



# Efficient Removal of Heavy Metals from Water Using Metal-Modified Biochar: A Study on Cadmium and Lead Adsorption.

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## *Abstract*

This study inscribes the issue related with heavy metal contamination in water bodies, specifically concentrates on cadmium (Cd) and lead (Pb), which are dominant pollutants owing to industrial pursuits. The research investigates the usage of metal-modified biochar, prompted from walnut shells, as an efficient adsorbent for the removal of Cd and Pb from aqueous aliquot blends. Experimental estimates reveal the pertinent impact of pH, initial concentration, contact time, and temperature on the adsorptive efficiency. Optimal circumstances for maximum removal rates were pre-examined at a pH of 5, with higher temperature levels incrementing the adsorptive efficacy. The adsorption trend was analysed using Langmuir and Freundlich isotherm models, which demonstrated stronger correlations and goodness of fitting in experimental data. Kinetic studies employing pseudo-first-order and pseudo-second-order models indicated that the pseudo-second-order model provided a better fit, suggesting a chemisorption process. Perhaps, metal-modified biochar emerges as a promising choice for Cd/Pb remediation and also deduce contamination in water, with potential scalability for real-world applications. This study explores the need for further experimental research to optimize operational parameters and render the practical feasibility of such improvised biochar in sustainable water treatment.

Keywords: Waste water; Heavy metals; Water treatment; biochar; Pollutants; removal; efficacy.

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## **1.Introduction**

The quality of ground water can be determined from the complex interactions of rock, soil, chemical constituents and gases within the aquifer. Heavy metal infiltration in ground water has raised significantly due to involvement of human activities includes use of pesticides, mining, effluents from textiles, battery industry, tanning industry and from petrochemical industry [1,2]. The significant rise in heavy metal concentration in ground water and water from other sources poses severe threat to both human and environment as these contaminants affects the ecosystem. Industrial expansion around the globe has contributed to the presence of contaminants, making the research on contaminants is essential to safeguard the water bodies[3]. There is also a urgent need to utilize the advanced analytical tools to efficiently identify and analyze the contaminants in the environment. Several toxic heavy metals found in waste water includes zinc (Zn), lead (Pb), mercury (Hg), chromium (Cr), copper (Cu), arsenic (As), nickel (Ni) and cadmium (Cd). In addition these heavy metals, other metals like iron (Fe), antimony (Sb), Cobalt (Co), Molybdenum (Mo) , boron (B) and manganese (Mn) were also present and there is a urgent need to remove these harmful pollutants from the water bodies[4,5]. To effectively address the seriousness of this situation, it is necessary to not only identify the heavy metals but also implement efficient techniques to remove them in order to protect both ecosystems and human health [6,7]. Recently, researchers are exploring advanced and innovative technologies for efficient waste water treatment includes chemical precipitation, adsorption, advanced filtration and membrane technologies [8]. These technologies not only detect the presence of heavy metals but also remediate heavy metal contamination in water bodies, ensuring safer and more sustainable environment to the society. Along with the application of advanced technologies, community awareness, stringent regulations, best industrial practices are considered to be vital to mitigate the heavy metal contaminants in the water bodies. Proper collaboration between industries and government are considered to be essential to discharge these pollutants along with implementation of effective waste water treatment measures [9,10].

Lead (Pb) and Cadmium (Cd) are found to be prominent pollutants on a global scale. These pollutants infiltrates in to the environment through manmade activities includes mining and electroplating [11]. Infiltration of the pollutants not only disrupts the ecological balance but also poses a severe threat to the human beings. Several researchers and organization has set a guideline value for arsenic and cadmium in drinking water as 10 $\mu$ g/L and 3 $\mu$ g/L respectively [14]. Due to the high toxic nature and safety thresholds, urgent care is needed to develop the cost-effective treatment technologies to address the arsenic and cadmium contamination in waste water. These cost-effective advanced treatments mitigate the environmental risks associated with these pollutants and safeguards the human health and ecosystems [12]. Among the various technologies adsorption is found to be economical and feasible option to remove the heavy metals in water effectively [13].

Biochar is a carbon- rich solid residue resulting from pyrolysis process in oxygen deficit environment when subjected to higher temperature [15]. Due to the availability of abundant raw materials, biochar has gained lot of attention and research in several applications, in particular to remove the heavy metals. The removal of heavy metals by means of biochar involves several interactions includes ion exchange, adsorption and precipitation between the oxygen functional groups and metal ions. These removal mechanisms may vary based on the differences in biomass feedstock and pyrolysis conditions. There is an urgent need to explore the multiple properties of biochar adsorption process. The deeper understanding of biochar is essential to utilize them for effective removal of heavy metals in the environments. The detailed analysis of biochar utilization to remove heavy metals on larger scale is essential to intricate the relationships and acts as sustainable solution. Qianqian Yin et al.,[22] explored the utilization of modified soybean straw biochars (Mg/BC, Al/BC, and Mg–Al/BC) to handle the water eutrophication issue by removing nitrogen and phosphorus pollutants. Individually, Mg–Al/BC, Al/BC, and Mg/BC showed high adsorption capacities for phosphorus (0.70 mg/g), nitrate (40.63 mg/g), and ammonia (74.47 mg/g), respectively. Mg–Al/BC demonstrated efficacy against all three pollutants simultaneously. Mg–Al variants acts as promising adsorbents for remedying eutrophic water contaminated with multiple pollutants. Weili Jiang et al.,[15] focused on using magnetic biochar derived from Suaeda biomass to remove oxytetracycline from wastewater. Biochar (800SBC), undergoes bimetal modification, forming nontoxic magnetic biochar primarily composed of ferrite. Mg-Fe@800SBC(1:1) exhibited a maximum oxytetracycline adsorption capacity of 82.83 mg/g according to the Sips L-F model. Liang T et al.,[23] used wheat straw modified biochar with manganese oxide and cerium to remove arsenic heavy metal in water. Characterization techniques revealed that the addition of these oxides significantly improved As(V) adsorption. Batch tests demonstrated MBC's adsorption followed pseudo-second order kinetics and the Langmuir equation, with a capacity of 108.88 mg As(V)/g at pH 5.0. Modified biochar showed superior performance across a pH range from 2.0 to 11.0. Yipeng Wang et al., [24] explored biochars from various pyrolysis methods and feedstocks

identifying their different properties and heavy metal adsorption characteristics. Higher Cu and Pb adsorption efficiency was achieved using biochar prepared using aerobic calcination. Adam Sochacki et al.,[25] used typha and spruce biochars to assess the adsorption characteristics of organic pollutants found in greywater. Different pyrolysis temperature was maintained i.e. (350°C and 600°C) to assess the temperature dependent biochar adsorption characteristics. The spruce biochar at 600°C showed superior sorption capacity, particularly for organics, with minimal desorption during dynamic tests. Molecular dynamics modeling highlighted hydrophobic interactions driving increased nitrate and boron sorption on spruce biochar (600°C). Organic pollutant adsorption was mainly governed by  $\pi$ - $\pi$  stacking, with hydrogen bonding playing a minor role on the biochar surface.

## 2. Materials and methods

Walnut shell biochar production involved subjecting 100-mesh walnut shell powder to a 2-hour pyrolysis process at 600 °C in a nitrogen-rich muffle furnace with a flow rate of 300 cm<sup>3</sup> min<sup>-1</sup>. Following production and subsequent washing with deionized water, the biochar underwent a 24-hour drying process in an oven at 75 °C. Subsequently, 5 grams of the dried biochar were introduced to an 80 mL iron nitrate solution (0.4 mol L<sup>-1</sup>) and stirred for 3 hours. The sample underwent 3 hours of sonication and then the solvent was evaporated at a constant temperature of 100 °C using a water bath, resulting in dry iron biochar. The dried material was ground and sifted through a 0.154 mm sieve before being calcined for 1 hour at 300 °C in a nitrogen environment. Concurrently, ammonium tetrathiomolybdate was added to a beaker containing 100 mL of deionized water and stirred for 0.5 hours to achieve homogeneity. A suitable quantity of the calcined biochar was added to the solution, and the pH was adjusted using either 0.1 M HNO<sub>3</sub> or NaOH. This suspension was stirred for 2 hours, followed by 1 hour of sonication, and subsequently sealed in a Teflon-lined autoclave where it was heated at 200 °C for 12 hours. The collected samples underwent rigorous cleaning with deionized water and ethyl alcohol. Afterward, they were freeze-dried, ground, and sifted through a 0.154 mm sieve. The resulting samples exhibited fixed mass ratios of calcined biochar to ammonium tetrathiomolybdate. After preparing the biochar, the biochar was impregnated with Fe<sub>2</sub>O<sub>3</sub> to improve the efficiency of water absorption process. Initially 0.3g of potassium ferrocyanide was mixed with biochar and subjected to magnetic stirring process for about 30 minutes. After stirring process, ferric chloride was added and makes the solution to turn blue color. The solution was subjected to centrifugation process to separate the solid residue from the solution. After extracting the solid sediment, the samples underwent thorough rinsing using distilled water and ethyl alcohol and then allowed to dry for about 12h at 60°C in oven.

## 3. Results and discussion

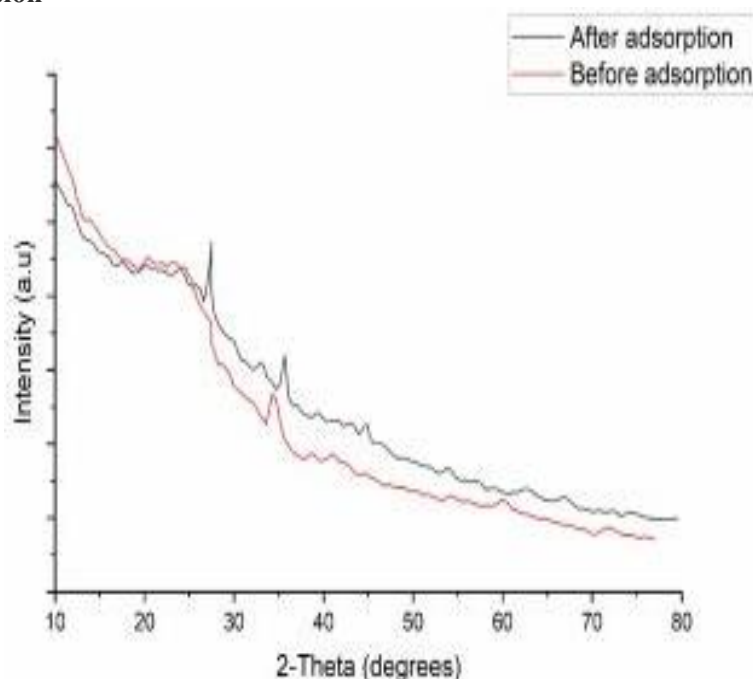


Figure 1a. XRD examination of metal modified biochar before/after heavy metal sorption

Crystalline based metallic acquisitions were of improvised biochar blends were examined using computer optimized X-ray diffractive device (XRD, Scintalytical, X'pert Program, India). XRD examination was utilized

to evaluate the crystalline state and metallic component type in sorbent samples. When compared with normal biochar, there are certain newly generated metal oxide patterns onto altered biochar. These metallic agglomerations favor in enhanced zeta potential along with surface coverage to certain degree. The metallic aggregates accounted to zeta increment from -25 to -10 mV. Certain double oxide Mg structural appearance can offer positive charge onto surface of such improvised biochar. Such positive amelioration (+26 mV) provide a pivotal role in heavy metals adsorptive process. The occlusion of iron metal positively incremented zeta charge value to about 40 mV. Perhaps, the peak obtained in XRD image also possessed maximum intensity for such observation.

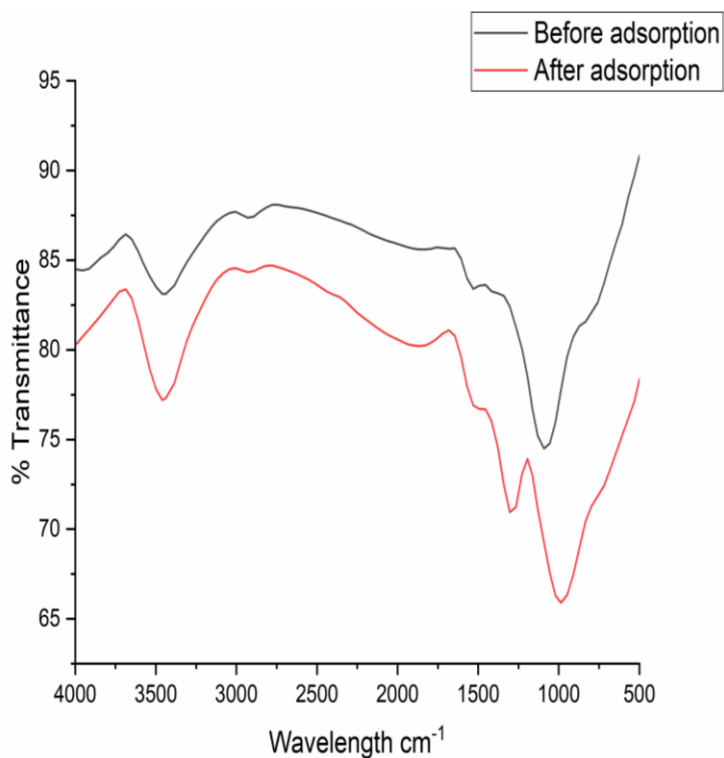


Figure 1b. FTIR impingement of metal modified biochar before and after heavy metal sorption

The surface functional acquisitions were estimated through Fourier transformed infrared spectroscopic device (FTIR, NICOLET 5800, Thermo Scientific Company, India). FTIR spectra portray that altered biochar components own extreme functional constituents. Whole of altered biochar contain O-H (along  $3545\text{cm}^{-1}$ ) stretches as well C-O stretching  $1000\text{cm}^{-1}$ . Aftermath sorption, peaks identified at  $1420\text{cm}^{-1}$  corresponds to metallic ions of iron and aluminium accustomed with positive charges which are incredibly attracting anionic counter parts. Such XRD report showcases the formation of co-ordination compound which is indefinitely confirmed after sorption. The iron compound observed after sorption of Fe metal is also a coordination structure. Hence, such metal altered biochar is extraneous for heavy metal adsorptive process.

### 3.1 Effect of pH

Figure 2 depicts adsorption efficiency of cadmium and lead using metal-modified biochar across different pH conditions. The optimum removal efficiency of metal modified biochar was observed at pH of 5 to 6. At pH of 5, the removal efficiency of both cadmium and lead was higher, indicates enhanced adsorption. The highest removal efficiency is for cadmium and lead was observed as 54% and 61% respectively at pH of 5. The least removal efficiency of cadmium and lead was observed as 28% and 33% at pH of 3. It is significant that the heavy metal removal efficiency by metal-modified biochar is mainly influence by pH. At low pH levels, adsorbents surface usually carry net positive charge due to protonation of functional groups. This positive charge might repel the cationic heavy metals like  $\text{Cd}^{2+}$  and  $\text{Pb}^{2+}$  and it results in few metal ion adsorption. At low pH, a higher concentration of hydrogen ions  $\text{H}^+$  is present in solution. These  $\text{H}^+$  ions compete with metal ions for adsorption sites on the adsorbent. This competition diminishes the available sites for metal adsorption, resulting in reduced adsorption efficiency. The higher adsorption rate at pH of 5 is may be due to surface of the adsorbent

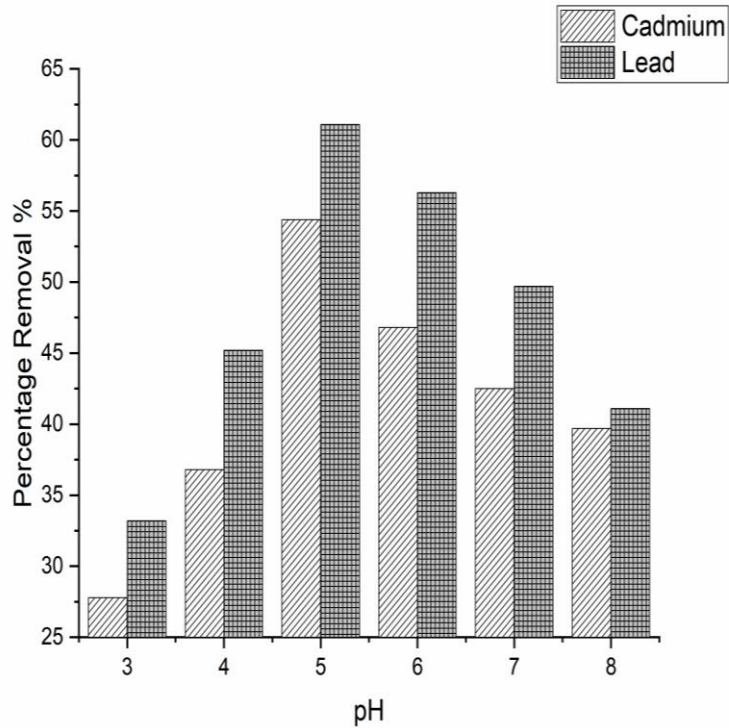


Figure 2. Effect of pH for solution of Pb and Cd

material have optimal charge that improves the affinity of heavy metal ions. In addition, at pH 5, heavy metal ions mostly tend to be less soluble and are often present in forms that are susceptible to the adsorption. Optimizing pH to a certain range, improves the adsorption process due to favourable conditions of interaction between heavy metal ions and adsorbent.

### 3.2 Effect of Initial Concentration

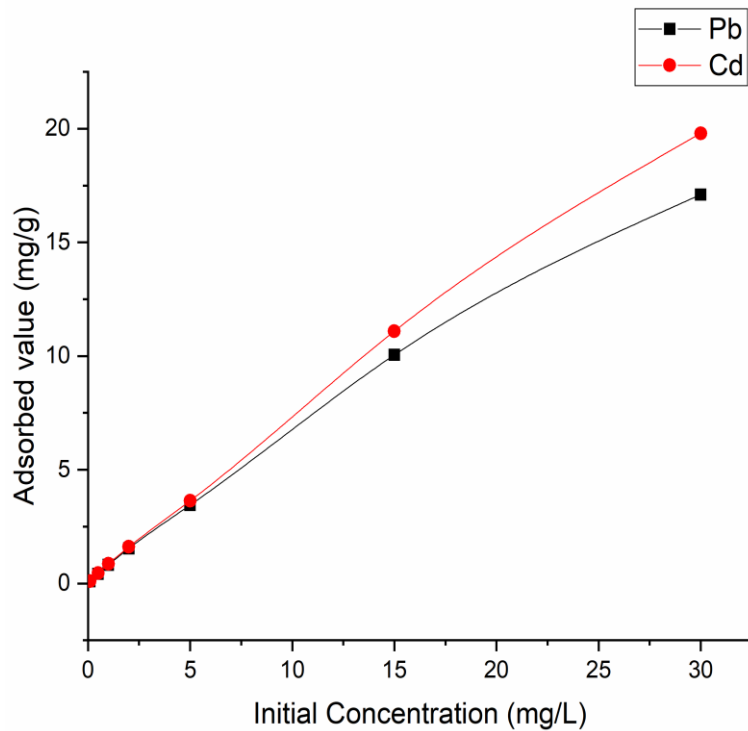


Figure 3: Effect of initial concentration and Adsorbed value for Cd and Pb heavy metal.

Figure 3 depicts the initial concentration and adsorption efficiency of cadmium and lead. It is seen that increase in initial concentration of lead from 0.1mg/L to 30mg/L increases the equilibrium adsorption capacity. As the initial concentration increases,  $q_e$  increases from 0.093 mg/g to 19.8 mg/g. Similar trend was observed in cadmium heavy metal adsorption capacity, as the initial concentration increases,  $q_e$  of lead increases from 0.091 mg/g to 17.1 mg/g. It is clearly evident that both cadmium and lead exhibit increased adsorption on to the metal modified biochar adsorbent as the initial concentration of metal ions increases. This is mainly due to high initial concentrations provide more driving force for adsorption on available sites on the biochar surface. The increasing trend of  $q_e$  becomes saturated at higher concentrations, this signifies that adsorption sites becomes gradually saturated on the surface area of biochar at higher metal ion concentrations, leading to reduced incremental increase in  $q_e$ . At lower initial concentration, the adsorption sites might not be fully occupied allowing for increase in  $q_e$  with increase in initial concentration values. At higher concentration, the available surface area on metal modified biochar is limited, leads to saturation effect.

### 3.3 Effect of Contact time

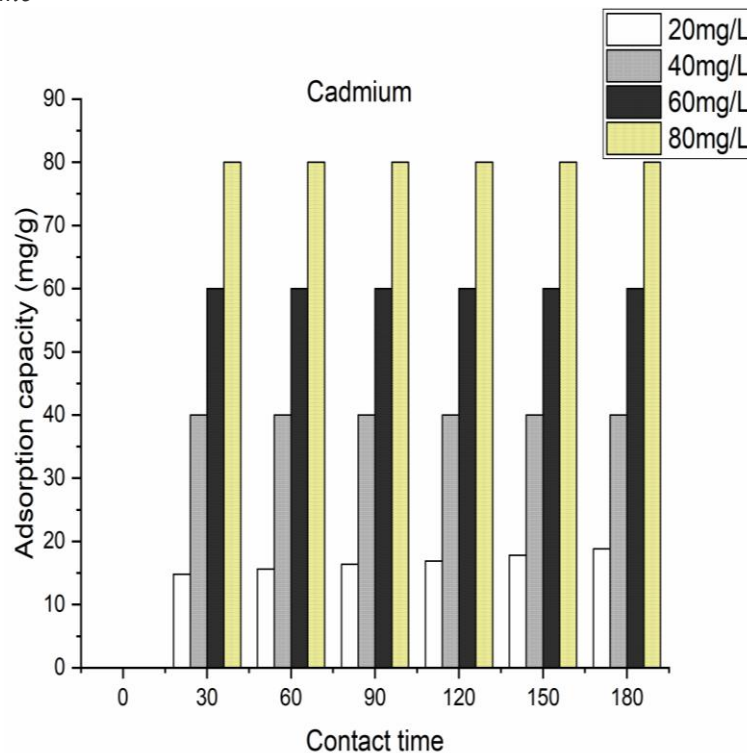


Figure 4. Effect of contact time and adsorption capacity for Cd heavy metal.

Figure 4 depicts the relationship between the contact time, different concentration of metal ions and cadmium adsorption capacity. As the contact time increases, there is a steady increase in amount of cadmium adsorbed ( $q_e$ ) for the different adsorbent dosages ranging from 20 mg/l to 80 mg/l. It is seen, increase of contact time, increases the adsorption of heavy metals, but the rate of increase of  $q_e$  shows to be slow down as the contact time exceeds from 120 to 180 mins. As the contact time increases, the active surface area of biochar becomes accessible, allowing more cadmium adsorption on lower adsorbent dosage. Whereas for higher adsorbent dosage, the active sites of adsorbent becomes occupied to less adsorption of heavy metals. The slower rate of increase in  $q_e$  was observed beyond 120 mins indicates saturation point was achieved, shows considerable portion of available sites have been occupied, resulting in reduced capacity of adsorption on biochar. Similar to cadmium, at the beginning, there's no adsorption observed for lead across all initial concentrations, indicating the absence of equilibrium at the beginning of the experiment. Figure 4 depicts the dependency of contact time in lead adsorption on the metal modified adsorbent surface. Similar to cadmium, increase of contact time increases the lead adsorption until it reaches the equilibrium.

### 3.4 Effect of temperature

The effect of temperature on the equilibrium adsorption capacity of cadmium and lead were shown in the figure 5. As the temperature increases from 25°C to 55°C, the equilibrium adsorption of cadmium increases from 45%

to 79%. This signifies the considerable increase in adsorption capacity with increase of temperature. Higher temperature provides additional energy, facilitates improved adsorbate and adsorbent interactions, and allows more cadmium and lead ions to bind on the biochar surface. Higher temperature activates the adsorption sites on the biochar, allows the available surface area for adsorption of cadmium ions.

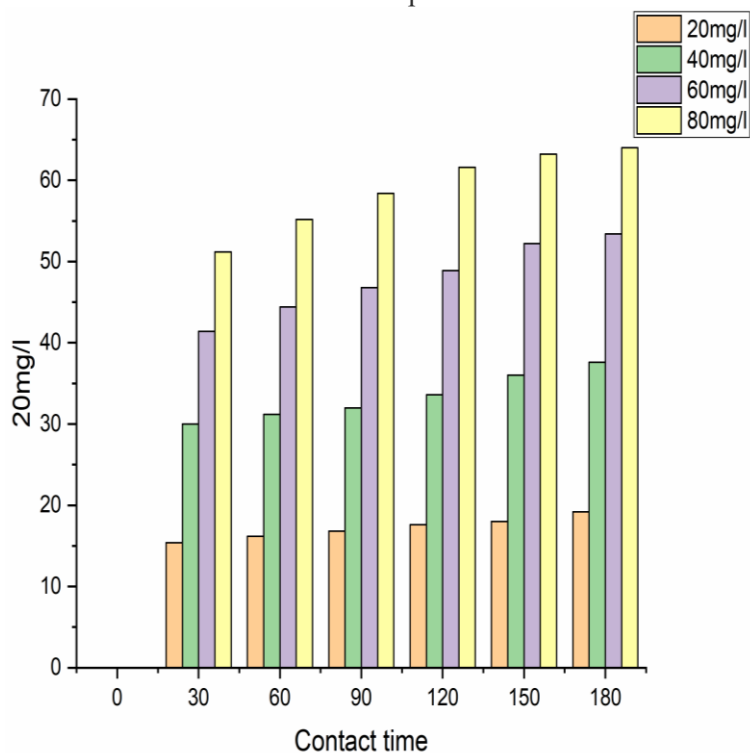


Figure 5. Effect of contact time and adsorption capacity for Pb heavy metal.

Similar to cadmium, increase of temperature increase the adsorption capacity of lead from 56% to 82%. There is a substantial increase in adsorption capacity with increasing temperature. This temperature dependent rise in adsorption for both cadmium and lead signifies the improved adsorption kinetics due to increased thermal energy.

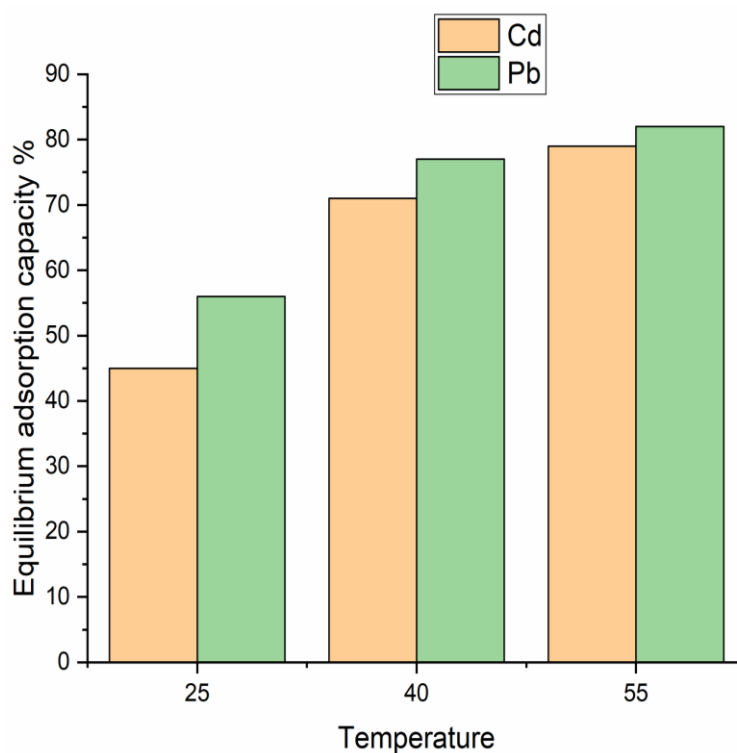


Figure 6: Effect of temperature and adsorption capacity of cadmium and lead heavy metal  
Adsorption isotherm

The Langmuir and Freundlich isotherm models utilization signifies the adsorption behavior of cadmium and lead heavy metal on metal modified biochar surfaces. Table 1 shows the values of different parameters of Langmuir and Freundlich isotherm models. The  $R^2$  values for both Cd and Pb adsorption onto the adsorbent surface using the Langmuir model indicate a strong correlation between the experimental and predicted data points. These high  $R^2$  values (0.935 for Cd and 0.975 for Pb) suggest that the Langmuir model fits the experimental data reasonably well, signifying a good conformity of the adsorption data to the Langmuir isotherm equation. The  $K_L$  values obtained for Cd (5.941 L/mg) and Pb (5.32 L/mg) imply the affinity of the adsorbent towards these metals. The lower  $K_L$  for Pb could suggest a slightly lower affinity compared to Cd. Generally,  $K_L$  reflects the strength of interaction between the adsorbate and the adsorbent surface, where higher values denote stronger binding. The  $q_m$  values for Cd (21.1 mg/g) and Pb (25.2 mg/g) illustrate the maximum adsorption capacity of the adsorbent for these metals. A higher  $q_m$  for Pb indicates that the adsorbent has a higher potential to adsorb Pb compared to Cd, aligning with its higher  $K_L$  value. The  $R^2$  values obtained for the Freundlich model are substantially higher than for the Langmuir model, indicating an excellent fit of the experimental data to the Freundlich equation. The values (0.987 for Cd and 0.995 for Pb) affirm the suitability of the Freundlich model in describing the adsorption process for both metals. The  $K_F$  values obtained for Cd (18.7) and Pb (16.1) signify the adsorption capacity of the adsorbent for these metals. The higher  $K_F$  for Cd implies a relatively higher adsorption capacity compared to Pb according to the Freundlich model.

Table 1: Langmuir and Freundlich isotherm model

| Isothermal model | Parameters            | Cadmium (Cd) | Lead (Pb) |
|------------------|-----------------------|--------------|-----------|
| Langmuir model   | R <sup>2</sup>        | 0.935        | 0.975     |
|                  | K <sub>L</sub>        | 5.941        | 5.32      |
|                  | q <sub>m</sub> (mg/g) | 21.1         | 25.2      |
| Freundlich Model | R <sup>2</sup>        | 0.987        | 0.995     |
|                  | K <sub>F</sub>        | 18.7         | 16.1      |
|                  | 1/n                   | 0.489        | 0.556     |

Table 2. Pseudo first order and second order kinetic model

| Kinetic Model       | Parameters            | Cadmium (Cd) | Lead (Pb) |
|---------------------|-----------------------|--------------|-----------|
| Pseudo first order  | R <sup>2</sup>        | 0.932        | 0.976     |
|                     | K <sub>1</sub>        | 0.345        | 1.12      |
|                     | q <sub>e</sub> (mg/g) | 13.1         | 14.7      |
| Pseudo second order | R <sup>2</sup>        | 0.955        | 0.987     |
|                     | K <sub>2</sub>        | 0.234        | 0.572     |
|                     | q <sub>e</sub> (mg/g) | 12.3         | 15.7      |

The pseudo-first-order and pseudo-second-order kinetic models are important to understand the rate and mechanisms of cadmium and lead heavy metal removal during adsorption processes and its parameters are shown in Table 2. The  $R^2$  values for both Cd (0.932) and Pb (0.976) removal using the pseudo-first-order model indicate a relatively good fit of the experimental data to the model. These values suggest a moderate to strong correlation between the experimental and predicted data points, implying that the pseudo-first-order model reasonably describes the kinetics of Cd and Pb removal. The  $K_1$  values obtained for Cd (0.345 min<sup>-1</sup>) and Pb (1.12 min<sup>-1</sup>) represent the rate constants of the pseudo-first-order kinetic model. The higher  $K_1$  value for Pb indicates a faster rate of adsorption compared to Cd. A higher  $K_1$  signifies a quicker attainment of equilibrium or a faster initial adsorption rate for Pb compared to Cd. The calculated  $q_e$  values for Cd (13.1 mg/g) and Pb (14.7 mg/g) represent the adsorption capacity at equilibrium predicted by the pseudo-first-order model. The higher  $q_e$  value for Pb indicates a higher predicted equilibrium adsorption capacity compared to Cd, aligning with the higher rate constant ( $K_1$ ) observed for Pb. The  $R^2$  values for the pseudo-second-order model for both Cd (0.955) and Pb (0.987) removal signify a good fit of the experimental data to the model. These higher  $R^2$  values compared to the pseudo-first-order model suggest a better fit of the pseudo-second-order model to the kinetic data. The  $K_2$  values obtained for Cd (0.234 g/mg·min) and Pb (0.572 g/mg·min) represent the rate constants of the pseudo-second-order kinetic model. Similar to the pseudo-first-order model, the higher  $K_2$  value for Pb indicates a faster rate of adsorption compared to Cd using the pseudo-second-order model. The  $q_e$  values



for Cd (12.3 mg/g) and Pb (15.7 mg/g) represent the predicted equilibrium adsorption capacity according to the pseudo-second-order model. The higher  $q_e$  value for Pb aligns with the pseudo-first-order results, suggesting a higher predicted equilibrium adsorption capacity compared to Cd using the pseudo-second-order kinetic model.

#### 4. Conclusion

The suggested research definitely promises the criticality in addressing heavy metal contamination in water bodies, specifically Cd and Pb, provoked by industrial activities. Metal-modified biochar emerges as a promising substitute owing to its abundant availability and enhanced efficacy in heavy metal sorption. Resultant outcomes designate the intricate relationship between pH, initial concentration, contact time, and temperature with the adsorptive efficiency of Cd and Pb on the modified biochar surface. The findings reveal optimal conditions for higher removal efficiencies, emphasizing pH 5 as the optimal point for maximum adsorption of both metals. Additionally, the research explores the equilibrium adsorption capacities affected by initial concentrations and contact times. Notably, a saturation effect is observed at higher concentrations, limiting further adsorption. Temperature elevation notably enhances adsorption capacity for both Cd and Pb, indicating improved kinetics at higher temperatures. The Langmuir and Freundlich isotherm models describe the adsorption behavior of Cd and Pb on metal-modified biochar surfaces. The models' parameters, namely  $R^2$ , KL,  $q_m$ , KF, and  $1/n$ , illustrate strong correlations, indicating favorable fitting of the experimental data to the models. Moreover, the pseudo-first-order and pseudo-second-order kinetic models elucidate the kinetics and equilibrium adsorption capacities for Cd and Pb. While both models display a good fit, the pseudo-second-order model showcases higher  $R^2$  values, suggesting a better fit for the kinetic data.

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