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ADSORPTION APPLICATIONS OF FRUIT AND VEGETABLE WASTE-BASED BIOCHAR FOR THE TREATMENT OF HEAVY METAL-CONTAINING WASTEWATER

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ABSTRACT
The present work explored the efficacy of fruit and vegetable waste-derived biochar (FVWB) for the removal of Pb^{2+}, Ni^{2+}, and Cu^{2+} ions. The physicochemical properties of the biochar were characterised by different analytical techniques. The effectiveness of the adsorbent was studied under different experimental conditions to determine the best operating parameters. The highest efficiency of adsorption of Pb^{2+}, Ni^{2+}, and Cu^{2+} ions was found to be 84.8, 89.9, and 87.9%. The equilibrium data of the adsorption was fitted with various isotherm and kinetics models. Further, FVWB was used for up to three cycles to remove Pb^{2+}, Ni^{2+}, and Cu^{2+} ions. After the first cycle, 62.1, 68.9, and 65.1% of Pb^{2+}, Ni^{2+}, and Cu^{2+} ions were removed. The percentage removal was found to be 34.8, 40.7, and 36.3% after the third cycle. This work correlates to SDG-6 and SDG-13. Future work may include surface modification of FVWB to improve the removal of metal ions, testing its efficiency with real wastewater, and evaluating its performance after several more cycles of regeneration and reuse.
Keywords: FVWB, Characterisation, Adsorption, Isotherm and kinetics, Re-utilisation.
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INTRODUCTION

An exponential increase in population, industrialisation, and urbanisation has detrimentally affected the quality of different water sources.[1] As per an estimate, over 2 million tons of agricultural, industrial and domestic wastewater are released into the water bodies every day globally.[2] The major toxicological effects of such effluents are the discharge of various organic and inorganic pollutants.[3] Among these pollutants, the existence of heavy metals in extreme amounts has severe health consequences on the entire ecosystem, including animals, plants and humans.[4] In general, heavy metals are categorised as non-biodegradable, harmful and toxic contaminants that remain in the ecosystem for a prolonged period, making the negative effects of pollution more severe or harmful over time.[5] Potential consequences associated with heavy metal pollution include their possible entrance into food chains, disruption of aquatic environments and rising risks of several acute health issues in human beings like carcinogenic, neurological and organ malfunctioning .[6] Therefore, in the 21st century, resolving the issues of heavy metal pollution has become a crucial global concern.[7] In recent years, several approaches have been utilised for removing heavy metals from wastewater. Such strategies involve chemical precipitation, solvent extraction, electro-flocculation, membrane separation, biotransformation, adsorption, coagulation, phytoremediation, ion exchange, electrochemical treatment and advanced oxidation processes.[8,9] However, many of these techniques are associated with various limitations like the requirement of very skilled labour, expensive equipment and materials and high energy demands.[8] In this perspective, the adsorption method, due to its distinctive benefits like easy operation, low cost, increased efficacy and liberty to

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choice of adsorbents have attracted an increased interest worldwide.[10] Generally, adsorption includes various methods at the boundary between a solution and an adsorbent like biochar. Such methods include magnetic bonding, ion exchange, co-precipitation/precipitation, intermolecular interactions, surface complexation and chemisorption. In particular, heavy metals mainly attach to the surface of biochar by mechanisms such as hydrogen bonding, π-electron acceptor-donor interactions, hydrophilicity, pore-filling and electrostatic interactions.[9] However, the efficiency of the adsorption method mainly depends on the selection of the adsorbent and its characteristics, such as regeneration potential, adsorption capacity and surface area.[10] Usually, the adsorbents like MXenes, dendritic polymers, metal-organic frameworks, layered double hydroxides and carbon-based materials like graphene or carbon nanotubes are used. But the production of all such adsorbents includes the utilisation of several chemicals and organic solvents, which can be toxic and increase the risk of secondary pollution because of leaching. In addition, the production process is energy-demanding and tedious.[8] Therefore, at present, biochar, because of its certain advantages like easy preparation, cost efficiency, reduced emissions, good adsorption potential and utilisation of a wide variety of biomass feedstock, has attracted an increased interest from scientists globally.[11] Biochar is a carbon-rich material known for its good pore structure, great specific surface area and many surface functional groups. Biochar is generally synthesised by the process of pyrolysis of different organic wastes like sewage sludge, manure and agricultural residues under restricted oxygen conditions.[12,13] However, in recent years, there has been a growing interest in utilising renewable and natural materials for wastewater treatment due to their non-toxicity and eco-friendly nature.[8] Fruits and vegetables derived waste materials can be useful and simple adsorbents because of their renewability, sustainability and low cost.[14] Previous studies have proved such waste materials to be good adsorbents because they have a high number of functional groups like hydroxyl groups and carboxylic acids, which assist the adsorption of heavy metals through complexation or ion-exchange, hence increasing their adsorption ability.[15] Secondly, the food industry is facing serious challenges due to the generation of large amounts of waste globally. According to the Food and Agriculture Organisation (FAO), around 1.3 billion tons of food is wasted yearly, exacerbating resource depletion and environmental pollution.[16] A considerable amount of this waste, i.e., around 1748 million tonnes, comes from fruits and vegetables yearly.[17] Therefore, conversion of such waste into useful products like biochar can be a potent solution for minimising problems related to waste generation.[15] In recent years, an extensive range of fruits and vegetables like watermelon rinds, carrot residues, sugar beet, mango peels, potato peels, garlic peels, corn residues, eggplant peels, cucumber peels, apple peels, lemon peels and many more have been utilised as adsorbents.[8] Therefore, the present work aimed to utilise biochar made from fruit and vegetable waste for heavy metal adsorption from artificially prepared wastewater. The present study aimed to achieve different United Nations Sustainable Development Goals (SDGs) like SDG-6 and SDG-13.

EXPERIMENTAL

All the reagents and chemicals used in the present study were of AR grade.

Biochar Preparation

Fruit and vegetable waste biomass was collected from local juice shops of Dehradun, Uttarakhand, India. The biochar was synthesised through slow pyrolysis in a pyrolysis chamber under oxygen-restricted conditions. Firstly, the moisture content was removed from the collected biomass waste by drying it in a hot air oven. The dried biomass was kept in the pyrolysis chamber at a temperature of 450 ℃ for 2.5 hours and with a heating rate of 0.1–1 °C/s. After pyrolysis, the prepared biochar was sieved through a sieve size of 2 mm and mixed well for uniformity. The overall scheme of the present study is depicted in Fig.-1. The prepared biochar was analysed using scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), energy-dispersive X-ray spectroscopy (EDX), and X-ray diffraction (XRD), respectively.

Preparation of Wastewater Solution

Heavy metal-contaminated wastewater was artificially synthesised by mixing the required amounts of nickel nitrate [Ni $(NO_3)_2.6H_2O$], cupric chloride ($CuCl_2$) and lead nitrate [Pb $(NO_3)_2$] in 100 mL of

double-distilled water. The solution was well-mixed, and after that, two to three drops of concentrated HCl were added. The prepared mixture was considered a stock solution and utilised for preparing all the requisite working solutions. 0.1 M HCl or 0.1 M NaOH was used for adjusting the pH of all working solutions.

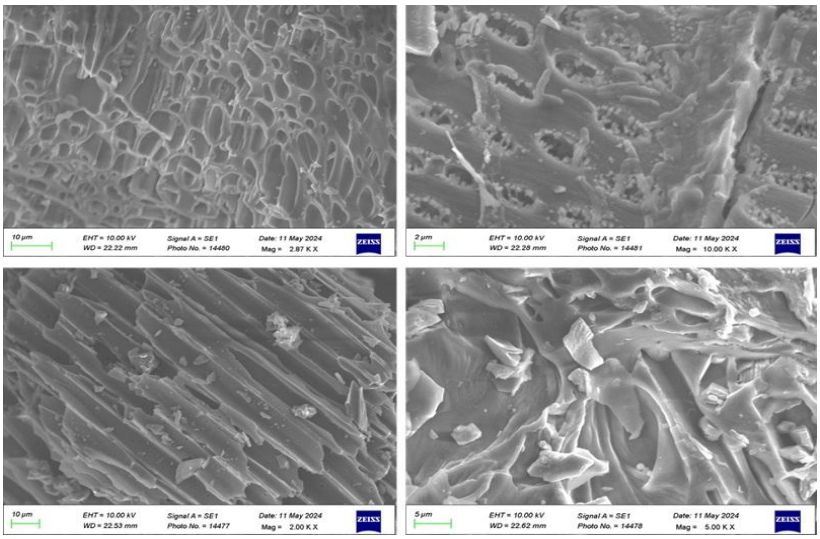


Fig.-1: SEM Images of FVWB (Before adsorption)

Adsorption Study

The adsorption of Pb^{2+}, Ni^{2+}, and Cu^{2+} ions via FVWB was conducted by varying key experimental parameters. These parameters include pH, contact time, dosage, temperature, and concentration. The adequate amount of FVWB was treated with 100 mL of synthetic wastewater at 270 rpm and specific time periods. After adsorption, the suspension was filtered, and the residual metal ion concentration in the filtrate was determined using AAS (Atomic absorption spectrophotometry). Initially, the metal ion concentration was 10 mg/mL for all adsorption parameters. Different kinetic and isotherm models were utilised for examining the practicability of adsorption processes. The biochar removal efficiency (% R) was calculated using the following equation (Eq.-1):

$$\% R = \frac{(C_1 - C_2)}{C_1} X\ 100 \quad (1)$$

In this equation, C_1 and C_2 are the initial and final concentrations of heavy metal ions (mg/L).

Regeneration Study

The FVWB has also been reused for further adsorption of Pb^{2+}, Ni^{2+}, and Cu^{2+} ions from wastewater. This experiment was performed at pH 5, contact time 30 min, and 270 rpm. Initially, the adsorbent was treated with 0.1 M HCl under constant shaking and then washed with double-distilled water. 100 mg of FVWB was added to 100 mL of solution containing Pb^{2+}, Ni^{2+}, and Cu^{2+} ions for 30 min. Then the solution was filtered, and the concentration of metal ions was measured in the filtrate.

RESULTS AND DISCUSSION

Characterisation of FVWB

SEM/EDX

The morphological features of FVWB were investigated using the SEM technique. Figure-1 depicts the SEM images of FVWB before the adsorption of Pb^{2+}, Ni^{2+}, and Cu^{2+} ions. The porous, regular, and elongated structure appears in these images. After adsorption, this morphology is disturbed, and FVBP becomes irregular and much less porous (Fig.-3). The EDX spectra and composition of FVBP are presented in Fig.-2 and 4 (before and after adsorption). The main elemental composition is illustrated in these figures. The association of Pb^{2+}, Ni^{2+}, and Cu^{2+} ions on FVBP after the adsorption is also illustrated in Fig.-4.

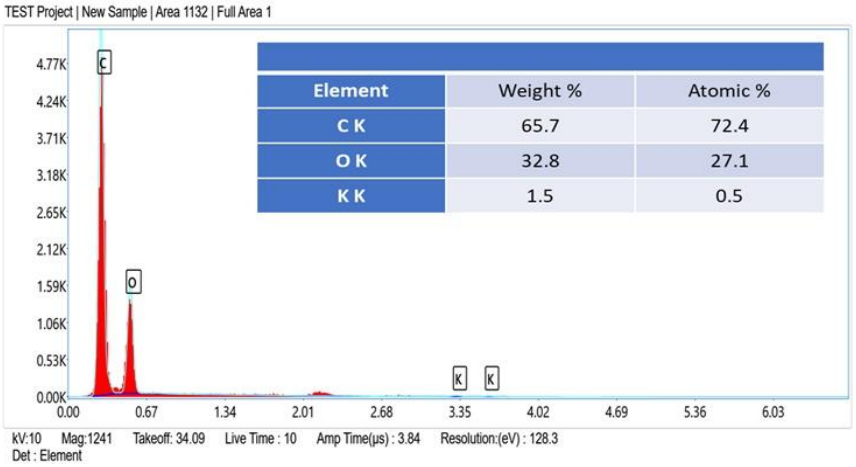


Fig.-2: EDX Spectra and Elemental Composition of FVWB (Before adsorption)

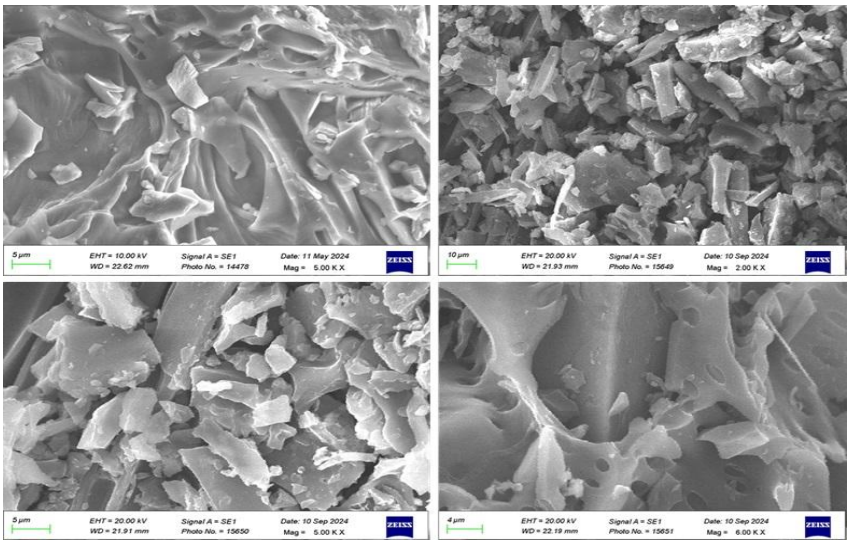


Fig.-3: SEM Images of FVWB (After adsorption)

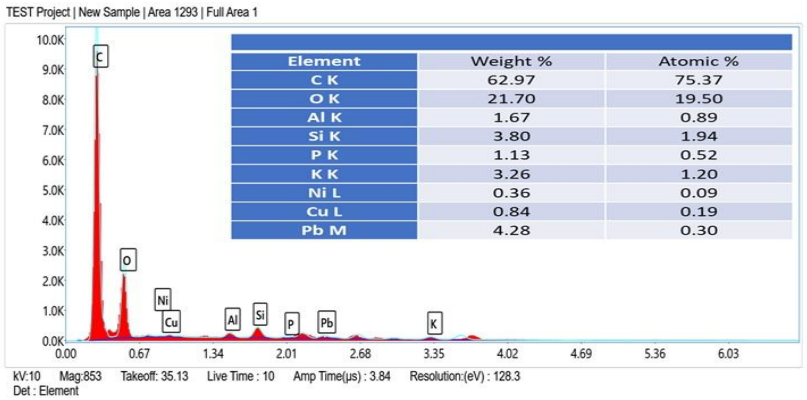


Fig.-4: EDX Spectra and Elemental Composition of FVWB (After adsorption)

FTIR

The presence of different functional groups or chemical bonds on the surface of FVWB was determined via FTIR analysis. Figure-5 (A) illustrates the FTIR spectra of FVWB. The important FTIR peaks were assigned at 3423, 2883, 2829, 1606, 1431, 875, and 781 cm^{-1}. The FTIR peak at 3423 cm^{-1} is relevant to O-H (str). The peaks at 2883 and 2829 cm^{-1} are assigned for C-H (str). The C-H (bend.) has been assigned

at 1431 cm^{-1}. After that, the peaks, such as 875 and 781 cm^{-1}, are assigned to the interaction of some inorganic constituents with carbon atoms of FVWP.[18-23]

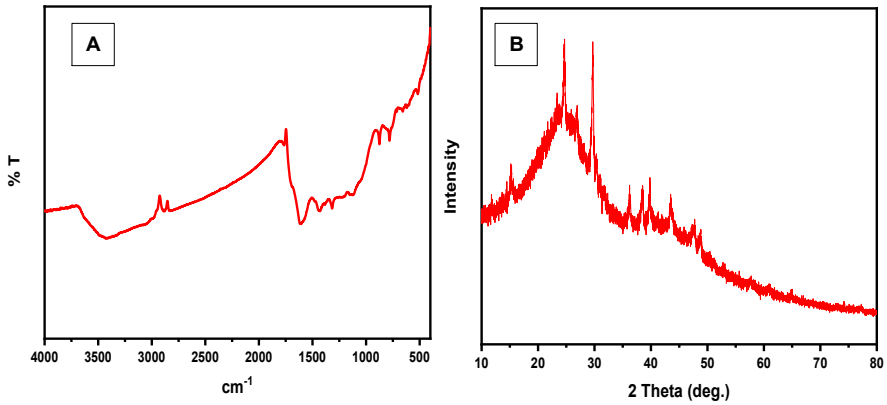


Fig.-5 (A) FTIR Spectra and (B) XRD Pattern of FVWB

XRD

Figure-5 (B) depicts the XRD pattern of FVWB and indicates its amorphous nature. The major XRD peaks are assigned at 2 θ = 15.1^0, 24.7^0, 26.8^0, 29.6^0, 36.2^0, 38.5^0, 39.8^0, 43.4^0, 47.7^0, and 48.8^0, respectively. The XRD peaks at 24.7^0 and 26.8^0 generally correspond to 002 planes (graphitic carbons), and 43.4^0 is relevant to the plane carbon structure (100). Other remaining peaks are generally due to the presence of different inorganic constituents on the surface of FVWP.[20,24-27]

Adsorption Study

Effect of Contact Time, Dosage, and pH

The adsorption study of selected heavy metal ions using FVWP was conducted via different adsorption parameters. Fig.-6 (A to C) depicts the effect of contact time, dosage, and pH on the adsorption of Pb^{2+}, Ni^{2+}, and Cu^{2+} ions, respectively. An essential factor in the adsorption phenomena is the contact time. This parameter is useful for both the kinetic study of adsorption and for the determination of the minimal time needed to adsorb contaminants from aqueous solutions.[28] After 10 min, the percentage of Pb^{2+}, Ni^{2+}, and Cu^{2+} ion adsorption was 19.2, 37.7, and 21.1%, and after 40 min, it increased to 35.6, 56.8, and 34.3%. After 60 min, the highest adsorption was determined to be 43.2%, 69.9%, and 38.2%, after which it remained constant. After 60 min, all active groups may have been occupied by Pb^{2+}, Ni^{2+}, and Cu^{2+} ions[12] (Fig.-6A). Rabiee Abyaneh *et al.*[27] have prepared *Ligustrum vulgare*-based biochar for the adsorptive removal of Pb^{2+}, Mn^{2+}, and Cd^{2+} ions. They used contact time ranges from 30 to 300 min and found maximum adsorption efficiency at a contact time of 120 min. After this contact time, adsorption efficiency became constant.[27] The effect of dosage was investigated by varying the amount of FVWP from 100 to 800 mg. The percentage adsorption of Pb^{2+}, Ni^{2+}, and Cu^{2+} ions was found to be 32.3, 48.1, and 17.1% at 100 mg of FVWP. It increased effectively to 52.1, 61.9, and 32% at 400 mg of FVWP and also achieved 58.9, 68.3, and 42.7% at 600 mg of FVWP. The maximum adsorption efficiency was found to be 61.4, 66.7, and 46.5% at 800 mg of FVWP (Fig.-6 B). The amount of adsorbent used has a major impact on the way heavy metal adsorption succeeds. Optimising the dose is essential for both economy and efficacy in adsorption processes. Chen *et al.* [29] studied the adsorption of Cd^{2+} ions using corn stover-based biochar under the dosage variation of 100 to 600 mg. They obtained similar trends of adsorption and a maximum removal efficiency of 97.9% at a dosage of 400 mg. The pH of working solutions is also an important parameter and is generally used to achieve the highest percentage removal of heavy metals from aqueous solutions. The higher acidic pH of working solutions helps to provide non-acidified active sites for the interactions with heavy metals. Repulsion occurs between heavy metals and acidified active sites at lower acidic pH.[14] The percentage adsorption of Pb^{2+}, Ni^{2+}, and Cu^{2+} ions was found to be 11.7, 28.8, and 12.2 % at pH 2 and increased to 58.9, 67.5, and 63.1% at pH 5. At pH 6, the adsorption achieved 84.8, 89.9, and 87.9 %, respectively (Fig.-6C). Li *et al.* [30] used clay-loaded biochar for the

removal of Cu^{2+} ions from wastewater under the pH range of 1 to 5. They also achieved maximum adsorption of Cu^{2+} ions at a higher acidic pH (5) (Fig.-6 C).

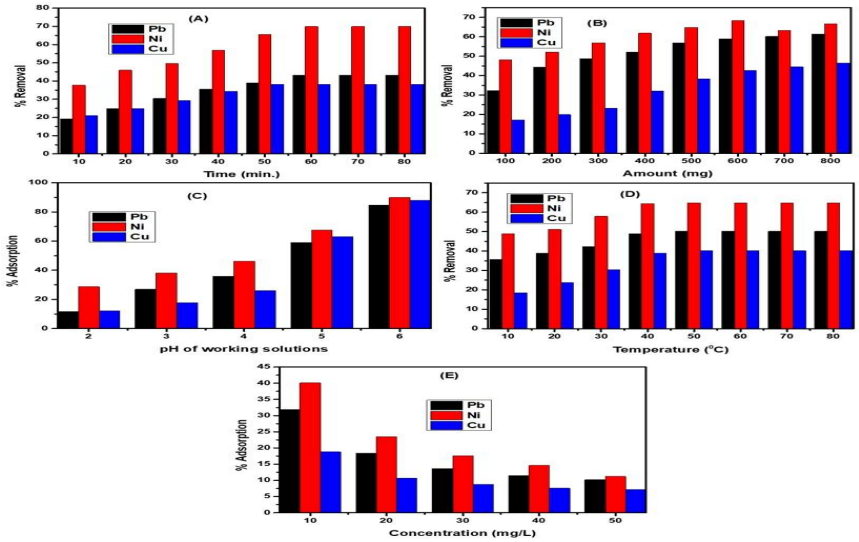


Fig.-6: Adsorption of Pb^{2+}, Ni^{2+}, and Cu^{2+} Ions on FVWB: (A) Effect of Contact Time, (B) Effect of Dosage, (C) Effect of pH, (D) Effect of Temperature, and (E) Effect of Concentration

Effect of Temperature and Concentration

Temperature is another important parameter of adsorption; heavy metal removal increases with the increase in temperature in solutions. After a certain temperature range, adsorption goes down or becomes stable. When the temperature increases, the interaction between heavy metals and active sites decreases. It led to more heavy metal dissolution from adsorbing substance to solution.[28] The removal efficiency of Pb^{2+}, Ni^{2+}, and Cu^{2+} ions was recorded to be 35.7, 48.9, and 18.5% at 10°C. These values increased to 48.9, 64.4, and 38.9% at 40°C. The highest adsorption percentages for Pb^{2+}, Ni^{2+}, and Cu^{2+} ions were recorded as 50.2, 64.9, and 40.2% at 50°C; after this, the values remained constant (Fig.-6 D). The concentration of heavy metals in the working solutions also influences the percentage removal of these metals when using any adsorbent. The removal efficiency rises with higher concentrations of heavy metals, attributed to the greater driving force for mass transfer. There is a limitation; the concentration of heavy metals in wastewater is not favourable for maximum adsorption due to saturation of available active sites on the adsorbent. The removal efficiency of Pb^{2+}, Ni^{2+}, and Cu^{2+} ions was found to be 31.9, 40.1, and 18.8% at 10 mg/L and decreased to 11.4, 14.6, and 7.6% at 40 mg/L. The removal efficiency declined to 10.2, 11.2, and 7.1% at 50 mg/L (Fig.-6E). Osman *et al.*[31] also observed the optimum temperature of 40°C for the removal of Pb^{2+} and Cd^{2+} using a chemically synthesised adsorbent (Fe_3O_4/SiO_2/PAM).

Isotherm Modelling

Adsorption isotherms are used in the quantification of the adsorption process. Dispersion of heavy metals in two different phases. These isotherm models are useful in the observation of mechanistic pathways involved during the adsorption of heavy metals on the surface of solid supports. In this study, Langmuir and Freundlich isotherm models were used for the estimation of the adsorption of Pb^{2+}, Ni^{2+}, and Cu^{2+} ions on the surface of FVWB. In general, the Langmuir isotherm model is used for the observation of the formation of a single layer on the surface of solid supports containing the same active sites. The linear form of the Langmuir isotherm model is written in the following equation:

$$\frac{C_2}{Q_e} = \frac{1}{Q_{max} K_L} + \frac{C_2}{Q_{max}} \quad (2)$$

In this eq., C_2, Q_e, Q_{max}, and K_L are the equilibrium concentration, metal adsorbate (mg/g), adsorption capacity (mg/g), and Langmuir constant (L/mg), respectively. The Q_{max}, K_L, and R^2 were obtained from the plot of the curve C_e/Q_e vs. C_e (Fig. 7 A). The maximum Q_{max} for lead, nickel, and copper ions was

calculated to be 5.743, 6.172, and 4.545 mg/g, and the Langmuir constant, KL, was 0.126, 0.366, and 0.056 L/mg, respectively (Table-1). The R^2 value shows that for the Ni^{2+} ion, its adsorption process follows the Langmuir isotherm model more closely as compared to the Pb^{2+} and Cu^{2+} ions (Table-1). The equilibrium data were also fitted to the other Langmuir (dimensionless factor) R_L. R_L can be mathematically represented as below:

$$R_L = \frac{1}{1+C_i X} \quad (3)$$

The values of R_L for Pb^{2+}, Ni^{2+}, and Cu^{2+} ions were found to be less than 1. This also indicates the appropriateness of the adsorption process. The Freundlich isotherm model explains the interaction of heavy metals on the heterogeneous surface of solid supports or adsorbents.[32] The linear equation for the Freundlich model is given by the following equation:

$$\log Q_e = \log K_F + \frac{1}{n}\log C_e \quad (4)$$

In equation (iv), K_F and 1/n are the adsorption capacity and adsorption intensity, and their values are evaluated from the curves shown in Fig.-7B. The values of the contribution of Pb^{2+}, Ni^{2+}, and Cu^{2+} ions were found to be 1.941, 2.745, and 2.135 mg/g. It was observed that the value of 1/n (less than 1) is suitable for adsorption of selected heavy metal ions on the surface of FVWB (Table-1). The values of R^2 shown in the table indicate the greater suitability of the Freundlich model for Pb^{2+} ions than for Ni^{2+} and Cu^{2+} ions.

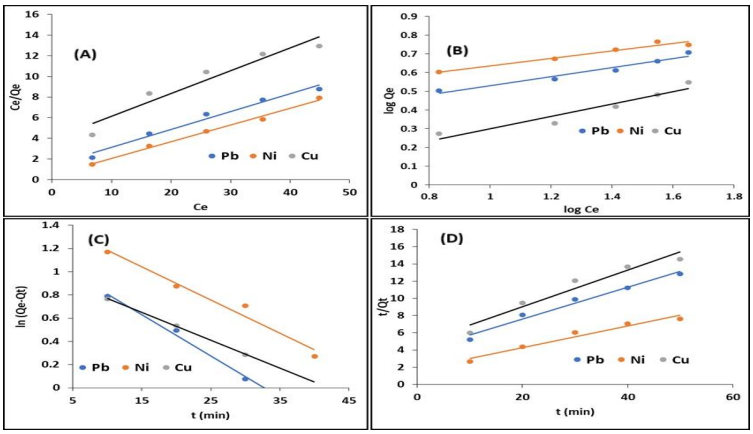


Fig.-7: (A) Langmuir (B) Freundlich (C) Pseudo First Order, and (D) Pseudo Second Order Models

Table-1: Evaluated Values of Isotherm Constants for the Removal of Pb^{2+}, Ni^{2+}, and Cu^{2+} Ions using FVWB

Isotherm models	Heavy metals	Parameters	Values
Langmuir	Pb^{2+}	Q_{max} (mg/g)	5.743
		X	0.126
		R^2	0.977
	Ni^{2+}	Q_{max} (mg/g)	6.172
		X	0.366
		R^2	0.992
	Cu^{2+}	Q_{max} (mg/g)	4.545
		X	0.056
		R^2	0.925
Freundlich	Pb^{2+}	K_F (mg/g)	1.941
		1/n	0.241
		R^2	0.953
	Ni^{2+}	K_F (mg/g)	2.745
		1/n	0.198
		R^2	0.954
	Cu^{2+}	K_F (mg/g)	2.135
		1/n	0.329
		R^2	0.921

Kinetics of Adsorption

The adsorption of Pb^{2+}, Ni^{2+}, and Cu^{2+} ions on the surface of FVWB was also analysed using pseudo-first- and second-order kinetics models. The kinetic models are generally used in the observation of mechanisms of adsorption and feasibility of selected adsorption parameters. Furthermore, selecting optimal conditions for enormous batches of metal removal operations requires knowledge of the kinetics of metal absorption.[33] Pseudo-first-order kinetics models generally explain that the rate-limiting step is physical adsorption, not the chemical interaction.[32] The linear form of this model is presented as below:

$$\ln\ (Q_e - Q_t) = \ln Q_e - k_1 t \qquad (5)$$

In the above equation, Q_e, Q_t, and k_1 are the metal adsorbed at equilibrium, at different time intervals, and the pseudo-first-order rate constant, respectively. The k_1 of Pb^{2+}, Ni^{2+}, and Cu^{2+} ions was derived using the graph of ln (Q_e – Q_t) vs t (Fig.-7 C), and the values were found to be 0.0356, 0.0286, and 0.0241 min^{-1}, respectively (Table-2). The values of R^2 suggest that the effectiveness of this model is greater for Cu^{2+} ions as compared to Pb^{2+} and Ni^{2+} ions (Table-2). The pseudo second-order model postulates that the heavy metal adsorption onto the surface of the adsorbent occurs through chemical interactions. The mathematical formulae of this model are given below:

$$\frac{t}{Q_t} = \frac{1}{k_2 Q_e^2} + \frac{t}{Q_e} \qquad (6)$$

In equation (vi), k_2 is the pseudo second-order rate constant (g/mg min) and is calculated from the curve t/Q_t vs t as shown in Fig.-7D. The k_2 was calculated as 0.0344, 0.0158, and 0.0454 g/mg min for Pb^{2+}, Ni^{2+}, and Cu^{2+} ions, respectively. The R^2 values indicate that the pseudo-second-order model is more suitable for Pb^{2+} than for Ni^{2+} and Cu^{2+} ions (Table-2).

Table-2: Evaluated Values of Kinetic Constants for the Removal of Pb^{2+}, Ni^{2+}, and Cu^{2+} Ions using FVWB

Kinetics models	Heavy metals	Parameters	Values
Pseudo-first-order	Pb^{2+}	k_1 (min^{-1})	0.0356
		R^2	0.991
	Ni^{2+}	k_1 (min^{-1})	0.0286
		R^2	0.969
	Cu^{2+}	k_1 (min^{-1})	0.0241
		R^2	0.993
Pseudo-second-order	Pb^{2+}	k_2 (g/mg min)	0.0344
		R^2	0.977
	Ni^{2+}	k_2 (g/mg min)	0.0158
		R^2	0.961
	Cu^{2+}	k_2 (g/mg min)	0.0454
		R^2	0.944

Reutilization of FVWB

The from working solutions. After the first cycle, the removal efficiency of Pb^{2+}, Ni^{2+}, and Cu^{2+} ions was FVWB was reutilized for the further adsorption of Pb^{2+}, Ni^{2+}, and Cu^{2+} ions found to be 62.1, 68.9, and 65.1%, respectively. It was declined to 54.1, 60.3, and 57.2%. Finally, the removal efficiency of Pb^{2+}, Ni^{2+}, and Cu^{2+} ions was calculated to be 34.8, 40.7, and 36.3% after the third cycle (Fig.-8).

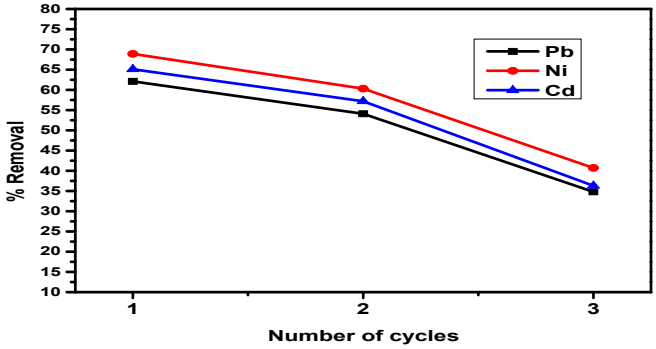


Fig.-8: Regeneration Study of FVWB for the Further Adsorption of Pb^{2+}, Ni^{2+}, and Cu^{2+} Ions

CONCLUSION

In this study, the food waste material has been utilised for the development of FVWB. The process of development was found to be cost-effective, efficient, and simple. The FVWB was found to be capable of removing toxic Pb^{2+}, Ni^{2+}, and Cu^{2+} ions from wastewater solution, and the important features of FVWB were analysed via different analytical methods. The maximum percentage adsorption was found at optimised conditions such as pH 6, a dosage of 800 mg, and a contact time of 60 min, respectively. Under these optimised conditions, FVWB can be utilised for large-scale operation of wastewater treatment. Further, FVWB has also been re-utilised up to three cycles and found to be an effective adsorbent for wastewater treatment. This research addresses SDG-6 by examining food wastage for the elimination of heavy metals from wastewater. The research addresses SDG-13 to promote waste minimisation.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-6: Clean Water and Sanitation*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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