

# EFFECT OF ADSORPTION CONDITIONS ON THE REMOVAL OF LEAD (II) USING SEWAGE SLUDGE AS ADSORBENT MATERIAL

\*Ahmed M. Fat'hi

Ahmed H. Ali

Environmental Engineering Department, College of Engineering, Mustansiriyah University, Baghdad, Iraq

Received 25/10/2021

Accepted in revised form 4/12/2021

Published 1/5/2022

**Abstract:** The introduction of more stringent legislation limiting the disposal and use of sewage sludge, combined with an increase in its production and the loss of previously recognized disposal routes, has sparked a search for alternate applications for sewage sludge. The conversion of sludge into adsorbents is one of the promising technologies give a more stable treatment of sludge. In a batch mode adsorption, adsorbent created by recycling sewage sludge was employed as a sustainable and low-cost material to remove pb (II) ions from manufactured polluted aqueous solutions. A laboratory test was done to determine the influence of a variety of operational parameters on Pb (II) adsorption, including the acidic function (pH), adsorbent dosage, agitation speed, agitation period, and initial concentration at room temperature. The maximum removal efficiency was 88.2 % at optimum factors pH=4, agitation time =250 rpm, agitation time=180 min, adsorbent dose=1.5 g and initial concentration=50 (mg/l). In this way sewage sludge, which pollutes the environment and is costly to treat, may be disposed of in a useful and economic way to achieve the lowest pb (II) concentration level.

**Keywords:** *lead(II), sewage sludge, adsorption, adsorption conditions, removal efficiency*

## 1. Introduction

With the fast expansion of industry in recent years, more and more harmful heavy metals have infiltrated the water body. [1,2]. Heavy metals

are metallic elements with densities of more than  $5\text{g/cm}^3$ . [3]. They are difficult to decompose, unlike other contaminants, and can accumulate through the food chain, posing significant human health hazards and ecological disruptions. Pollution from these metals is a global issue, however the severity and degrees of pollution vary by location. pb (II) is one of at least 20 elements classified as hazardous, and is released into the environment [4]. Pb has a long history of use by humans due to its malleability, low melting point, and resistance to corrosion. Nevertheless is poisonous and a hazard to humans and other creatures life [5]. It enters the water system either by rural runoff or by releases from sewage treatment plants and industrial plants. The major sources of Pb (II) include industrial manufacturing manners and associated emissions, mining operations, smelting, burning sources, and solid waste furnaces [6]. paint, batteries, and lead pipe used in water distribution systems are further sources [7]. Human exposure to Pb (II) can happen due to the Consumption of polluted food, polluted drink, as well as through

\*Corresponding Author: [alamdar\\_ah@yahoo.com](mailto:alamdar_ah@yahoo.com)

the breathing of contaminated dust particles and aerosols. Pb(II) poisoning impacts the kidneys, liver, heart, brain, skeleton, and neurological system in humans [8]. Pb (II) is also affecting the aquatic system. Certain populations of aquatic invertebrates are more susceptible than others. Invertebrate populations from contaminated regions, on the other hand, may be more tolerant than those from non-polluted places. High Pb (II) concentrations can impede adaption to low oxygen circumstances in other aquatic invertebrates. Adults and eggs are less vulnerable than young fish. Typical signs of Pb (II) poisoning in aquatic species include spinal deformities and tail blackening [9]. As a result, the World Health Organization (WHO) recommends safe Pb (II) limits of 0.01 mg/l for both drinking water and wastewater effluent. The US Environmental Protection Agency (US EPA) regulates Pb(II) levels in drinking water and wastewater effluent at 0.05 and 0.006 mg/l, respectively [10]. Conventional treatment ways are employed in the elimination of variable heavy metals by most productions [11]. These techniques, which include electro dialysis, electro flotation, chemical precipitation, ion-exchange, reverse osmosis, solvent extraction, and membrane separation, have drawbacks such as irregular metal ion removal, sludge formation, high reagent requirements, and significant cost implications [12]. Among all of the strategies listed above, adsorption is one of the most effective. it is easy and effective [13]. Activated carbon is common used as adsorbents for the removal of different heavy Metals. However due to their high production costs, researchers shifted their focus to the use of biomass and waste such as peanut shell, garden grass , green algae , ground coffee, sunflower leaves , egg shells, almond shell , orange peel , for the removal of metals from aqueous solutions[14]. Sewage sludge is an one of example of solid waste and,

simultaneously, is a major by-product of wastewater treatment plant operation[15]. Due to increasing quantities and carbonaceous nature of Sewage sludge conversion it to adsorbents might be a promising way, not merely to ecofriendly solve for the problems of secondary pollution that result from Improper disposal of sludge, but also to allow its reuse in water and wastewater treatment applications[16]. The aim of this study is to test the feasibility and efficacy of the adsorbent produced from sewage sludge recycling for the elimination of Pb (II) from simulated polluted water. To fulfil the study's goal, the investigation was carried out under a set of adsorption factors, including pH, agitation speed, contact duration, adsorbent dosage, and starting metal ion concentration, with respect to the effectiveness of metal removal.

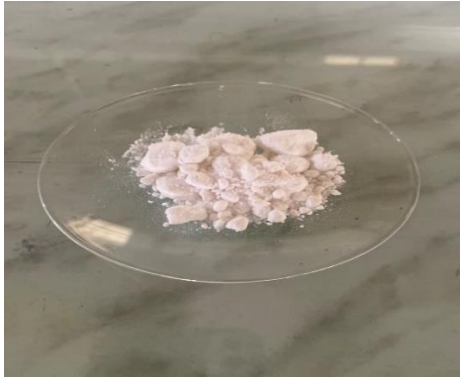
## **2. Materials and Experimental Work**

### **2.1. Materials**

#### *2.1.1. Preparation of Adsorbate*

A standard solution of Pb (II) with a concentration of 1000 mg/l was made using Pb (II) Dichloride salt. The salt was bring from commercial local market whose chemical formula, color, molecular weight, and CAS number are  $PbCl_2$ , white, 278.106 g/mole, and 12208-13-8, as shown in figure (1).The preparation process was achieved via dissolving A 1.333 g of this salt in 1 L of distilled water in laboratory temperature in volumetric flask and shake manually for 1 minute to ensure obtains perfect dissolving. The prepared solution used to prepare a 50 mg/l of Pb (II) as a stock solution, this concentration used for all experiments, Except for the experiments to examine the effect of initial metal ion concentration. The specific concentration of pb (II) solution and stock solution was found out

using atomic absorption spectrophotometer (EPA method 213.2) (AAS nova 400, German 2007) in science and technology ministry, environment and water researches directorate, water analysis Laboratory - Baghdad -Iraq.



**Figure 1.** Sample of commercial lead dichloride salt.

## 2.1.2 Adsorbent

### 2.1.2.1 Source of adsorbent

In this study, the important part of sludge was the solid fraction. The required sludge was obtained from the Rustamiya wastewater treatment plant, third expansion project - Baghdad, where samples were collected from drying basins at a depth of 5 cm, where the sludge is muddy and little moisture.

### 2.1.2.2 Preparation of adsorbent

The steps in the preparation of sewage sludge as follows:

- Drying of sewage sludge collected under sun rays for 14 day at ( $20 \pm 2^\circ\text{C}$ ) as shown in figure (2).
- Grinding the dried sludge by using a mill as shown in figure (3).
- Sieving the milled sludge with two (England-BS410) sieves of 1.18/0.6 mm respectively as shown in figure (4). The prepared sewage sludge as shown in figure (5). Storing in sealed plastic container to prevent reabsorption of moisture.



**Figure 2.** Bed of collected sewage sludge under sun-ray.



**Figure 3.** Mill Machine



**Figure 4.** Sieves 1.18mm and 0.6mm



**Figure 5.** Prepared Sewage sludge (adsorbent).

Table 1 shows the physical, chemical tests of prepared sewage sludge.

**Table 1.** Physical and chemical features for prepared sewage sludge

<u>Physical property</u>	<u>value</u>	<u>Chemical property</u>	<u>Value</u>
Actual $\rho$ , kg cm <sup>-3</sup>	1741.6	PH	7.5
Apparent $\rho$ , kg cm <sup>-3</sup>	609.9	Ash content %	12
Particle porosity	0.65	CECmeq/100g	51.15
Average particle diameter, mm	0.89		

These tests were conducted in laboratories of the Ministry of Industry and Minerals (Ibn Sina State Company).

## 2.2. Experimental Work:

The batch approach was used to measure the adsorption of Pb (II) on sewage sludge. In batch Adsorption tests, 100 ml of metal solution was added to 250-ml capped glass bottles containing 2 g of sewage sludge. The bottles were then put on a rotary shaker at 200 rpm for 3 hours to ensure proper mixing. At the end of the agitation period, the solutions are filtered using filtration paper (Whatman 150 mm filter paper). A sample of 20 ml was obtained from each container for analysis to determine the concentration of Pb (II) using (ASS) (EPA Method 213.2). Various parameters, including pH, agitation speed, adsorbent dose, contact time, and initial concentration, were tested. Equations (1) and (2) calculate the percentage of removal and adsorption capacity, respectively [17].

$$R = \frac{C_i - C_o}{C_i} \times 100 \quad (1)$$

$$q = \frac{V}{m} \times (C_{in} - C_f) \quad (2)$$

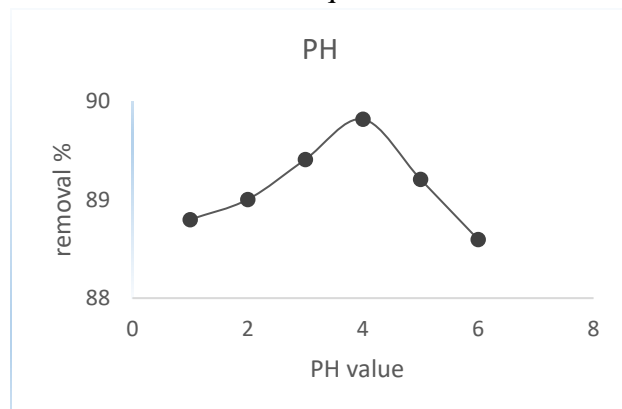
Where: R: is the percent removal of Pb(II) ions (-), q: is the capacity of adsorption by sewage sludge (mg/g),  $C_{in}$  and  $C_f$ : initial and final concentrations of Pb(II) ions respectively (mg/l), m: mass of sewage sludge used for adsorption (g) and V: volume of solution (l).

## 3. Results and Discussions

### 3.1. Influence of Acidic Function (PH)

The acidic function (pH) has been identified as the most significant variable affecting not just site dissociation but also the chemistry of heavy metal solutions, over and above, pH strongly influences hydrolysis, complexation by organic and/or inorganic ligands, redox reactions, and precipitation; moreover, pH strongly influences heavy metal speciation and adsorption availability [18]. To avoid the development of Pb(II) hydroxide precipitation, the effect of pH was examined at various PH values of the samples (1-6) [19]. The previously indicated pH values are determined by correcting the prepared solutions with 0.1 M (HCl, NaOH) for acidic and basic pH, as needed, using a portable digital PH meter (HANNA Instruments, Romania) , Other experimental variables include adsorbent dose, agitation speed, contact time, and starting concentration were held constant (2g, 200 rpm, 3 hours, 50mg/l) respectively. The experimental solution's temperature was within the range of room temperature (30-35 C). Figure (6) illustrates the relation between the solution's acidic function and the removal %. Pb (II) absorption was lower at very low pH (1-3), which is attributable to the fact that the adsorbent surface protonate and the adsorbent surface become positively charged, limiting metal ion access to the adsorbent surface due to repulsive interactions. Metal uptake increased as pH approached 4, with the maximum removal effectiveness of 89.81 percent. At this pH,

functional groups on the adsorbent surface deprotonate and the adsorbent surface becomes negatively charged (i.e., the number of OH-groups increases), therefore metal ions is able to attract to the adsorbent in this region of pH [20]. Furthermore, increased the pH leads a decrease of binding sites, reducing removal efficiency [21]. This result agreement with [22]. The ideal PH was set at 4 in subsequent trials.

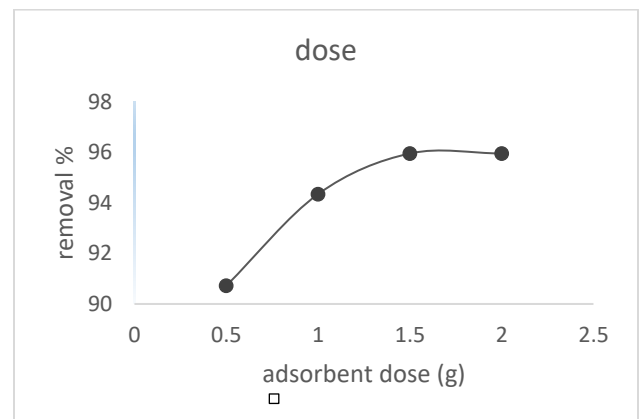


**Figure 6.** Influence of PH in %adsorption of pb (II)

### 3.2. Influence of Adsorbent Dose

Because of its impact on the amount of metal ions removed per unit mass of adsorbent, the adsorbent dosage is a significant parameter in the adsorption of metal ions from aqueous solution [23]. The influence of adsorbent dose was studied by variable dose of the adsorbent (0.5, 1, 1.5 and 2) g/100 ml. Other experimental parameters for example PH, agitation speed, contact time and initial concentration were kept fixed on (4, 200 rpm, 3 hours, 50 mg/l) respectively. The temperature of the experimental solution was within the range of room temperature (30-35 C). Figure (7) shows the percentage of adsorption of Pb (II) was increased from 90.72 to 95.96. This finding can be attributed to the fact that when the adsorbent dosage increases, more and greater surface area becomes available, exposing more active sites for Metal ion binding. After 1.5 g point, adsorption efficiency was steady due to no Vacancies in the

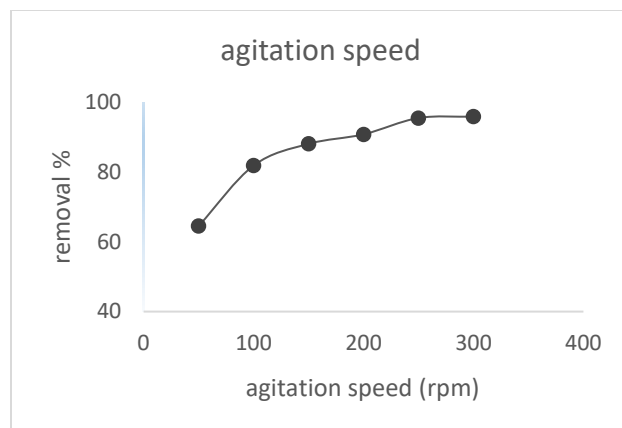
sludge to absorb more metal ions. A similar tendency was noticed and discussed in the literature for the influence of adsorbent dosage. [24]. The optimal sewage sludge dosage was fixed in subsequent experiments to be 1.5 g.



**Figure 7.** Influence of adsorbent dose in %adsorption of pb(II)

### 3.3. Influence of Agitation Speed

Effect of agitation speed was studied by varying the speeds (100,150,200,250,300) rpm. Other experimental variables include: PH, adsorbent dose, contact time and initial concentration were kept fixed on (4, 1.5g, 3 hours, 50 mg/l) respectively. The temperature of the experimental solution was within the range of room temperature (30-35 C). The efficiency value of Pb(II) increase from 64.52 to 95.90 % as shown in figure (8). It is apparent that increasing the mixing speed increases the removal efficiency, also we note that the difference in the efficiency of removal between 250-300 rpm is 0.2 %, this means that increasing the speed above 250 rpm gives a slight change in the percentage of removal therefore the optimum agitation speed was set at 250 rpm in subsequent experiments. The rise in efficiency is attributed to a rise in turbulence and, as a result, a reduction in the external mass transfer resistance thickness around the adsorbent particles as speed increases. [25].

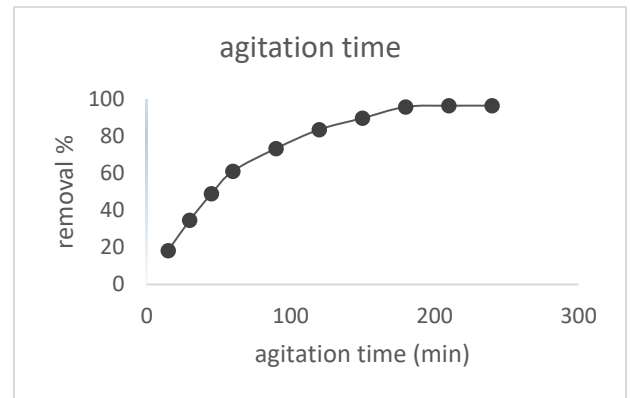


**Figure 8.** Influence of agitation speed in %adsorption of pb(II)

### 3.4. Influence of Agitation Time

The time-dependent function of Pb(II) adsorption was tested by changing the time from 15 to 240 minutes, Where 10 samples of Pb(II) were prepared and placed in the shaking device. In the first hour, four samples were taken every 15 min, in the second, third and fourth hours, samples were taken at a rate of every 30 min, Other experimental variables include: PH, adsorbent dose, agitation speed and initial concentration were kept fixed on (4, 1.5g, 250 rpm, 50 mg/l) respectively. The temperature of the experimental solution was within the range of room temperature (30-35 C). Figure (9) shows the adsorption movement of Pb (II) consisting of two stages: a fast initial stage and a slower second stage. The first stage showed rapid adsorption during the first 120 min where the removal efficiency reaches to 83.67%, after this time the rate of removal increased with increasing contact time, but slowly until an equilibrium was reached at 210 and 240 min where the removal efficiency was 96.53 %. In the beginning, a significant number of active places on the adsorbent are available, the metal ions occupied selectively the active sites on the adsorbents. As the sites become exhausted, the uptake rate depends on the rate of adsorbate transportation from the outside to inside the

adsorbent particles [26]. This result agreement with [27]. To save time, and because the removal difference between 180 and 210 minutes is simple, It is possible to choose 180 minute (3 hrs.) As a best agitation time in the next experiment.

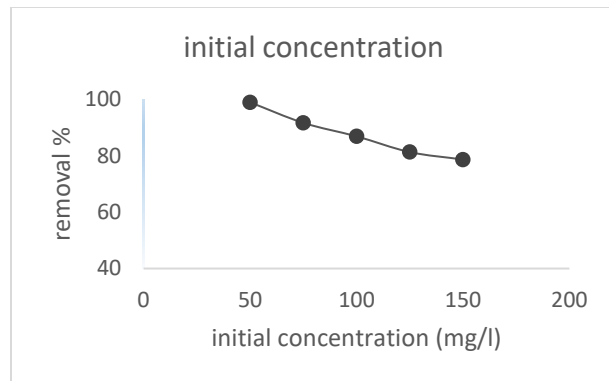


**Figure 9.** Influence of agitation time in %adsorption of Pb(II)

### 3.5. Influence of Initial Metal Ion Concentration

Within a range of (50, 75, 100,150) mg/l, the influence of initial concentration on the treatment process efficacy of solutions contaminated with Pb(II) was investigated with Other experimental variables include: PH, adsorbent dose, contact time and agitation speed were kept fixed on (4, 1.5g, 3 hrs. , 250 rpm) respectively. The temperature of the experimental solution was within the range of room temperature (30-35 C). The efficiency values declines from: 98.95 to 78..65 % for Pb (II) as the initial concentration increased from 50 to 150 mg/L as shown in figure (10). This is because the particles of adsorbent sample lacks binding sites for metal ion adsorption at higher concentrations. The following could be used to explain the influence of initial metal concentration: Metal ion adsorption involves more energy binding sites when the metal ion/adsorbent ratio is low. The greater energy active sites are saturated as the metal ion/adsorbent ratio increases (i.e., at greater starting concentration), and adsorption commences on minor energy binding sites, bring

about a drop in adsorption efficiency [28]. This result agreement with [29]. The best initial concentration was chosen as 50 mg/l.



**Figure 10.** Influence of Initial concentration in % adsorption of Pb(II)

#### 4. Conclusion

Prepared sewage sludge showed effective performance on Pb (II) adsorption. The pH has the most influence on the adsorption of this metal ion from aqueous solutions. The percentage elimination of Pb(II) ions by prepared adsorbent was increased with increasing agitation speed, agitation time and dose of adsorbent and after some point was steady while it was increased by increasing PH reaches to 4 then Started decreasing but this removal percentage decreased with increasing of ion metal concentration. The maximum removal efficiency was 98.95 % at optimum factors pH=4, agitation time =250 rpm, agitation time=3 hours, adsorbent dose=1.5 g and initial concentration=50 (mg/l). This study, which produced adsorbents from recycling sewage sludge, found a effective and sustainable mutual solution to a dual problem represented by sewage sludge disposal and lead ions removal.

#### Conflict of interest

The authors declare that this article does not cause a conflict of interest.

#### 5. References

1. Z. Wang, E. Nie, J. Li, Y. Zhao, X. Luo, and Z. Zheng.( 2011). "Carbons prepared from *Spartina alterniflora* and its anaerobically digested residue by H3PO4 activation: Characterization and adsorption of cadmium from aqueous solutions," *Journal of hazardous materials*, vol. 188, no. 1–3, pp. 29–36.
2. N. Zhou et al.( 2017). "Effect of phosphoric acid on the surface properties and Pb (II) adsorption mechanisms of hydrochars prepared from fresh banana peels," *Journal of cleaner production*, vol. 165, pp. 221–230.
3. 3.D. H. Nies.( 2000). "Microbial heavy-metal resistance," *Appl. Microbiol. Biotechnol.*, vol. 51, pp. 451–460.
4. O. B. Akpor and M. Muchie.( 2010). "Remediation of heavy metals in drinking water and wastewater treatment systems: processes and applications," *International Journal of Physical Sciences*, vol. 5, no. 12, pp. 1807–181.
5. L. Järup.( 2003). "Hazards of heavy metal contamination," *British medical bulletin*, vol. 68, no. 1, pp. 167–182.
6. D. Singh, A. Tiwari, and R. Gupta.( 2012 )."Phytoremediation of lead from wastewater using aquatic plants," *J Agric Technol*, vol. 8, no. 1, pp. 1–11.
7. J. Eisele, M. Sharma, S. J. G. Galer, J. Blichert-Toft, C. W. Devey, and A. W. Hofmann.( 2002 )."The role of sediment recycling in EM-1 inferred from Os, Pb, Hf, Nd, Sr isotope and trace element systematics of the Pitcairn hotspot," *Earth and Planetary Science Letters*, vol. 196, no. 3–4, pp. 197–212, doi: 10.1016/S0012-821X(01)00601-X.
8. S. J. S. Flora, G. Flora, and G. Saxena.( 2006). "Environmental occurrence, health effects and management of lead poisoning," in *Lead*, Elsevier, pp. 158–228.
9. M. Rashid, I. Ahmad, A. Muhammad, and M. U. Hassan, "Phytoremediation of battery industry effluent through aquatic macrophytes."

10. G. K. Kinuthia, V. Ngunjiri, D. Beti, R. Lugalia, A. Wangila, and L. Kamau.( 2020). “Levels of heavy metals in wastewater and soil samples from open drainage channels in Nairobi, Kenya: community health implication,” Scientific Reports, vol. 10, no. 1, pp. 1–13, doi: 10.1038/s41598-020-65359-5.
11. S. E. G. A. El-Asmy, and A. M. El-Nokrashy.(v).“Separation of chromium (III) and chromium (VI) from environmental water samples using eggshell sorbent,” Indian Journal of Science and Technology, vol. 1, no. 6, pp. 1–7.
12. K. Kadirvelu and C. Namasivayam.( 2003). “Activated carbon from coconut coirpith as metal adsorbent: adsorption of Cd (II) from aqueous solution,” Advances in Environmental Research, vol. 7, no. 2, pp. 471–478.
13. A. Abbas *et al.*( 2016). “Heavy metal removal from aqueous solution by advanced carbon nanotubes: critical review of adsorption applications,” Separation and Purification Technology, vol. 157, pp. 141–161.
14. D. Mehta, S. Mazumdar, and S. K. Singh.( 2015). “Magnetic adsorbents for the treatment of water/wastewater—a review,” Journal of Water Process Engineering, vol. 7, pp. 244–265.
15. S. Werle and S. Sobek.( 2019). “Gasification of sewage sludge within a circular economy perspective: a Polish case study,” Environmental Science and Pollution Research, vol. 26, no. 35, pp. 35422–35432.
16. X. Wang *et al.*( 2011). “Adsorption of Copper (II) onto activated carbons from sewage sludge by microwave-induced phosphoric acid and zinc chloride activation,” Desalination, vol. 278, no. 1–3, pp. 231–237.
17. B. H. Hameed, D. K. Mahmoud, and A. L. Ahmad.( 2008). “Equilibrium modeling and kinetic studies on the adsorption of basic dye by a low-cost adsorbent: Coconut (*Cocos nucifera*) bunch waste,” Journal of hazardous materials, vol. 158, no. 1, pp. 65–72.
18. K. László, E. Tombácz, and P. Kerepesi.( 2003).“Surface chemistry of nanoporous carbon and the effect of pH on adsorption from aqueous phenol and 2, 3, 4-trichlorophenol solutions,” Colloids and Surfaces A: Physicochemical and Engineering Aspects, vol. 230, no. 1–3, pp. 13–22.
19. H. Lalhruaitluanga, K. Jayaram, M. N. V Prasad, and K. K. Kumar.( 2010). “Lead (II) adsorption from aqueous solutions by raw and activated charcoals of *Melocanna baccifera* Roxburgh (bamboo)—a comparative study,” Journal of hazardous materials, vol. 175, no. 1–3, pp. 311–318.
20. A. Selatnia, M. Z. Bakhti, A. Madani, L. Kertous, and Y. Mansouri.( 2004). “Biosorption of Cd<sup>2+</sup> from aqueous solution by a NaOH-treated bacterial dead *Streptomyces rimosus* biomass,” Hydrometallurgy, vol. 75, no. 1–4, pp. 11–24.
21. M. A. Martín-Lara, F. Hernáinz, M. Calero, G. Blázquez, and G. Tenorio.( 2009). “Surface chemistry evaluation of some solid wastes from olive-oil industry used for lead removal from aqueous solutions,” Biochemical Engineering Journal, vol. 44, no. 2–3, pp. 151–159.
22. M. Otero, F. Rozada, A. Morán, L. F. Calvo, and A. I. García.( 2009 ).“Removal of heavy metals from aqueous solution by sewage sludge based sorbents: competitive effects,” Desalination, vol. 239, no. 1–3, pp. 46–57.
23. P. Miretzky, C. Muñoz, and A. Carrillo-Chávez.(2008). “Fluoride removal from aqueous solution by Ca-pretreated macrophyte biomass,” Environmental Chemistry, vol. 5, no. 1, pp. 68–72.
24. K. Jayaram, I. Murthy, H. Lalhruaitluanga, and M. N. V Prasad.( 2009). “Biosorption of lead from aqueous solution by seed powder of *Strychnos potatorum* L.,” Colloids and surfaces B: Biointerfaces, vol. 71, no. 2, pp. 248–254.
25. Y. Bulut and Z. Tez.( 2007). “Adsorption



- studies on ground shells of hazelnut and almond,”* Journal of hazardous materials, vol. 149, no. 1, pp. 35–41.
26. P. S. Kumar and R. Gayathri.( 2009). “*Adsorption of Pb<sup>2+</sup> ions from aqueous solutions onto bael tree leaf powder: isotherms, kinetics and thermodynamics study,*” J. Eng. Sci. Technol, vol. 4, no. 4, pp. 381–399.
27. X. Yang, G. Xu, and H. Yu.( 2019 ).“*Removal of lead from aqueous solutions by ferric activated sludge-based adsorbent derived from biological sludge,*” Arabian Journal of Chemistry, vol. 12, no. 8, pp. 4142–4149, doi: 10.1016/j.arabjc.2016.04.017.
28. Z. Al-Qodah.( 2006). “*Biosorption of heavy metal ions from aqueous solutions by activated sludge,*” Desalination, vol. 196, no. 1–3, pp. 164–176.
29. M. qin Jiang, X. ying Jin, X. Q. Lu, and Z. liang Chen.(2010). “*Adsorption of Pb(II), Cd(II), Ni(II) and Cu(II) onto natural kaolinite clay,*” Desalination, vol. 252, no. 1–3, pp. 33–39, doi: 10.1016/j.desal.2009.11.005.