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Adsorption of Copper, Zinc, Chromium and Nickel Using Plantation crop waste as Adsorbents in Contaminated Soil

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ABSTRACT

This study investigates the adsorption behaviour of copper, zinc, chromium and nickel on plantation crops such as Arecanut husk (AH) and Coconut husk (CH) using a batch technique. Key parameters affecting the adsorption phenomenon were contact time, initial metal ion concentration, and temperature. The results showed equilibrium was reached within four hours, with adsorption capacity being temperature-dependent and endothermic. Efficiency of removal was maximum at lower concentrations, while adsorption capacity improved at higher concentration. Freundlich and Langmuir models are appropriate for predicting the adsorption isotherm. At 30^{0}C, the maximum adsorption of Cu, Zn, Cr and Ni on AH and CH were 0.95 mg/g, 1.27 mg/g, 0.78 mg/g, 0.88 mg/g, 1.22 mg/g, 1.64 mg/g, 1.66 mg/g, and 1.69 mg/g respectively. CH exhibited higher removal and adsorption capacities than AH, with zinc showing the strongest adsorption attraction, following the order Cu < Ni < Cr< Zn for plantation crop types of adsorbents.

Keywords: adsorption, isotherms, plantation crop waste, traces, removal, attraction

1. INTRODUCTION:

Heavy metals are a type of toxin commonly found in industrial and municipal waste-waters (Bradl, 2004). They originate from various industries, including mining, electroplating, staining, electro-chemical metal processing, and battery storage, as well as from human activities (Kadirvelu, et al., 2001). Due to their stability, heavy metals are not degraded or removed (Shi, et al., 2009 and Vinodh, et al., 2011). When wastewater is discharged without adequate treatment, heavy metals can accumulate as residues in the ecosystem. These contaminants can be noticed in soil, surface water, groundwater (Xing, et al., 2011), sediments (Dube, et al., 2001), plants, and even dust (Argun, et al., 2007) particles (Zhao, et al., 2010; Wei, 2010; Yaylali, 2011). Exposure to heavy metals is linked to numerous health issues, including lung and kidney damage, Wilson's disease (characterized by neurological or psychiatric signs from liver disease combined with metal deposits), sleeplessness, dermatitis, sickness, long-lasting asthma, headaches, giddiness, rapid breathing, coughing, and cancer (Abat, et al., 2012; Meena, et al., 2005).

This pressing issue is driving researchers to dedicate their efforts to identifying the significant methods for eliminating heavy metals. Several approaches, including purification, chemical precipitation, ion-exchange, adsorption with activated carbon, electrode position, and membrane processes, have been explored. Among these, adsorption is regarded as one of the most efficient and cost-effective techniques for removing heavy metal ions from aqueous solutions (Cazanga, et al., 2007).

When wastewater is discharged into soil, it first percolates through the soil layers before either infiltrating downward into groundwater or moving across the surface to lower-lying areas. Numerous studies have established that soils can successfully remove heavy-metals from wastewater sources (Gonzalo Montes, et al., 2015; Vieira, et al., 2014; Duwiejuah, et al., 2017). Thus, soil acts as a natural filter, contributing to wastewater treatment by trapping metals before they reach groundwater or nearby bodies of water. The primary mechanism that Influences the behavior of heavy-metals in soil is their adsorption from the liquid phase onto the solid phase of the soil particles (James, et al., 2021;

Fomina, et al., 2014). Although number of studies have highlited heavy metal adsorption by soil, only a limited range of soil types has been examined. Additionally, the studies did not fully replicate the real-world conditions under which wastewater interact with soil.

The study specifically aims to examine the adsorption of Cu, Zn, Cr, and Ni using plantation crop waste are Arecanut husk (AH) and Coconut husk (CH) from different soil samples from in and around Davangere district, Karnataka India, focusing on contact-time, initial-concentration and temperature effects. This study examines the adsorption condition of common heavy-metals like copper (Cu), nickel (Ni), chromium (Cr), and zinc (Zn) in various soils by employing batch methods. The batch method assessed the effects of contact time, adsorption isotherms, and temperature on adsorption, using the two isotherms are Freundlich and Langmuir models used.

2. MATERIALS AND METHODS ADSORBENTS :

Plantation crop wastes like Arecanut husk (AH) and Coconut husk (CH) samples were collected from the surrounding arecanut and coconut plantation in and around Davangere district, Karnataka State. The collected Arecanut husk (AH) and Coconut husk (CH) were thoroughly washed with tap water and then rinsed with distilled water to remove all traces of impurities, oil, dirt, dust, and salts. The samples were then sun-dried followed by oven-drying at 100°C for 24 hours (Samarghandi, et al., 2011). Once dried, the samples were cut and pulverized into smaller particles using a mortar and pestle, and subsequently sieved into different particle sizes: 75 µm, 150 µm, 355 µm, 425 µm, and 835 µm.

2.1 Soil Samples

Soil samples were obtained from the waste dumping yard in Davangere city. The samples collected as core samples from a depth of 30 cm below the ground surface. The properties of these soil cores samples are appraised in accordance with IS 2720 standards (Song, et al., 2019a).

2.2 Adsorbents

In this study, Copper (Cu), Nickel (Ni), Chromium (Cr) and Zinc (Zn) were chosen as representative heavy-metals and prepared in solution form to act as adsorbents. Stock solutions of $Cu(NO_3)_2$, $Ni(NO_3)_2$, K2Cr2O7 and $Zn(NO_3)_2$, each at a concentration of 100 g/mL, were dissolved using deionized water to attain the desired specific concentrations. The initial concentration of heavy-metal solution is 40mg/L for these. Table 1 presents the properties of the heavy metal concentrations.

Table 1: Properties of Heavy metal solutions

Properties	Copper Nitrate	Zinc Nitrate	Nickel Nitrate
Formula	Cu(NO3)2	Zn(NO3)2	Ni(NO3)2
Molecular Weight	187.56 g/mol	189.36 g/mol	241.39 g/mol
Density (g/cm3)	2.32	2.06	2.05
Solubility (g/100ml)	156.0	184.30	94.20
Formula	Cu(NO3)2	Zn(NO3)2	Ni(NO3)2

Adsorption tests conducted to estimate the adsorption behaviors of Cu, Zn, Cr, and Ni adopting Arecanut husk (AH) and Coconut husk (CH) as adsorbents. Parameters examined included contact-time, initial solute and effect of temperature. Contact time (t) varied from ½ hours to 1 day, and initial solute concentrations (C_o) ranged from 5 – 1000 mg/L. The adsorption phenomenon was conducted at three temperatures: 30°C, 40°C, and 50°C. Equilibrium time (t_e) and equilibrium concentration (C_e) were identified by observing the constancy of remaining solute concentration (C_t). Equilibrium refers to the interval at the system achieves chemical equilibrium, with stable concentrations of reactants (Morales-Barrera, et al., 2020). Table 2 summarizes the scope of this experiment.

Table 2: Batch adsorption study

Parameters	Description
Mass of adsorbents m (g)	0.5
Volume of adsorbents V (ml)	50
pH	5.5
Temperature T (0C)	30-40-50
Contact time t (h)	2 hours

In this study, 0.5g dosage of adsorbents was used, and each 50 ml aqueous solution contained in a 100 mL plastic container. The batch studies pH was set to pH (7.0) of the adsorbents. Initial pH measurements of the adsorbents and slurries determined the volume of acid and alkaline solutions used as needed for pH adjustment. The desired pH was achieved by adding these reagents, with regular verification using a calibrated pH meter. In adsorption study, a electric oven maintained the required temperature at a rotational speed of 130 rpm for various contact times. Afterward, the heavy metal solutions prepared using Millipore water (filter size 0.45 µm) (Abhijith L. and Elza Mathew Varghese, 2021). The filtered solution's remaining solution Ct was further diluted with 0.1% nitric acid (HNO_3) to meet the detection limits for Cu, Ni, Cr, and Zn (4 mg/L, 4 mg/L, 3 mg/L, and 2 mg/L, respectively). A Shimadzu AAS7000 atomic absorption spectrometer (Japan), operating with an air-acetylene flame, measured the remaining concentration of each diluted solution. Calibration curves were plotted for each heavy metal content based on its wave-length and standard solutions.

2.3 Sorption Isotherm Models

The batch tests were conducted under the following conditions: synthetic solution pH of 5.5 (± 0.05), bio-sorbent dose of 0.1 g/50 ml, sorbent particle sizes of 75-150 µm and 300-600 µm, contact times of 60 and 120 minutes, a volume of 50 ml for the Cu, Ni, Cr^{3+}, and Zn standard solutions, initial Cu, Ni, Cr^{3+}, and Zn concentrations of approximately 40 mg/l, and room temperature (22 ± 1 °C). These models estimate the maximum capacity for metal uptake from an aqueous solution in an equilibrium batch adsorption system (Awad, et al., 2019). The isotherm models are adopted widely in the literature, in this study, batch equilibrium data for raw bio adsorbent, with particle sizes ranging from 75 µm to 150 µm and an optimal dose of 4 g/l, were analyzed using the Langmuir and Freundlich isotherms. Initial concentrations of Cu, Ni, Cr^{3+}, and Zn varied between 10 and 105 mg/l (James and Yadav, 2021).

3. RESULTS AND DISCUSSION :

3.1 Characterization of Adsorbents

The surface heterogeneities of Arecanut husk (AH) and Coconut husk (CH) particles were examined at a magnification of 500×, as illustrated in Fig. 1 and 2 respectively. It was higlited the surface of Coconut husk (CH) particles was rougher compared to that of Arecanut husk (AH). Basic parameters of the adsorbent microstructures and their natural pH were measured. The specific surface areas of Arecanut husk (AH) and Coconut husk (CH) were 21.84 m^2/g and 18.00 m^2/g, respectively. This suggests that Coconut husk (CH) has a larger specific surface area than Arecanut husk (AH), indicating that it possesses more adsorption locations.

3.2 Effect of Contact -Time

The gradual adsorption of Cu, Zn, Cr, and Ni on Arecanut husk (AH) and Coconut husk (CH) over time is illustrated (Pongsakorn & Panha, 2015) in Figures 3 (a,b,c,d). The removal percentages of heavy metal ions at concentrations varied from 5mg/L, 250mg/L and 500 mg/L were analysed as a function of contact time, ranging from 0.5 to 24 hours. The liquid of pH and temperature were maintained at approximately 6.7 and 30ºC, respectively. It was highlighted that the removal percentage for the 5 mg/L concentration reached around 80%, whereas the uptake for the more concentrations was <20%. Coconut husk (CH) demonstrated higher removal efficiency than Arecanut husk (AH), likely due to its larger surface area and more obtainable adsorptions.

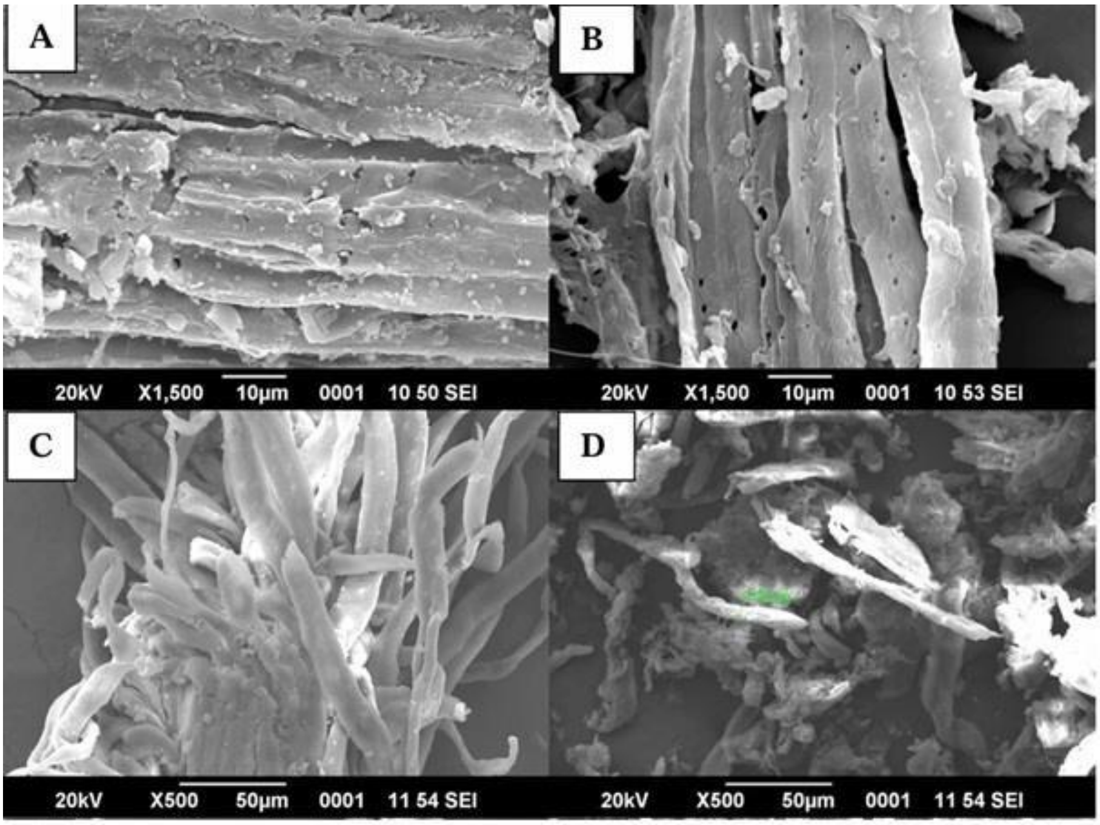


Fig. 1 500x magnification of Arecanut husk (AH)

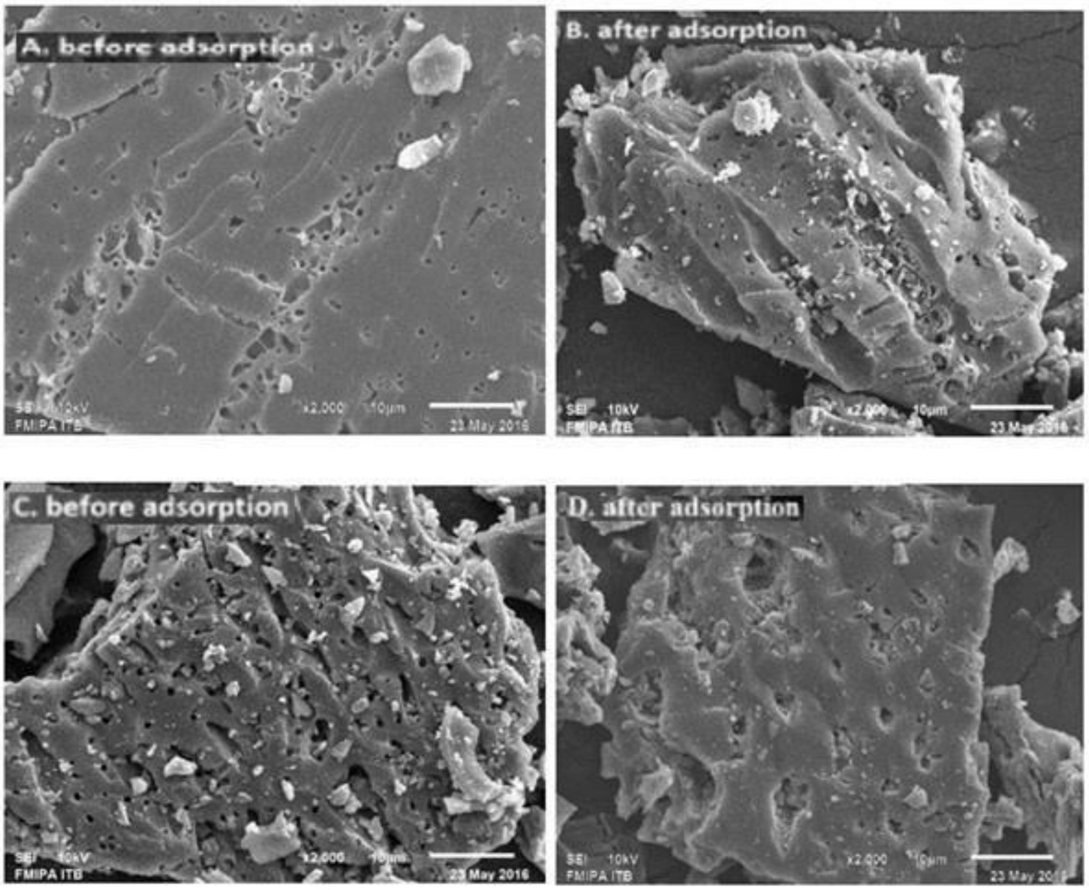


Fig. 2. 500x magnification of Coconut husk (CH)

Initially, the adsorption phenomenon exhibited rapid uptake, followed by a gradual approach to equilibrium, with the removal percentage increasing until it stabilized around 4 hours. This initial rapid uptake is accredited to the abundant availability of adsorption at surface of the adsorbent during the early stages of the process (Gombos, et al., 2012). The adsorption became saturated, the uptake rate slowed and steadied, which can be explained by the slow transport of heavy-metal ions from inside and outside the adsorbent particles of the metals through the micro-pores of adsorbent.

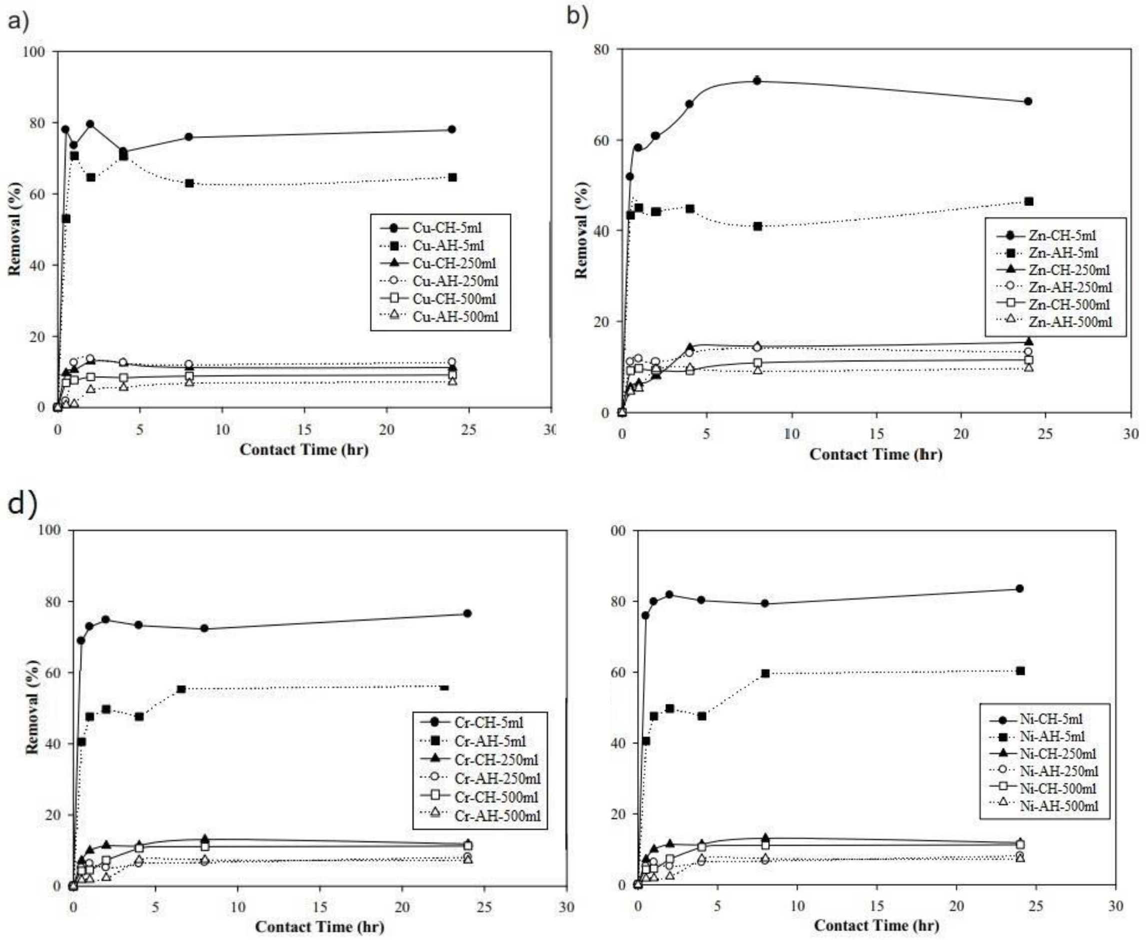


Fig. 3 (a, b, c, d) Effect of contact-time pH=6.7, T=30ºC (Pongsakorn & Panha, 2015)

3.3 Effect of Initial Content

The investigation into the adsorption of Cu, Zn, Cr, and Ni using Arecanut husk (AH) and Coconut husk (CH) as adsorbents on selected soil was focused on the relationship between initial concentrations of heavy-metal ions. To explore the removal proficiency of the adsorbents across varying concentrations, a deliberate gap was established between the lowest and highest initial concentrations. The on-going trends in ion removal percentage (%R) and the quantity of uptake per unit mass of the adsorbents (Qe, mg/g) are illustrated in Figures 4 (a) and 4 (b) respectively (Peng, et al., 2018).

Figure 4 (a) showed the elimination state at an initial content of 5 mg/L reached up to 80.87%, subsequently decreasing to a low of 2.59% at 1000 mg/L. This suggests that the adsorption phenomenon is more effective at lower concentrations, while a decline in removal efficiency occurs at maximum concentrations. This phenomenon may is to be sufficient availability of adsorption sites at lower concentrations, contrasted with a lack of available sites at higher concentrations as the number of metal ions increases (Unuabonah, et al., 2009). This trend is inversely related to the variation in adsorption capacity.

Figure 4 (b) indicates that at the initial content of 5 mg/L, the adsorption capacity began at approximately 0.06 mg/g and demonstrated a progressive increase with rising initial concentrations.

The maximum adsorption capacity observed at an initial concentration of 1000 ppm, approximate 1.525 mg/g, of Zn at 30ºC. This recommends that adsorption capacity is higher at greater initial concentrations, likely due to the enhanced driving strength for mass-transfer as the primary concentration increases (Shi, et al., 2009).

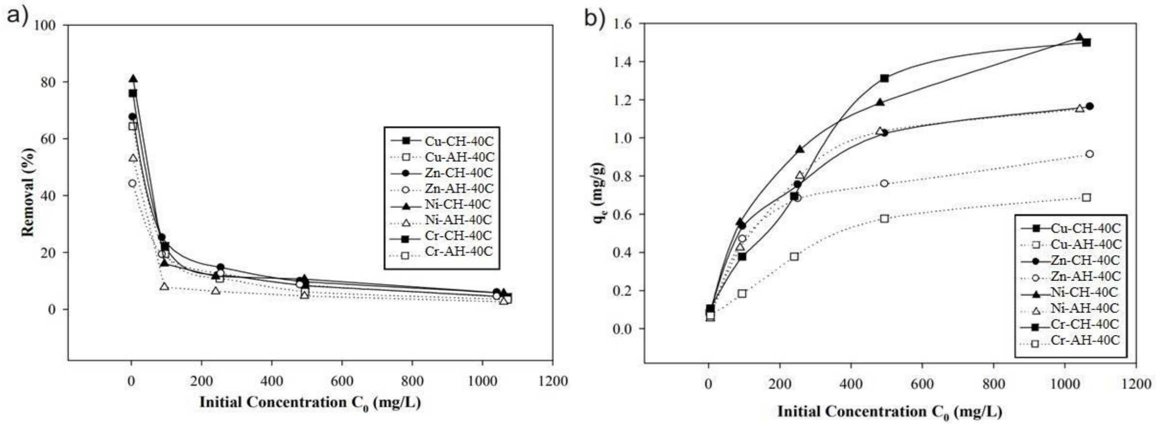
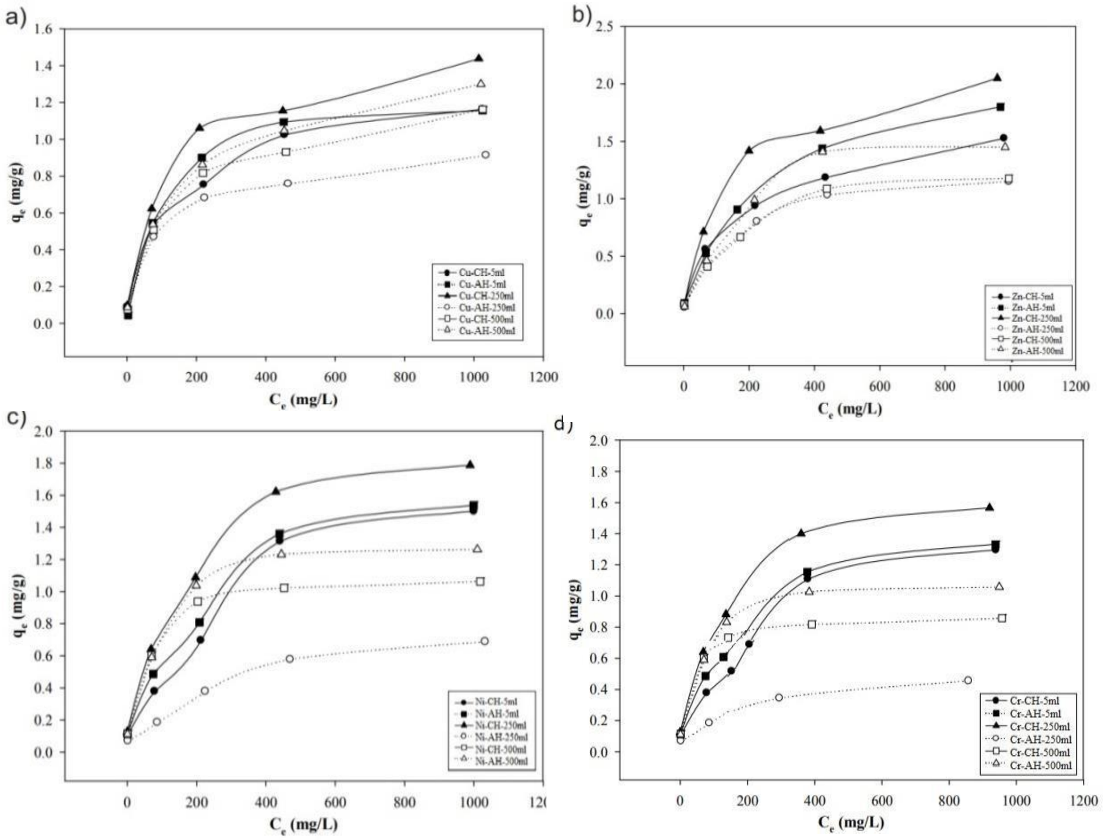


Fig. 4 (a, b) Effect of initial concentration at pH=6.7, T=30ºC. Effect of Temperature

The study of temperature effects was conducted based on the temperature settings typical in Davangere District Karnataka, India. Figures 5 (a, b,c,d) (Pongsakorn & Panha, 2015) show the capacity of adsorption, qe (mg/g) and equilibrium Ce (mg/L) derived using equation $q_e = \frac{(C_0 - C_e)V}{m}$, at temperatures of varied from 30ºC to 50ºC.

Fig. 5 (a,b,c,d) Effect of temperature, isotherm curves at pH=6.7, T=30, 40, 50ºC (Pongsakorn & Panha, 2015)



The results indicate an upward trend in the amount of adsorbed heavy-metal as the equilibrium level increased. At 40ºC and 50ºC, the adsorption isotherms were positioned more than at 30ºC, suggesting improved adsorption efficiency with increasing temperature. This enhancement in adsorption efficiency highlights the significant influence of temperature on this process (Meena, et al., 2008).
The current findings imply that adsorption capacity is temperature-dependent, potentially due to expansion of active surface sites on the adsorbents or the desolvation of adsorbing species. Additionally, a higher temperature can decrease the thickness and boundary-layer around the adsorbent, lowering the mass-transfer resistance in the boundary layer. Another factor may be the accelerated diffusion rate of solute within the adsorbent pores at elevated temperatures (Alinnor, 2007).

3.4 Models of Adsorption Isotherm

The analysis of adsorption isotherms is a commonly effective approach for exploring the theoretical aspects of adsorption processes. The plots that display the adsorbed amounts of metal ions against the equilibrium concentration of solute are referred to as "Isotherm Curves," as shown in Figures 5 (a,b,c,d) (Pongsakorn & Panha, 2015). This study examined the adsorption behavior of four heavy metals (Cu, Zn, Cr, Ni) using two adsorbents. The resulting curves, identified as "L" isotherm curves, showed decrease in the ratio of equilibrium and adsorbed amount on solute content increases.

Table 3. Parameters of Isotherm models

Adsorbates	Adsorbents	$T(^0C)$	Freundlich Parameters			Langmuir Parameters			Q_{0T}/Q_{030}
			K_F	n	R^2	B	Q_0	R^2	
Cu	CH	30	0.089	2.61	0.991	0.011	1.24	0.988	1.00
		40	0.028	1.68	0.942	0.011	1.25	0.998	1.02
		50	0.111	2.55	0.987	0.012	1.49	0.987	1.22
	AH	30	0.068	2.54	0.977	0.013	0.96	0.992	1.00
		40	0.065	2.25	0.981	0.011	1.22	0.977	1.29
		50	0.081	2.41	0.991	0.010	1.31	0.985	1.44
Zn	CH	30	0.077	2.15	0.992	0.009	1.66	0.991	1.00
		40	0.083	2.19	0.995	0.007	2.02	0.988	1.21
		50	0.075	1.96	0.982	0.010	2.22	0.978	1.33
	AH	30	0.036	1.82	0.977	0.009	1.28	0.988	1.00
		40	0.065	2.26	0.992	0.008	1.31	0.985	1.01
		50	0.042	1.80	0.981	0.009	1.64	0.992	1.26
Ni	CH	30	0.098	2.55	0.959	0.008	1.66	0.982	1.00
		40	0.128	2.81	0.982	0.009	1.72	0.978	1.03
		50	0.200	3.12	0.988	0.010	1.94	0.961	1.16
	AH	30	0.043	2.55	0.961	0.006	0.78	0.975	1.00
		40	0.134	3.02	0.972	0.034	1.08	0.977	1.09
		50	0.128	2.78	0.982	0.021	1.29	0.976	1.16
Cr	CH	30	0.096	2.49	0.978	0.007	1.34	0.972	1.00
		40	0.126	2.78	0.988	0.008	1.78	0.977	1.06
		50	0.021	3.10	0.982	0.009	1.69	0.978	1.18
	AH	30	0.044	2.54	0.981	0.010	1.80	0.982	1.00
		40	0.133	3.10	0.976	0.007	0.88	0.981	1.34
		50	0.124	2.77	0.980	0.006	1.34	0.994	1.68

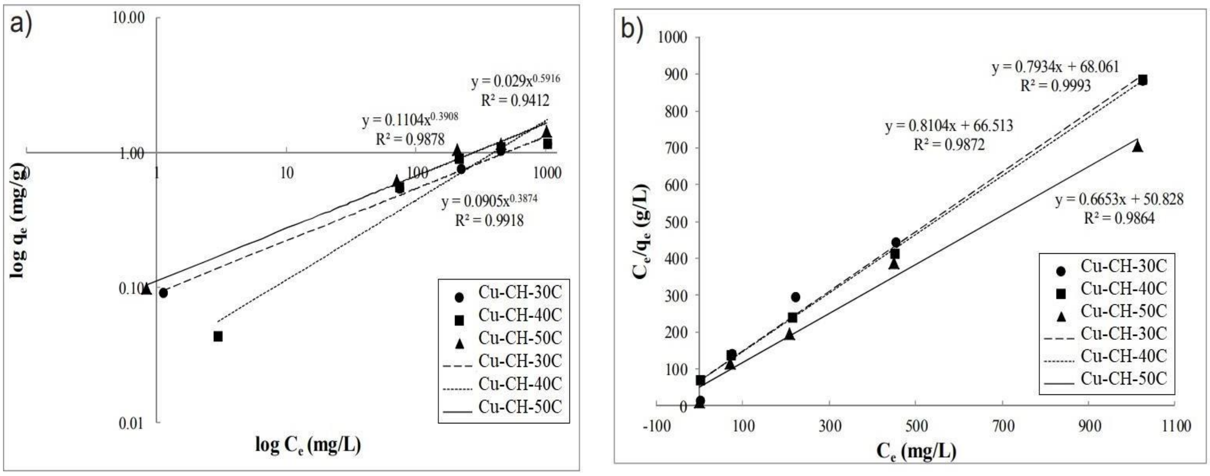


Fig. 6. (a) Freundlich Model **6 (b) Langmuir Model**

To evaluate adsorption behaviour, isotherm models were applied, with equations $q_e = \frac{(C_0 - C_e)V}{m}$, $q_e = \frac{Q^0 b C_e}{1+bC_e}$, $\frac{C_e}{q_e} = \frac{1}{bQ^0} + \frac{C_e}{Q^0}$, $q_e = K_F C_e^{1/n}$ and $\log q_e = \log K_f + \frac{1}{n} \log C_e$ corresponding equations in linear form. Figures 6(a) and 6(b) illustrate the use of these models, specifically for copper adsorption on Coconut husk (CH) at different temperatures (Pongsakorn & Panha, 2015). The adsorption parameters were calculated based on each model's trend lines, with the correlation coefficient (R^2) being a key metric for assessing the fit. These findings are summarized in Table 3.

The Freundlich model's adsorption intensity n ranged from 1.69 to 3.13, indicating that heavy-metal ion adsorption on both AH and CH was effective and primarily physical in nature. According to the Langmuir model, (Veli and Alyuz, 2007; Argun, et al., 2007) the maximum adsorption-capacity of each metal ion followed the order: Cu < Ni < Cr < Zn, with Zn demonstrating the highest sorption affinity. This reflects the varying affinities of metal ions for donor atoms in the adsorbent's structure. Additionally, the adsorption efficiency of yellow loess was found to be superior to that of AH due to its larger surface area (Suna, et al., 2005).

At temperatures of 40ºC and 50ºC, the maximum adsorption capacities were notably higher than at 30ºC, confirming that increased temperature enhances adsorption capacity. This endothermic nature of adsorption for Cu, Zn, Cr, and Ni aligns with similar findings in heavy metal removal studies conducted in China (Bai, et al., 2001). The correlation coefficients R2 for both Freundlich (0.941 to 0.996) and Langmuir (0.945 to 0.999) models (Unuabonah, et al., 2009) suggest that both isothermal models provide an accurate prediction of adsorption condition in this study.

4. CONCLUSIONS :

The adsorption of Cu, Zn, Cr, and Ni on Arecanut husk (AH) and Coconut husk (CH) in soil samples was influenced by contact-time, initial heavy-metal ion content and temperature. Equilibrium was attained for four hours, following an intensive initial uptake. Maximum removal efficiency was noticed at the lowest initial concentration (5 mg/L), while the removal rate declined progressively with higher concentrations (up to 1000 mg/L). Adsorption capacity significantly increased at higher temperatures, with Coconut husk (CH) showing superior performance over Arecanut husk (AH) due to a higher density of surface-active sites. The adsorption capacity in order of trace metals Cu < Ni < Cr < Zn. Isothermal data is showed, fit well with the Freundlich and Langmuir models, demonstrating the adsorbents' substantial adsorption potential. With modifications and treatments to enhance adsorption efficiency, these abundant raw materials could offer even greater probable for heavy-metal removal.

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