The utilization of date palm waste as an efficient adsorbent for the elimination of heavy metals from polluted water

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Research Article

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Abstract

This study focuses on the removal of heavy metal ions, such as cadmium (Cd^{2+}), lead (Pb^{2+}), Chromium (Cr^{6+}), and Zinc (Zn^{2+}) from water. Metals, which are often present in water, can have various origins i.e. industrial emissions, mining, melting, corrosion, municipal and industrial waste and agrochemicals. These metals have the potential to cause adverse effects on human health. The study employed activated carbon derived from date palm waste as a means of removing heavy metals. The activated nanoparticles were characterized using various analytical techniques, including SEM, ICP, XRD, BET, Raman, and FTIR analysis. Batch studies were conducted to optimize the metal ion adsorption onto the activated carbon of date palm waste. The adsorption process was evaluated using isotherm models and reaction kinetics under various conditions, including contact time, dosage, pH, and initial concentration. The findings of this study revealed that the prepared activated carbon from date palm waste effectively removed up to 85% of Cr^{6+}, Pb^{2+}, Cd^{2+}, and Zn^{2+} ions from water. This study highlights the potential of using agricultural waste, such as date palm waste, for the production of effective adsorbents for heavy metal removal.

1. Introduction

Access to clean and safe drinking water is crucial for the survival and well-being of all living creatures, and it is found in various forms such as glaciers, rivers, and groundwater. Most Earth water is non-drinkable due to high amounts of salts and other pollutants (McBride, Lake, & Varanasi, 2023). Water, air, and soil contamination are primarily caused by industrial development and the lack of innovation in finding cleaner and more sustainable production methods. The exploitation of natural resources and the release of harmful chemicals and pollutants into the environment have led to a serious deterioration of our planet's natural systems. Unfortunately, nearly two billion people worldwide use contaminated water, which can pose significant health risks. It is estimated that more than 80% of the wastewater produced by human activities is released back into the environment without treatment, exacerbating the problem (Bijekar et al., 2022). Efforts are being made to improve access to safe drinking water and to address water pollution issues globally. These efforts include investments in water infrastructure, the reduction of pollution from various sources such as industry and agriculture, improving wastewater treatment, and promoting sustainable water-use practices (Baby & Hussein, 2020). Without a concerted effort to implement more environmentally friendly practices, contamination will continue to be a major problem that threatens both human health and existence of fauna and flora including aquatic creatures. Therefore, it is essential that businesses and industries prioritize sustainable practices and innovation in order to mitigate the harmful effects of their operations on the environment (Baby, Saifullah, & Hussein, 2019). Heavy metals and other toxins released by metal mining, pesticide, paper, fertilizer, and chemical industries may cause a significant negative impact on the environment. Pollutants can harm the environment and human health by contaminating soil, water, and air for extended durations (Abel Uche Augustine, 2019). Overall, heavy metal toxicity is a serious health concern and it is important to minimize exposure to these harmful substances as much as possible (Snigdha Singh & Indra Jeet Chaudhary,

2019). Methods like ion exchange, extraction, foam flotation, chemical precipitation, activated carbon adsorption, and coagulation can remove heavy metals from water. These methods work by different mechanisms to separate the metals from the water, depending on their physical and chemical properties (Baby & Hussein, 2020). Although these techniques are working, however, they are expensive with limited applications; for example, the precipitation method produces a large amount of mud, electrolytic and membrane processes consume a large amount of energy (Renu, Agarwal, & Singh, 2017; Shrestha, Kour, & Ghimire, 2016; Ziarati P & Kermanshah A, 2015). Using agricultural waste as an adsorbent to remove toxic metals from polluted water through adsorption is a prevalent method. This method purifies water by binding metal ions to agricultural waste's surface (Taralgatti, 2016). Cellulose and lignin are chemical components found in agricultural waste that exhibit high efficacy in removing heavy metals from contaminated water. Functional groups like carboxyl, alcohols, aldehydes, ketones, and ethers contribute to this phenomenon. These functional groups enable the formation of complexes with heavy metal ions present in the water, resulting in their removal (Kamar, Nechifor, Mohammed, Albu, & Craciun, 2015). Activated carbon is ideal for removing heavy metals from water due to its large surface area, porous structure, and versatile pollutant adsorption capabilities (Muhammad, Yahya, Abdul Khalil, Marwan, & Albadn, 2023). It is widely used in water treatment plants and industrial processes for this purpose. This research aims to assess the effectiveness of chemically activated carbon derived from date palm waste in purifying contaminated water by removing heavy metals. Despite being abundant in by-products, the date palm tree is undervalued. The activated carbon was produced from date palm petioles through chemical activation utilizing H_3PO_4 at varying temperatures. The resulting materials underwent multiple analyses, including nitrogen sorption, scanning electron microscopy, X-ray analysis, and Raman spectroscopy, to determine their physico-chemical and surface properties.

2. Materials and Methods

2.1. Chemicals

Phosphoric acid (H_3PO_4) and metal ion salts ($ZnCl_2$, $CdSO_4$, $Pb(NO_3)_2$, Na_2CrO_4) were employed for creating standard solutions. The solutions were of analytical grade. Additionally, 0.1N NaOH and HCl solutions, along with deionized water, were utilized.

2.2. Instrumentation

The characterization of parent and prepared material was realized by Raman spectrometer, WiTech, Ulm, Germany, X-Ray Diffraction Spectrometer, Shimadzu, Kyoto, Japan, FT-IR spectrometer Perkin-Elmer 100 series Waltham, MA, USA, Field Emission Scanning Electron Microscope (FESEM) JOEL JSM-6400, Tokyo, Japan, and an Inductively Coupled Plasma-Optical Emission Spectrometer (ICP-OES) Optima 2100 DV, Perkin Elmer, Waltham, MA, USA.

2.3. Methodology

2.3.1. Pretreatment of samples

The samples of date palm waste were collected from agriculture farms located in Khairpur, Pakistan. The collected waste was washed thoroughly with deionized water to eliminate any impurities such as dust. Afterward, the material was subjected to dry in an oven at 70 ^{0}C to remove any water content. The desiccated substance was pulverized into a fine powder, followed by activation by phosphoric acid using a standard protocol described below.

2.3.2. Activation of the sample

Experiment involved treating 20g of washed and dried date palm waste with varying concentrations of phosphoric acid (5%, 10%, 20%, 30%, and 40%) and subjecting the samples to agitation at 120 rpm for 24 hours. The samples underwent filtration and were subsequently subjected to drying in an oven at a temperature of 70°C. To activate the material, a tabular electric furnace was utilized, and a steady stream of nitrogen gas was passed through it at a rate of 1 cm^3/min. The aim was to enhance the surface area of the sample by optimizing parameters including phosphoric acid concentration, temperature, and holding time to attain the maximum possible value.

3. Results and Discussion

3.1. Description of activated charcoal

The activated carbon was characterized using surface area analysis (BET), porosity measurements, Nitrogen adsorption and Desorption Isotherm, FT-IR spectroscopy, SEM imaging, X-ray diffraction (XRD), and Raman spectroscopy.

3.1.1. Surface Area and Porosity

To optimize the production of activated carbon, temperature ranges between 500°C and 900°C were investigated. After analysing samples produced at different temperatures, the study revealed that the sample synthesized at a temperature of 600°C exhibited the largest surface area, which measured 810 m^2/g (Fig. 1a). As a result, this temperature was chosen for additional analysis of other parameters. Various holding times ranging from 1h to 5h were tested, and after a holding time of 2 hours, the sample with the highest surface area of 945 m^2/g was observed (Fig. 1b). The effect of acid concentration (H_3PO_4) at concentrations of 0%, 10%, 20%, 30%, and 40%, was also studied. Based on the findings, it was observed that the sample with the greatest surface area, measuring at 920 m^2/g, was generated using an acid concentration of 20%. (Fig. 1c).

3.2. Nitrogen Adsorption and Desorption Isotherm

The porosity of activated carbon synthesized from DPW can be determined through the standard procedure of nitrogen adsorption, which follows the six types of IUPAC isotherms. The Type I isotherm, also known as Langmuir isotherm, indicates that the activated carbon from DPW is microporous with a

pore diameter of less than 2nm, and has a surface area of 920 m^2/g, as depicted in Fig. 2a. This isotherm suggests that the adsorption process occurs through a monolayer, making it highly suitable for chemical adsorption. The activated carbon's N_2 adsorption-desorption isotherms from DPW, as shown in Fig. 2a, follow the Type I isotherm, confirming its microporous nature. The results indicate that the rate of adsorption decreases as pressure increases.

3.3. FT-IR Study

FT-IR spectra of untreated and heavy metal-treated activated carbon samples are shown in the Fig. 2b. This analysis aimed to identify any alterations in the activated carbon's structure after the treatment. The OH bands are typically observed at wavelengths ranging from 2500 cm^{-1} to 3000 cm^{-1}, and these bands exhibit minor changes in their positions following the binding of heavy metal ions. The observed OH band is likely caused by the interaction between the DPW, H_3PO_4, and moisture present in the sample, leading to activation of the DPW (Baby & Hussein, 2020; Hegazy, Abdel-Ghani, & El-Chaghaby, 2014). The results of FTIR are depicted in Table 1.

Table 1
the spectral assignments of infrared (Bijekar et al.) bands in DPW

Assignment	AC carbon of DPW before adsorption	AC of DPW-Cr^{6+}	AC of DPW-Pb^{2+}	AC of DPW-Cd^{2+}	AC of DPW-Zn^{2+}
O-H (stretching)	2658.04	2651.85		2639.27	
C = O	1687.17	1683.09	1699.43		1703.52
Aromatic C = C (stretching)	1555.87	1545.16	1553.35	1530.92	1555.38
CH_2 & CH_3 (sym)	1103.14	1128.65	1142.68	1178.40	1120.19
C-C (stretching)	888.20	878.09	874.01	874.01	874.01
	735.07	739.16	735.07	743.25	735.07

3.4. SEM Analysis

The parent material of date palm waste (PM DPW) was observed under a microscope, and the resulting image displayed a rough surface with no visible pores (Fig. 3a). However, after the activation process, the surface of the adsorbent material displayed a porous structure (Fig. 3b). This observation indicates that the activation process successfully transformed the PM DPW into activated carbon. Activated carbon's porous structure enhances its adsorption capacity, making it effective for water pollution removal.

3.5. X-ray Diffraction analysis

In the X-ray analysis spectrum provided in number 4a figure, it is noticeable that there is a peak observed at approximately 2θ = 24°, which appears to be broad and less intense. This feature is indication of a high degree of disorder within the activated carbon prepared from date palm waste, which is consistent with the amorphous nature of organic materials. This result is not unexpected, as activated carbon derived from organic sources is generally known to possess an amorphous structure due to the lack of long-range order within the material. This is in contrast to crystalline materials, which exhibit sharp and intense peaks in their X-ray diffraction spectra due to their ordered atomic arrangement. Overall, the presence of a broad and less intense peak in the X-ray diffraction spectrum of the activated carbon obtained from date palm waste provides strong evidence for the amorphous nature of the material, which is in line with previous studies on activated carbon derived from organic sources (Bohli, Ouederni, Fiol, & Villaescusa, 2015; Köseoğlu & Akmil-Başar, 2015).

3.6. Raman spectrum

In the case of PM DPW, the absence of a Raman band indicates that there is no graphitic carbon present in the sample. Graphitic carbon is a common component of many organic materials and can produce a distinct Raman spectrum. However, the presence of a hub suggests that there may be other molecular structures present in the sample that are contributing to the Raman spectrum. However, the Raman spectrum of activated carbon derived from DPW showed the appearance of the G and D bands. The G band in the Raman spectra of graphite-like structures indicates the presence of sp^2 hybridized carbon atoms in the graphene lattice, while the D band is associated with defects or imperfections in the lattice structure, which can be indicative of various types of defects or disorders. The presence of both the D band and G band at 1395 cm^{-1} and 1595 cm^{-1}, respectively, has been detected, which provides evidence that the conversion of DPW (presumably referring to Depolymerized Wood) into activated carbon has been successful. Figure 4b. These findings suggest that DPW can be a potential source for the production of activated carbon, which can be used in various applications, including wastewater treatment, air purification, and energy storage.

3.7. Influence of pH on adsorption

The adsorption behaviour of metal ions is significantly influenced by the pH level of a solution. In particular, there is a notable difference when the pH is raised from 2 to 6. Increasing the pH can greatly enhance the linkages of metal ions, however, if the pH continues to rise beyond a certain point, it can cause the metal ions to precipitate out of the solution. Based on the experimental data, it was observed that the maximum percentage removal of four ions, namely chromium (Cr^{6+}), lead ion (Pb^{2+}), cadmium (Cd^{2+}) , and zinc (Zn^{2+}), occurred at a pH of 6. This indicates that the removal efficiency of these heavy metals is highest at a slightly acidic pH. It is worth noting that the percentage removal of these heavy metals may vary at different pH values, and pH is an important factor that can significantly influence the removal efficiency of heavy metals in various treatment processes. Lead (Pb) exhibiting 84% removal, Cr 80% removal, Cd 72% removal, and Zn demonstrated 65% removal (Fig. 5). The presence of hydrogen ions in acidic conditions impedes the adsorption of metal ions due to their competitive nature.

Consequently, in acidic conditions, metal ion adsorption is significantly reduced. Conversely, under alkaline conditions, the adsorption of metal ions is favoured as a larger proportion of metal ions have a positive charge, promoting their attachment to negatively charged surfaces. As a result of above findings, all subsequent batch experiments were conducted at a pH of 6.

3.8. Effect of adsorbent Dose

AC from DPW was used to study the adsorption of zinc, lead, cadmium, and chromium ions. The AC dosages used in this study were 0.1g, 0.15g, 0.2g, and 0.25g, while the preliminary metal ion concentration was maintained at 20 ppm in 100 mL liquid. Increasing AC derived from DPW led to higher adsorption efficiency for heavy metal ions. Maximum adsorption capacities were obtained for Zn^{2+} (86%), Pb^{2+} (82%), Cd^{2+} (75%), and Cr^{6+} (above 70%) at an AC dosage of 0.25 g per 100 mL of liquid (Fig. 5). This phenomenon can be attributed to the greater number of active sites available on the surface of the adsorbent, which facilitate the elimination of more ions from the solution. Therefore, increasing the amount of adsorbent can lead to higher adsorption efficiency.

3.9. Effect of the initial concentration of metal ions

The study utilized initial metal ion concentrations ranging from 5 ppm to 25 ppm in order to investigate the efficacy of the adsorption method. Results indicated that the adsorption process exhibited a decline in performance as metal ion concentrations increased due to saturation of the available adsorbent sites. At concentrations up to 10 ppm, all metal ions exhibited nearly complete adsorption, but as concentrations exceeded this threshold, adsorption rates gradually decreased (Fig. 5).

3.10. Effect of contact time on adsorption

A batch study was conducted by keeping the contact time fixed at various intervals, namely, 15, 30, 60, 90, and 120 minutes and adsorption behaviour of Zn^{2+} ion, Cr^{6+} ion, Pb^{2+} ion, and Cd^{2+} ion on activated carbon derived from date palm waste (DPW) was observed. The results showed that the adsorption efficiency increased with an increase in contact time. The highest adsorption percentages of approximately 83%, 80%, and 70% were recorded for Zn^{2+} and Cr^{6+}, Pb^{2+}, and Cd^{2+} ions, respectively, at a contact time of 120 minutes (Fig. 5). The initial stage of the adsorption process was characterized by a rapid uptake of metal ions on the surface of DPW activated carbon, presumably due to the availability of vacant adsorption sites. As the contact time increased, the accessibility of the binding sites reduced, resulting in a slower adsorption rate over time.

3.11. Adsorption Kinetics

In this research, we examined the adsorption characteristics of metal ions (Zn^{2+}, Cr^{6+}, Cd^{2+}, and Pb^{2+}) on AC DPW's surface and explored the step that controls the rate of the process. To achieve this objective, three different kinetic models. Pseudo-First Order, Pseudo-Second Order, and Parabolic Diffusion were employed. The Pseudo-First Order model equation is given as $1n\left(q_e - q_t\right) = 1nq_e - k_1 t$ where qe is the equilibrium adsorption, qt is the adsorption at time t, and k1 is the rate constant. The Pseudo-Second

Order model equation is $(t/q_t - t/q_e) = 1/k_2 {q_e}^2$ and the Parabolic Diffusion model equation is $1 - (M_t/M_0)/t = {K_t}^{-0.5} + b$ where Mo is the amount adsorbed at time 0 and Mt is the amount adsorbed at time t. According to the findings, the Pseudo-First Order model was the most suitable fit for all metal ions during the adsorption on AC of DPW, exhibiting a high correlation coefficient (R^2) of 0.99. Conversely, the other two models yielded lower correlation coefficients which implies that the bonding of metal ions to activated carbon's surface is attributed to the chemisorption mechanism. Figure 6 presents the results of the kinetic study.

Table 2

Overall kinetic study of DPW AC using correlation coefficient (R^2) and rate constant

Metal ion	R^2			Pseudo second order Kinetics
	Pseudo First Order	Pseudo second Order	Parabolic Diffusion	Rate Constant K (mg/min)
Cr 6+	0.91	0.99	0.97	2.79×10^{-4}
Pb2+	0.80	0.99	0.93	2.34×10^{-4}
Cd2+	0.97	0.99	0.93	3.07×10^{-4}
Zn2+	0.87	0.99	0.89	3.30×10^{-4}

3.12. Adsorption isotherms analysis

Adsorption isotherms models can provide insights into the behaviour of adsorbent materials and the efficiency of adsorption processes, as well as help to predict the behaviour of adsorption systems under different conditions for elimination of heavy metals. The Freundlich and Langmuir isotherm models are commonly applied to analyze the adsorption process and the interaction between heavy metal ions and activated carbon.

The equations for Freundlich isotherms:$Q_{eq} = K_f \times {C_{eq}}^{(1/n)} C_e$

The linear form:$\log Q_{eq} = \log K_f + (1/n) \times \log C_e$

The equation for Langmuir isotherms:$Q_e = (bQ_m C_e) / (1 + bC_e)$

The linear form:$C_e/Q_e = (C_e/Q_m) + (1/bQ_m)$

Where Ce indicates the equilibrium concentration of heavy metal ions in mg/L, Qe shows the quantity of metal ions in mg/g, Qm maximum adsorbed amount in mg/g on the surface of activated carbon, b is a constant, and Freundlich coefficients are Kf and 1/n (Fig. 7). The data from the isotherms exhibits a correlation with the Langmuir and Freundlich models for isotherms, the outcomes are presented in Table

3. It was determined that the Langmuir isotherm model provided the best fit for all of the metal ions. The findings of the study revealed a strong correlation between the concentrations of Cr^{+6}, Zn^{+2}, Cd^{+2} (correlation coefficient = 0.99) and Pb^{+2} (correlation coefficient = 0.93), which exceeded the correlation observed in the Freundlich isotherms and R2. The Langmuir isotherm also exhibited a high value, indicating that the adsorption process occurred through a monolayer and followed the chemisorption process.

Table 3
Heavy Metal (Pb^{+2}, Cd^{2+} Zn^{2+} and Cr^{6+}.) Adsorption Isotherms on AC DPW

Metal ions	Langmuir isotherm			Freundlich isotherm		
	q_e (mg/g)	b (L/mg)	R^2	K_f	n_f	R^2
Cr^{6+}	31	0.16	0.99	0.08	0.68	0.35
Pb^{2+}	35	0.22	0.93	3.74	3.69	0.53
Zn^{2+}	26	0.15	0.99	0.26	0.55	0.15
Cd^{2+}	29	0.15	0.99	0.30	0.54	0.23

4. Conclusion

Each year, thousands of tons of waste are generated from date palm trees, making it a promising and sustainable source of activated carbon. In this study, the conversion of date palm waste to activated carbon using acid activation was investigated. The resulting material was characterized using various spectroscopic techniques, including FT-IR, SEM, XRD and BET analysis. Subsequently, Activated carbon used to remove Zn^{2+}, Cd^{2+}, Cr^{6+}, and Pb^{2+} heavy metal ions from water. The present study aimed to optimize the adsorption process of metal ions onto activated carbon derived from date palm waste and isotherm models and reaction kinetics were applied to evaluate the performance of the adsorption process. The date palm waste's activated carbon demonstrated a significant surface area of 920 m^2/g, indicating a considerable amount of available surface area for adsorption and other surface-based processes, with a microporous structure (pore diameter < 2nm), as determined by type (1) isotherm analysis. The prepared activated carbon effectively removes 85% of Cr^{6+} ions, 83% of Pb^{2+} ions, and over 80% of Cd^{2+} and Zn^{2+} ions. Furthermore, the adsorbent was effective in removing all metal ions up to a concentration of 10ppm and up to 95% removal for concentrations up to 15ppm under optimal parameters. The adsorption process was well-described by the second-order kinetic model. Based on the results of the isotherm analysis, it was found that the Langmuir isotherm model provided a more accurate representation of the adsorption process compared to the Freundlich model. Henceforth, activated carbon is an appropriate material for the remediation of water contaminated with heavy metals due to its porous structure, large surface area, and presence of active sites. These properties enable activated carbon to effectively adsorb heavy metal ions and remove them from the water. This research

introduces an eco-friendly method to purify water contaminated with toxic heavy metal ions. The mentioned adsorbent is easily accessible in substantial amounts and has a low cost, making it a renewable resource that can be used repeatedly.

5. Deceleration

5.1 Ethics Approval and Consent to Participate

The research protocol and procedures outlined in this study have been reviewed and approved by Shah Abdul Latif University Ethics Committee (Approval Number: 2035/05). All participants provided informed consent before their participation in the study. They were informed about the study's objectives, procedures, potential risks, and benefits, and were assured that their participation is voluntary. Participants were also informed about their right to withdraw from the study at any time without consequences. Confidentiality of participants' information and data will be strictly maintained throughout the study and reporting process.

5.2 Consent for Publication

Participants have consented to the publication of anonymized and aggregated data obtained from this study. Any identifiable information will be carefully removed or altered to ensure the privacy and confidentiality of the participants. Images, audio recordings, or other forms of identifying information that could compromise anonymity will not be included unless explicit written consent has been obtained from the participants.

5.3 Conflict of Interest

The authors declare that they have no conflicts of interest that could affect the objectivity or interpretation of this research.

5.4 Author Contributions

Amjad Ali Maitlo conceived and designed the study, collected and analyzed the data, and wrote the manuscript. Wahid Bux Jatoi contributed to data analysis and manuscript editing. Mushtaque Ali Jakhrani provided critical insights during study design and contributed to manuscript revisions.

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Figures

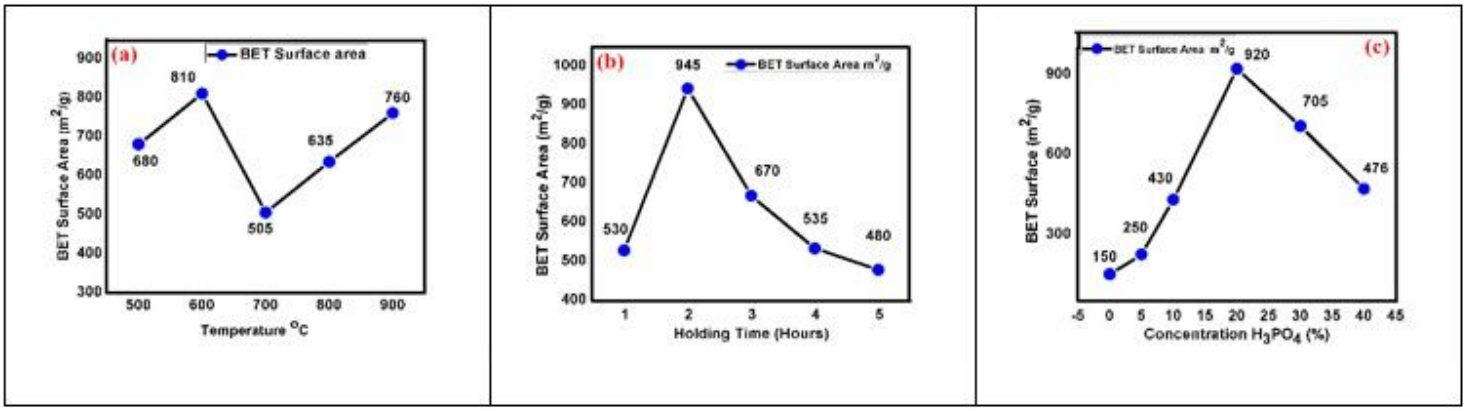

Figure 1

(McBride et al.) The effect of temperature at holding time of 2 hours & 20% H_3PO_4, (McBride et al.) The effect of holding time at 600 ^{0}C & 20% H_3PO_4, and (McBride et al.) The effect of H_3PO_4 concentration at 600 ^{0}C & holding time 2 hours.

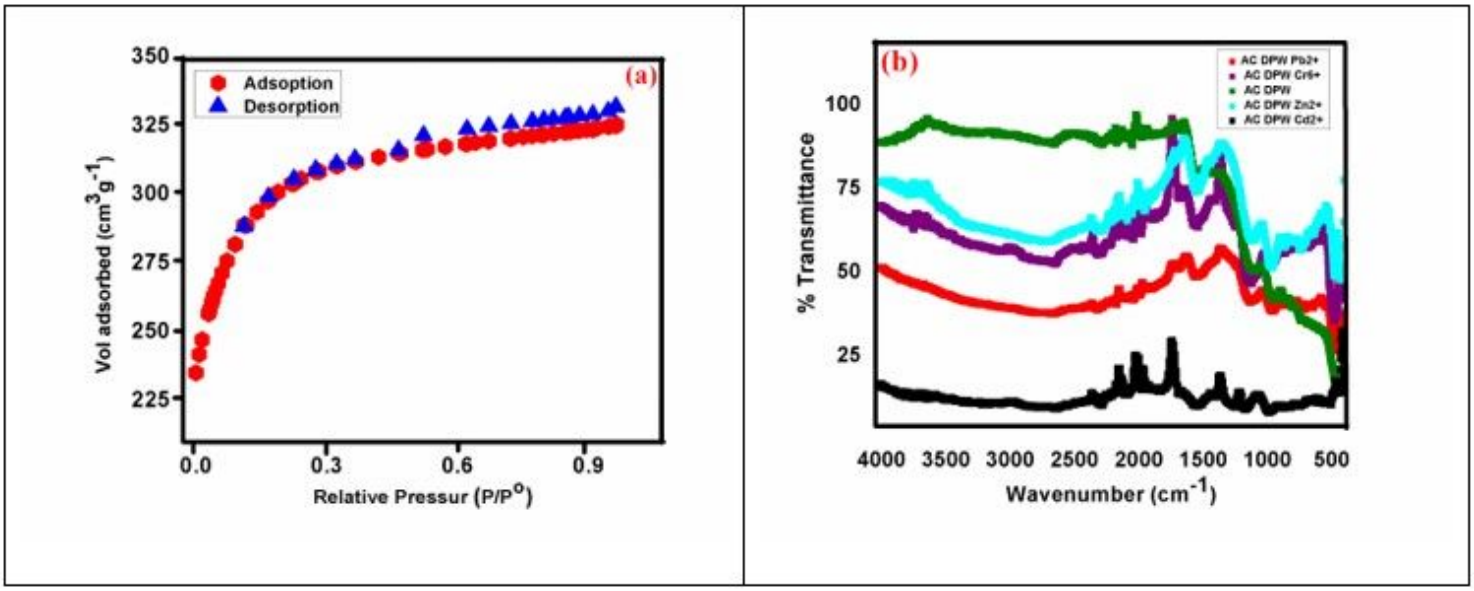

Figure 2

a) N_2 adsorption-desorption Isotherms, b) FT-IR spectra of the activated carbon of DPW.

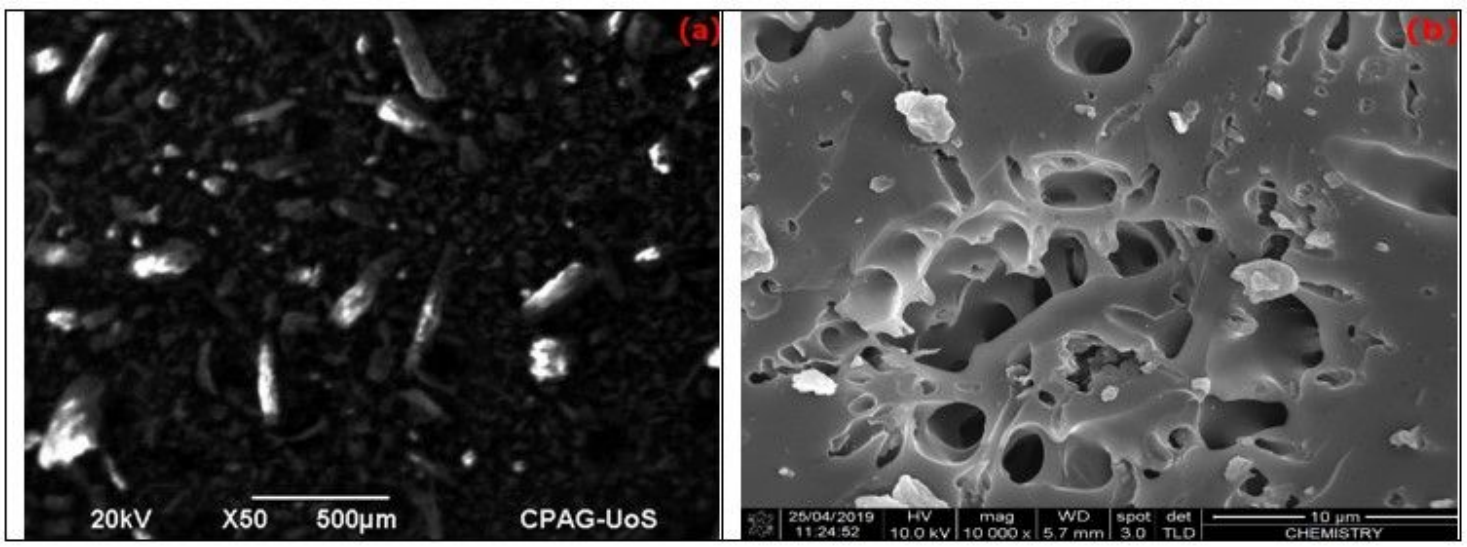

Figure 3

3a) Surface morphology of PM DPW 3b) Surface morphology of AC DPW

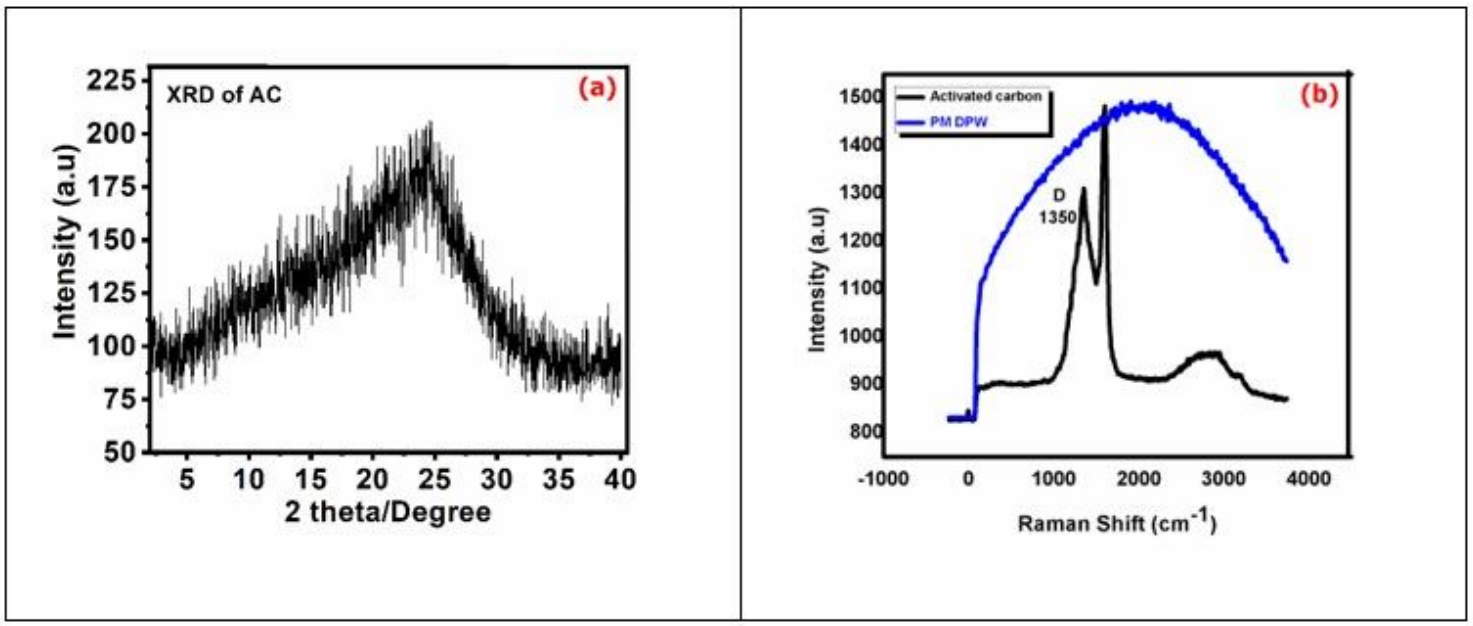

Figure 4

a) X-ray Diffraction of AC, b) Raman spectrum of PM DPW & AC.DPW.

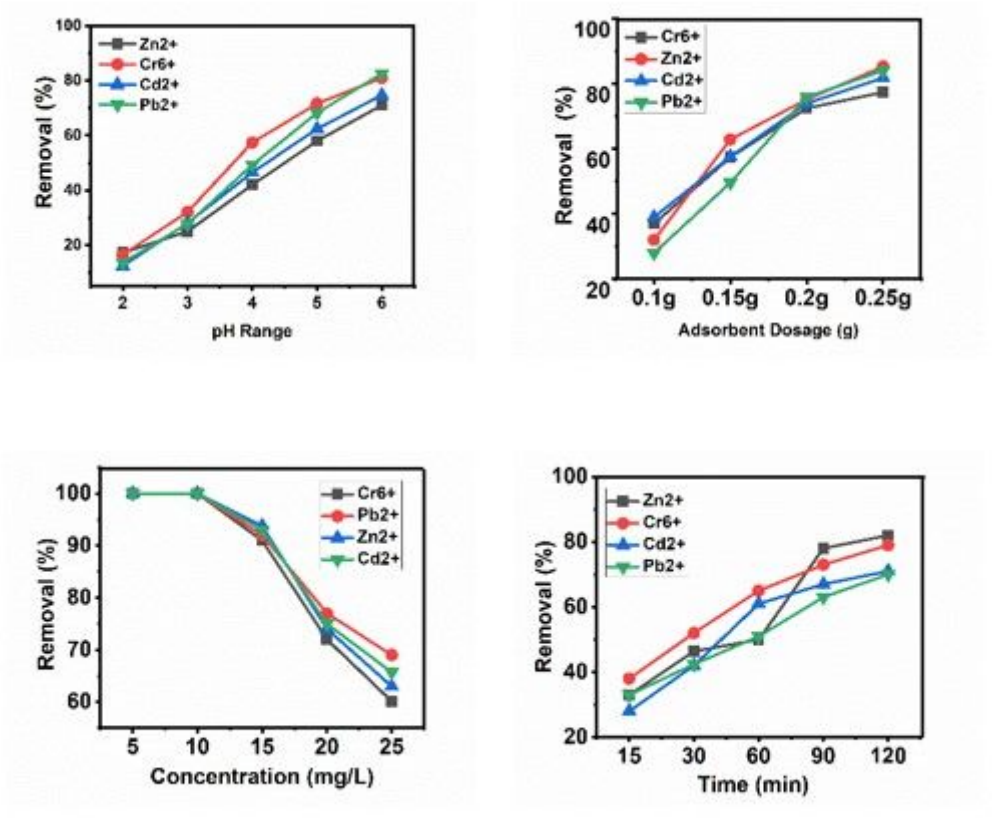

Figure 5

the influence of pH, dosage, concentration, and duration on metal ions adsorption onto DPW's AC adsorbent.

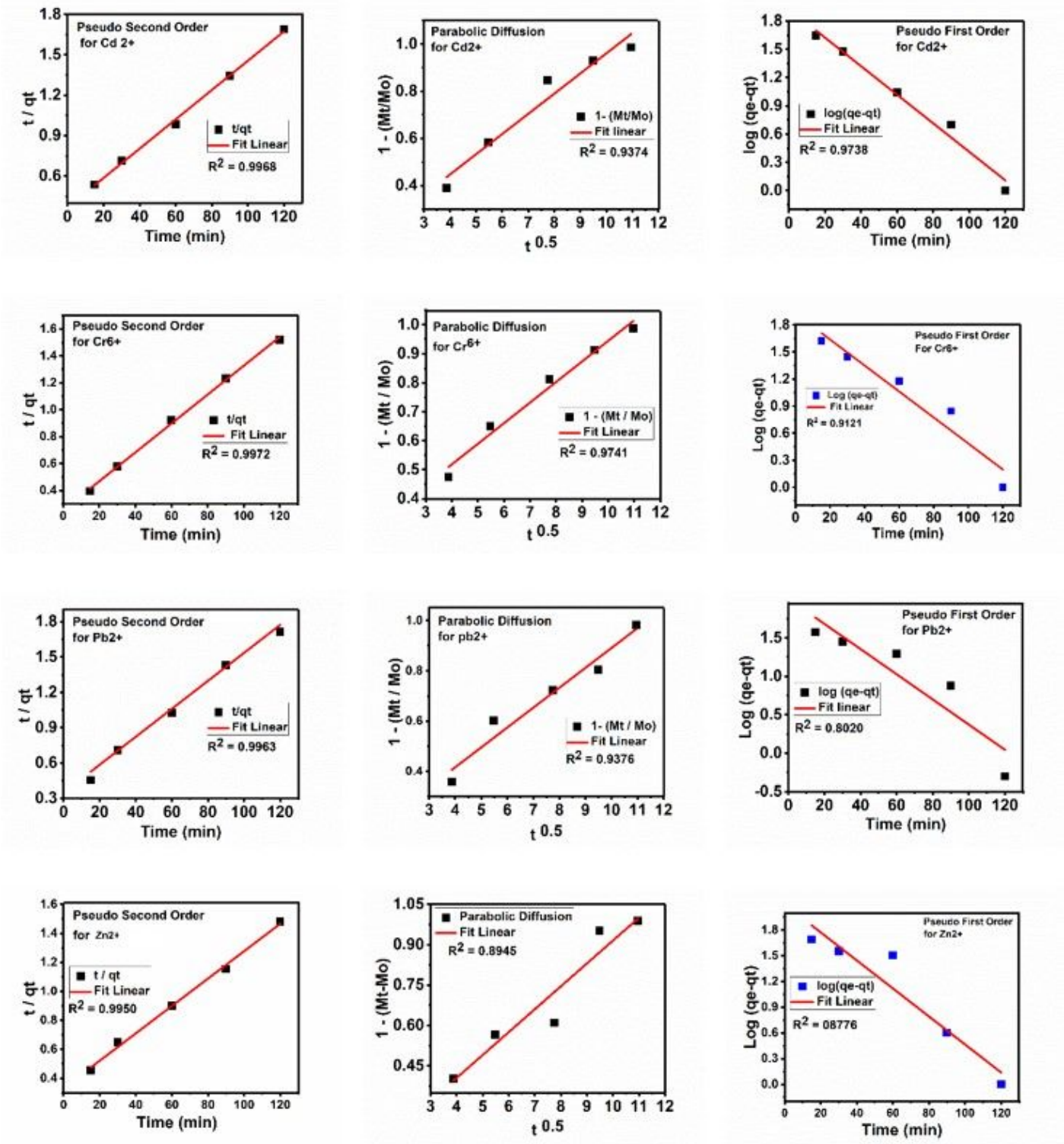

Figure 6

Metal ion kinetics with AC of DPW using pseudo-first, pseudo-second, and parabolic diffusion models for Cr^{6+}, Zn^{2+}, Cd^{2+}, and Pb^{2+}.

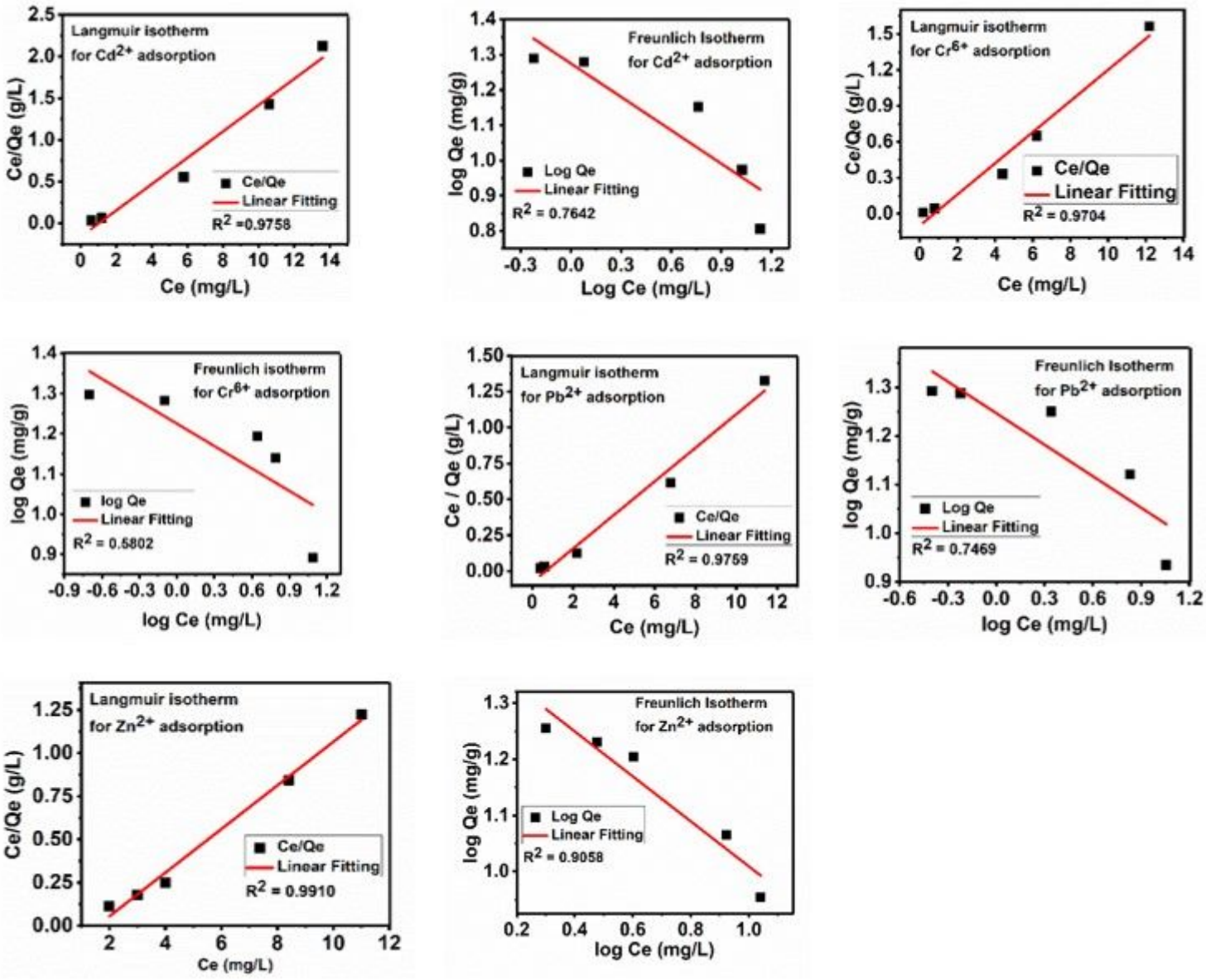

Figure 7

Freundlich and Langmuir models fitted Cr^{6+}, Pb^{2+}, Cd^{2+}, and Zn^{2+} adsorption onto AC DPW.