may be expressed as;

\[
\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \left(\frac{1}{q_e}\right) x t
\]  

(19)

Where, \( K_2 \) is the pseudo-second-order adsorption rate constant (g mg\(^{-1}\).min\(^{-1}\)). A plot of \( t/q_t \) versus \( t \) gave straight line for all experimental concentrations (Fig. 2.15). The values of \( q_e \) and \( K_2 \) were calculated from the slope and intercept, respectively. The initial adsorption rate \( h \) (mg.g\(^{-1}\).min\(^{-1}\)) was calculated from the relation [Ho et al., 2003].

\[
h = k_2 \times q_e^2
\]  

(20)
The values of $q_e$, $h$, $K_2$ and $R^2$ are listed in Table 2.6. When the values of $q_{e(\text{cal})}$ were compared with $q_{e(\text{exp})}$, it was found that both the values were in good agreement for all experimental concentrations. Moreover, the correlation coefficient ($R^2$) values were also found to be very close to 1, thus it can be concluded that pseudo-second order model was well fitted with the experimental data. It can also be seen from Table 6 that with the increase in initial concentration of Pb(II), the rate constant ($K_2$) decreased. A similar observation was also reported by earlier researchers [Yao et al., 2010].

Kinetic data were also analyzed using the Weber and Morris [Volesky et al., 2003] intra-particle diffusion model which can be expressed as

$$q_e = k_{id} \times t^{1/2} + I$$  \hspace{1cm} (21)

Where $K_{id}$ is the intra-particle diffusion rate constant (mg.g$^{-1}$.min$^{-1}$) and $I$ is the intercept (mg.g$^{-1}$) which gives an idea about the thickness of a boundary layer, $q_t$ is the amount of Pb(II) ions adsorbed (mg.g$^{-1}$) at time $t$ (min). Fig. 2.16 shows the plots of $q_t$ versus $t^{1/2}$ for different initial Pb(II) concentrations. The values of $K_{id}$, $I$ and $R^2$ are listed in Table 2.7. The $R^2$ values (between 0.8304 and 0.9739) suggested that adsorption of Pb(II) can be followed by intra-particle diffusion model. The deviation in the plots from origin for all concentrations indicated that intra-particle diffusion is not the only rate-controlling step but some other processes like film diffusion and pore diffusion were also involved in the adsorption process. The increase in intercept values with increase in concentration is indicative of increased boundary layer effect [Godhana et al., 2008].
Table 2.6: Pseudo-first order and pseudo-second order kinetics constants for the adsorption of Pb (II) on TAFP

<table>
<thead>
<tr>
<th>Concentration (mg L⁻¹)</th>
<th>qₑ(exp) (mg g⁻¹)</th>
<th>qₑ(cal) (mg g⁻¹)</th>
<th>K₁ (min⁻¹)</th>
<th>R²</th>
<th>qₑ(cal) (mg g⁻¹)</th>
<th>K₂ (g mg⁻¹ min⁻¹)</th>
<th>h (g mg⁻¹ min⁻¹)</th>
<th>R²</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>0.95</td>
<td>0.4268</td>
<td>0.7026</td>
<td>0.7312</td>
<td>0.95</td>
<td>13.078</td>
<td>011.905</td>
<td>0.9989</td>
</tr>
<tr>
<td>20</td>
<td>1.92</td>
<td>0.5253</td>
<td>1.0822</td>
<td>0.9176</td>
<td>1.92</td>
<td>15.674</td>
<td>058.166</td>
<td>0.9999</td>
</tr>
<tr>
<td>50</td>
<td>4.91</td>
<td>0.5385</td>
<td>0.4214</td>
<td>0.9332</td>
<td>4.92</td>
<td>04.005</td>
<td>097.093</td>
<td>1.0000</td>
</tr>
<tr>
<td>60</td>
<td>5.90</td>
<td>0.2378</td>
<td>0.1829</td>
<td>0.5987</td>
<td>5.89</td>
<td>05.130</td>
<td>178.571</td>
<td>1.0000</td>
</tr>
<tr>
<td>80</td>
<td>7.85</td>
<td>0.5623</td>
<td>0.1829</td>
<td>0.8971</td>
<td>7.86</td>
<td>02.078</td>
<td>128.206</td>
<td>1.0000</td>
</tr>
<tr>
<td>100</td>
<td>9.80</td>
<td>0.4991</td>
<td>0.1179</td>
<td>0.7353</td>
<td>9.79</td>
<td>01.533</td>
<td>147.059</td>
<td>1.0000</td>
</tr>
</tbody>
</table>

84
Table 2.7: Intra-particle diffusion parameters for the adsorption of Pb(II) on TAFP

<table>
<thead>
<tr>
<th>Concentration (mg L⁻¹)</th>
<th>$K_{id}$ (mg g⁻¹ min⁻¹)</th>
<th>I (mg g⁻¹)</th>
<th>R²</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>0.0751</td>
<td>0.7822</td>
<td>0.9739</td>
</tr>
<tr>
<td>20</td>
<td>0.0779</td>
<td>1.7554</td>
<td>0.9095</td>
</tr>
<tr>
<td>50</td>
<td>0.0604</td>
<td>4.6941</td>
<td>0.9174</td>
</tr>
<tr>
<td>60</td>
<td>0.0262</td>
<td>5.7795</td>
<td>0.8636</td>
</tr>
<tr>
<td>80</td>
<td>0.0428</td>
<td>7.6335</td>
<td>0.8304</td>
</tr>
<tr>
<td>100</td>
<td>0.0372</td>
<td>9.5851</td>
<td>0.9296</td>
</tr>
</tbody>
</table>
Fig 2.14: Pseudo-first order kinetics model (conditions; pH = 4; adsorbent = 0.2g; temp = 30°C)

Fig 2.15: Pseudo-second order kinetics model (conditions; pH = 4; adsorbent = 0.2g; temp = 30°C)
Fig 2.16: Intra-particle diffusion model (conditions; pH = 4; adsorbent = 0.2g; temp = 30°C)
2.3.8. Breakthrough capacity

Determination of breakthrough capacity is an important aspect in process design as it directly affects the feasibility and economics of the process [Gupta et al., 2001, Rao et al., 2012] Breakthrough capacity can be determined by making use of the concentration gradient between the solute adsorbed by the adsorbent and that remaining in the solution. The column is operational until the metal ions in the effluent start appearing and for practical purposes the working life of the column is over, called breakthrough point. The breakthrough curve (Fig. 2.17) indicated that 50 mL of Pb(II) solution could be passed through the column without detecting Pb(II) ions in the effluent. The breakthrough and exhaustive capacities were found to be 5 and 15 mg.g⁻¹ respectively.

2.3.9. Desorption studies

In order to explore the practical utility of the adsorbent, desorption of Pb(II) was carried out by column process. In this study, aqueous solution of HCl was used as the desorbing agent because of its economic feasibility [Kwon et al., 2012; Liu et al., 2014]. Desorption studies were carried out by using the spent column obtained from breakthrough experiment. Thus, in a breakthrough capacity experiment, it was found that a total of 7.13 mg of Pb(II) ions were retained when 250 mL of Pb(II) ion solution of concentration 50 mg.L⁻¹ was passed through the column. For desorbing Pb(II) ion from TAFP, 0.05M HCl solution was passed through this column and the effluent was collected in 10 mL fractions. A total of 6.0 mg Pb(II) ions (84%) was recovered upon elution within 30 mL of the effluent (Fig. 2.18).
Fig 2.17: Breakthrough curve (conditions; adsorbent = 0.5g; Pb(II) = 50 mg.L\(^{-1}\); flow rate = 1 mL.min\(^{-1}\))

Fig 2.18: Adsorption and desorption of Pb (II) by column process (conditions; adsorbent = 0.5g Pb(II) = 50 mg.L\(^{-1}\); flow rate = 1 mL.min\(^{-1}\))
2.4. CONCLUSIONS

*Terminalia arjuna* is a medicinal plant and is used in ayurveda, siddha and unani system of medicine. The adsorption properties of *Terminalia arjuna* have been explored using batch process. The adsorption of Pb(II) ions was pH-dependent with maximum adsorption (96%) occurring at pH 4. Presence of electrolyte (KNO₃) in the solution has negligible effect on the adsorption of Pb(II). Therefore it can be concluded that the maximum amount of Pb(II) can be removed from aqueous solution even in presence of electrolyte without any significant loss of adsorption capacity. Kinetic data showed that pseudo-second order rate equation was followed by the system since qₑ values calculated from the model were very close to qₑ determined experimentally. The fitting of the data in Langmuir, Freundlich, Temkin and D–R isotherms indicated that these models were best obeyed at 30°C as indicated by their correlation coefficient ($R^2$). The maximum monolayer adsorption capacity was found to be 27.39 mg g⁻¹. The magnitude of the mean free energy indicates that adsorption of Pb(II) occurred via chemisorption. The thermodynamic parameters $\Delta H^0$ and $\Delta G^0$ indicated that the adsorption process was endothermic and spontaneous. Adsorption was fast and equilibrium was reached within 30 min over a wide range of Pb(II) concentration. Breakthrough studies showed that with Pb(II) concentrations of 50 mg L⁻¹, 50 mL of the effluent could be passed through the column without detecting Pb(II) ions. Desorption studies were carried out by column method using 0.05M HCl as desorbing solution and it was found that 84% of Pb(II) could be recovered within 30 mL effluent.
References


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Iqbal, M.; Saeed, A.; Zafar, S.I., FTIR spectrophotometry, kinetics and adsorption isotherms modeling, ion exchange, and EDX analysis for understanding the


95


