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Efficient and Eco-Friendly Removal of Heavy Metals from Wastewater by Low-Cost Adsorbents

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ABSTRACT

The present study emphasizes the utilization ability of less expensive industrial waste adsorbents such as fly ash (FA) to adsorb heavy metals from wastewater r to remove its constituent pollutants, amazing eco-friendly technology guarantees benefits with the decrease in the formation of dangerous solid sludge. In the extended experimental work program: parameters such as pH, contact time, and adsorbent dosages were conducted. The results showed a remarkable treatment equilibrium time after two hours. From all the factors, the pH ranges from 5 to 8 significantly influenced the elimination of heavy metals removal efficiencies, and the highest achieved uptake efficiency during the whole three hours of the experiment period was found to be 93%, 90%, 85%, 79 %, 75 % and 70% for Cu, Fe, Ni, Zn, Pb, and Cd respectively with an optimum fly ash treatment dose of 15 g/L. Based on several performance metrics and visual indicators, different predictive regression-based models for wastewater heavy metals removal efficiencies were developed and compared to the experimental data. The statistical validation indicators revealed that a high correlation was obtained. In addition, the experiment's data were utilized using the Langmuir isotherm model. The results of adsorption data were highly satisfactory statistically match providence to Langmuir heavy metals kinetics removal with mean values of 8%, 11%, and 93% for relative mean absolute error $(MAE)_{rel}$, percent bias (PBIAS), and Nash-Sutcliffe efficiency (NSE) respectively.

Keywords: eco-friendly, fly ash, heavy metals, pollutants, wastewater.

INTRODUCTION

Heavy metals and dyes are prevalent pollutants found in a significant amount of wastewater produced by various industries (Gunatilake et al., 2015). Depending on their initial composition, there are several effective techniques that can be employed for wastewater treatment. Among these techniques, adsorption technologies offer numerous advantages, including the presence of unburnt carbon, low overall cost, minimal sludge production, and simplified operation procedures (Husain et al., 2016). Fly ash (FA) is a complex particulate byproduct that is generated during the combustion of coal in power plants and requires proper disposal (Sillanpaa et al., 2010). FA has proven to be an effective and affordable adsorbent for removing heavy metals from industrial wastewater (Ahmaruzzaman et al., 2016). Volatile fly ash (VFA) has garnered attention as an affordable and readily available option for treating wastewater. Comparative analyses indicate that VFA is proficient at eliminating heavy metals and organic pollutants from water, performing similarly to traditional adsorbents such as activated carbon and zeolites. Studies show VFA achieves a 70–90% removal rate for metals like lead and cadmium, close to the 80–95% rate of activated carbon (Ahmaruzzaman, 2010). Nevertheless, VFA's effectiveness can be affected by its inconsistent composition and the presence of harmful substances, which may pose environmental hazards (Wang et al., 2008). Moreover, the adsorption capacity of VFA can be less than that of artificial adsorbents in certain scenarios, necessitating pre-treatment or enhancement to boost its performance. Despite these limitations, VFA's low cost and plentiful supply make it an attractive option for extensive wastewater treatment, particularly in areas where waste management and resource recovery are crucial issues.

Table 1 illustrates a comparison overview of the efficiency of volatile ash with other commonly used adsorbents, which could highlight its advantages and disadvantages in the context of practical application (Tang et al., 2019).

Extensive efforts have been made to enhance the practical applications of fly ash by employing eco-friendly methods to maximize its adsorption capacity (Sharma et al., 2017). The efficiency of fly ash in eliminating heavy metals from wastewater is primarily influenced by key parameters such as the dosage of fly ash (FA) adsorbent, initial concentration of heavy metals, pH, and contact time (Vishwakarma, 2021). Experimental studies have shown that the efficiency of FA decreases as the initial concentration of heavy metals increases, and vice versa (Kawasaki, 2020). pH is a critical parameter that significantly affects the adsorption process, as it plays a crucial role in determining and controlling the surface charge of the adsorbent (Deng et al., 2018). Within a specific pH range, most heavy metal ions can be effectively adsorbed as the pH levels increase, until a certain threshold is reached. Beyond this threshold, further increases in pH result in negligible metal adsorption (Mirza et al., 2018).

Numerous studies have shown that fly ash has the ability to effectively remove various metallic ions from wastewater, following the Langmuir isotherm trends. The Langmuir isotherm model is commonly used for modeling the adsorption kinetics of fly ash, especially due to its tendency to form a monolayer on the surface. Researchers have also found that heating fly ash to high temperatures can activate it, creating a highly porous material with enhanced contaminant removal capabilities.

This research project focuses on investigating the adsorption behavior of fly ash as a cost-effective adsorbent, specifically targeting the removal of heavy metals such as Cu, Fe, Ni, Zn, Pb, and Cd from industrial wastewater. An extensive experimental program was carried out to monitor various parameters including pH, contact time, and adsorbent dosages in order to determine the optimal treatment conditions.

MATERIALS AND METHODS

Preparation of adsorbent

The solid waste material, raw fly ash, was acquired from a brick factory located in an industrial zone on 6 October City, Giza, Egypt. To ensure the successful implementation of thermal activation, the fly ash samples were subjected to a drying process at 100 °C for two hours prior to conducting tests. Additionally, the samples were sieved to achieve the desired particle size of less than 100 μm before being utilized. The wastewater treatment process utilizing fly ash adsorbent is depicted in Figure 1. To determine the chemical composition of the fly ash, X-ray fluorescence (XRF) analysis was performed using the PANalytical Epsilon3 instrument. The results of the XRF analysis, which provide an overview of the characterization of the fly ash, are presented in Table 2.

Batch study

Heavy metal solutions containing copper, iron, nickel, zinc, lead, and cadmium were prepared by dissolving copper sulfate pentahydrate, iron sulfate heptahydrate, nickel nitrate hexahydrate, zinc chloride hexahydrate, lead nitrate hexahydrate, and cadmium chloride hexahydrate in double distilled water to achieve desired concentrations of the metal ions for the creation of synthetic wastewater. The Langmuir isotherms were determined by mixing metal ion solutions with different adsorbent doses ranging from 5 to 30 g/l, at various contact times between 20 and 180 minutes, under equilibrium pH and 180 rpm, with an initial metal concentration of 5 to 20 mg/l at room temperature.

Adsorption experiments

The adsorbents were combined with 500 ml of distilled water at an adsorbent dose of 5–30 g/l. The pH of the mixture was adjusted to the desired level using 0.1 N HCl and 0.1 N NaOH

Table 1. Adsorbent removal efficiency, advantages, and disadvantages

Adsorbent	Removal efficiency (%)	Advantages	Disadvantages
Volatile fly ash	70–95	Low cost, abundant	Variable composition, potential environmental risks
Activated carbon	80–95	High efficiency, well-studied	Higher cost requires, regeneration
Zeolites	75–90	High selectivity, stable structure	Higher cost, limited by natural availability

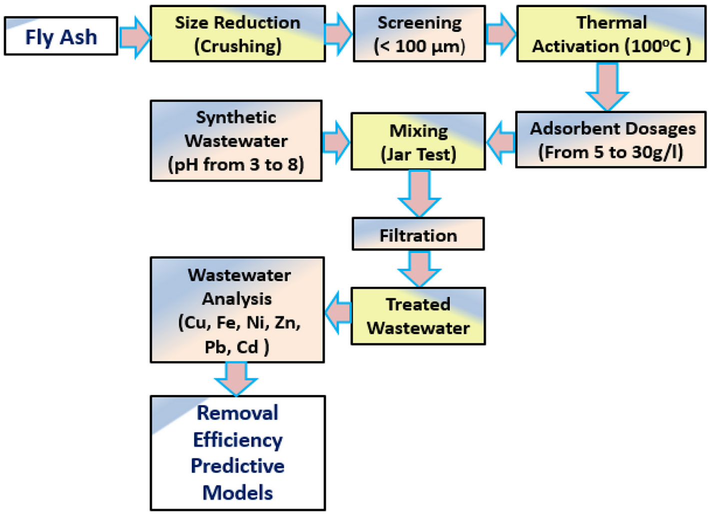

Figure 1. Scheme of the wastewater treatment process using fly ash adsorbent

Table 2. Chemical composition of fly ash (%w/w)

Species	(% w/w)
SiO_2	88.45
Al_2O_3	4.88
Fe_2O_3	4.88
CaO	0.01
MgO	0.15
Loss of ignition (L.O.I)	0.8
Others	0.83

until it reached a stable pH. The mixture was then agitated in a jar test at a temperature of 27 ± 2 °C for one hour. Following this, the prepared concentration of Cu, Fe, Ni, Zn, Pb, and Cd salts was introduced into the bottles to achieve initial concentrations ranging from 5–20 mg/L. The adsorbent was thoroughly stirred and allowed to reach equilibrium for different contact times ranging from 40 to 180 minutes. After the mixing process, the adsorbent particles were separated from the suspensions and filtered using Whatman filter paper of grade 40. The remaining concentration of heavy metals was determined using the GBC Scientific Equipment Ltd.-932 plus-atomic absorption spectrometer.

RESULTS AND DISCUSSIONS

The potential impact of various batch study treatment conditions including pH values, contact times, and FA adsorbent dosages on the wastewater heavy metals removal efficiencies are investigated and analyzed in detail in the following sections.

Effect of adsorbent dosages

Figure 2 illustrates the impact of FA dosage on the removal efficiencies of heavy metals at an initial concentration of 10 mg/l, pH 4, and a contact time of 100 minutes. It is evident that a notable removal efficiency for heavy metals in wastewater was attained, with maximum values of 90.23%, 85.87%, 83.31%, 78.24%, 68.78%, and 65.88% for Cu, Fe, Ni, Zn, Pb, and Cd respectively. Furthermore, a significant increase in removal efficiency was observed when the adsorbent dosage increased from 5g/l to 15g/l, with average percentages of 35.17%, 30.21%, 35.82%, 36.61%, 28.32%, and 30.41% for Cu, Fe, Ni, Zn, Pb, and Cd respectively. This enhancement can be attributed to the rise in fly ash content, leading to an increase in available binding sites for heavy metal ions and thereby enhancing adsorption (Singh et al., 2017). At an adsorbent dose of 15 g/l, a stabilization in treatment conditions was noted, indicating consistency in the uptake of various heavy metals, with removal trends remaining relatively constant thereafter. This could be explained by the lack of sufficient surface area, resulting in a reduction in adsorption capacity due to the limited number of available sites and varying adsorbent dosages within a fixed volume (Marei et al., 2021). Therefore, 15 g/l represents the optimal FA adsorbent dose for achieving maximum treatment efficiency under the specified experimental conditions. These findings align with those of Hegazi (2013), who reported Fe removal rates using fly ash ranging from 46.18% to 86.757%, and Pb removal percentages varying from 21.79% to 76.06%.

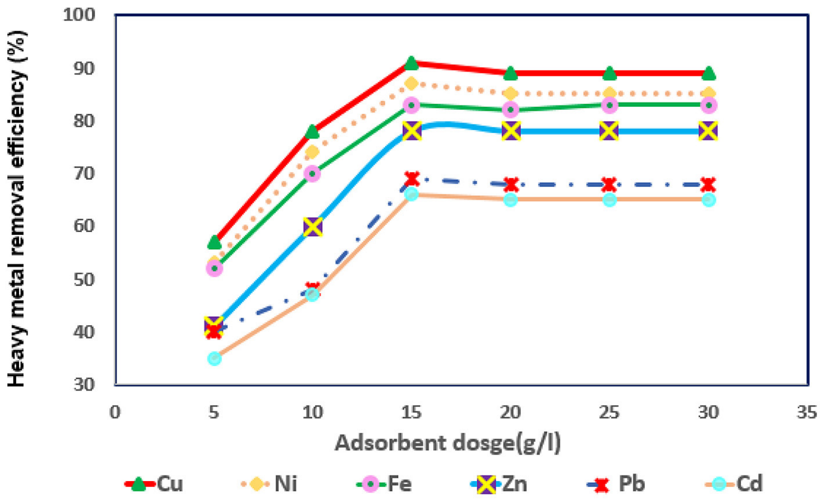

Figure 2. Effect of adsorbent dosage on the heavy metals removal percentage: initial concentration 10 mg/l, pH 4, contact time 100 minutes

Effect of pH

Figure 3 illustrates the efficiency of removing heavy metals at an initial concentration of 10 mg/l, a fly ash dosage of 10 g/l, and a contact time of 90 minutes. The pH of the solution plays a crucial role in enhancing the excess of the required surface adsorbent charge and influencing the ionization processes (Deng et al., 2018). The study revealed that as the pH increased from 3 to 7, the removal percentages for heavy metals were 90.85% for Cu, 86.12% for Fe, 84.25% for Ni, 78.86% for Zn, 69.81% for Pb, and 66.91% for Cd. Interestingly, there was a noticeable increase in metal removal efficiency with rising pH levels until a specific threshold was reached, after which the adsorption rate remained relatively constant (Mirza et al., 2018). Specifically, the removal percentage for Pb ranged from 21.79% to 76.06%. These results agree with that experimentally reported by Shivaprasad et al. (2022) for the adsorption of heavy metals on coal fly ash from aqueous solutions.

Effect of contact times

In a specific time frame ranging from 40 to 180 minutes, the impact of contact time on the absorption of heavy metals was examined. The heavy metal concentrations were maintained at 10 mg/l, with a constant fly ash dosage of 15 g/l and a pH of 5, as illustrated in Figure 4. The data collected indicated that the efficiency of reducing various heavy

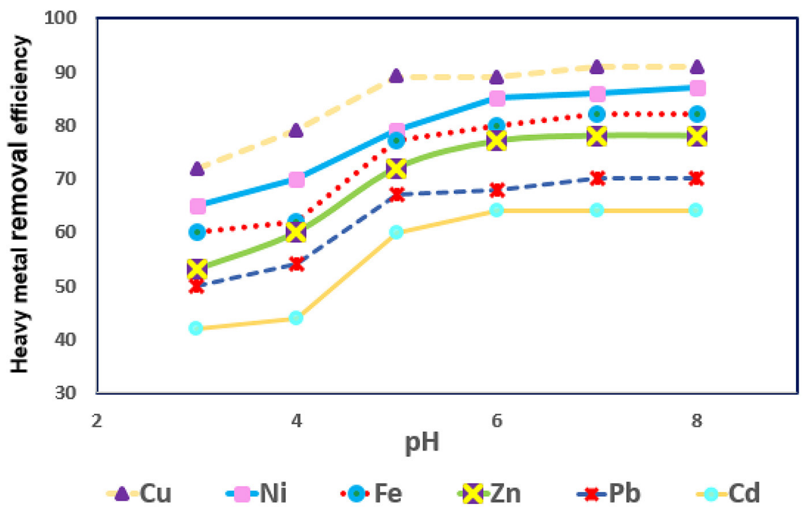

Figure 3. Effect of pH on the heavy metal removal percentage: initial concentration 10 mg/l, fly ash dosage 10 g/l, contact time 90 minutes

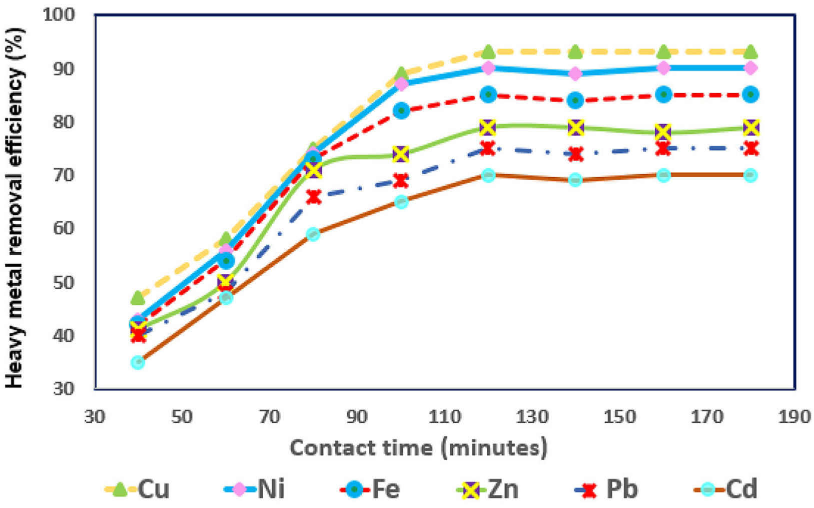

Figure 4. Effect of contact time on the heavy metal removal percentage: initial concentration 10 mg/l, pH 5, fly ash dosage 15 g/l

metal concentrations is directly proportional to the increase in contact time. It reached the equilibrium point after 120 minutes, with a maximum removal efficiency of 93.13%, 90.06%, 85.11%, 79.08%, 75.14%, and 70.07% for Cu, Fe, Ni, Zn, Pb, and Cd, respectively. These findings align with those reported by Saleh et al. (2022) regarding the removal of heavy metals from industrial wastewater using fly ash on a pilot scale.

It can be noted that the use of volatile fly ash) for removing heavy metals such as copper (Cu), iron (Fe), nickel (Ni), zinc (Zn), lead (Pb), and cadmium (Cd) from wastewater holds significant promise due to its high removal efficiency and cost-effectiveness. However, the potential environmental impact must be carefully evaluated.

Removal efficiency predictive models developing

As previously aforementioned the optimum equilibrium contact time for the predetermined experiment's parameters and conditions was successfully obtained after 120 minutes. However, Ridge multiple regression (RR) is a specialized technique was used to analyze the multicollinear experiment's data. The formation of RR prediction model is mainly based on the following Equations

$$y_t = \hat{\beta} x_t + \epsilon_t \tag{1}$$

$$\hat{\beta}_\lambda = argmin_\beta \left[\sum_{t=1}^{n} (y_t - \hat{\beta} x_t)^2 + \lambda \hat{\beta} \beta \right] \tag{2}$$

$$\hat{\beta}_\lambda = \left[\sum_{t=1}^{n} x_t x_t + \acute{} \lambda I_k \right]^{-1} \left(\sum_{t=1}^{n} x_t y_t \right) \tag{3}$$

where: β is the coefficient vector, λ is the ridge parameter that has k x k identity matrix, and $\lambda > 0$.

Table 3 illustrates the used codes for the other affecting experiment factors: pH and adsorbent dose (D). Moreover, soft computing and regression-based models were used to develop the six RR predictive models: M_{Cu}, M_{Fe}, M_{Cu}, M_{Ni}, M_{Zn}, and M_{Cd} for Cu, Fe, Ni, Zn, Pb, and Cd removal efficiency percentage estimation respectively. The following shows the deduced Equation for the aforementioned proposed models:

$$M_{Cu} = 70.023 - 38.47pH_3 - 1.786\,pH_4 + 1.865pH_5 + \\ + 21.979pH_6 + 22.648pH_7 - 21.425pH_8 + \\ + 1.954pH_3 \times D_1 + 1.974pH_4 \times D_1 - 2.643pH_5 \times D_2 \\ + 2.587pH_5 \times D_3 - 1.643pH_6 \times D_1 - 1.395pH_6 \times D_2 + \\ + 0.854\,pH_6 \times D_3 - 0.396pH_6 \times D_4 - 0.004pH_7 \times D_3 - \\ - 0.002pH_7 \times D_4 + 0.003pH_7 \times D_5 + 0.0005pH_8 \times D_5 \\ - 0.0006pH_8 \times D_5 R^2 = 0.956 \tag{4}$$

Table 3. The code used in predictive model

pH	Value	3	4	5	6	7	8
	Code	pH_3	pH_4	pH_5	pH_6	pH_7	pH_8
Adsorbent dose	Value	5g/l	10g/l	15g/l	20g/l	25g/l	30g/l
	Code	D_1	D_2	D_3	D_4	D_5	D_6

$$M_{Ni} = 61.014 – 32.55pH_3 – 1.933pH_4 + 1.543pH_5 + 21.972pH_6 + 22.606pH_7 – 22.521pH_8 + 1.711pH_3 \times D_1 + 1.853pH_4 \times D_1 - 2.611pH_5 \times D_2 + 1.913pH_5 \times D_3 – 1.8352pH_6 \times D_1 - 1.415pH_6 \times D_2 + 0.765pH_6 \times D_3 – 0.513pH_6 \times D_4 - 0.005pH_7 \times D_3 – 0.003pH_7 \times D_4 + 0.003pH_7 \times D_5 + 0.0004pH_8 \times D_5 – 0.0007pH_8 \times D_5 R^2 = 0.965 \quad (5)$$

$$M_{Fe} = 56.023 – 37.39pH_3 – 1.779pH_4 + 1.799pH_5 + 21.879pH_6 + 22.593pH_7 – 21.609pH_8 + 1.837pH_3 \times D_1 + 1.988pH_4 \times D_1 – 2.584pH_5 \times D_2 + 2.603pH_5 \times D_3 – 1.712pH_6 \times D_1 – 1.395pH_6 \times D_2 + 0.854 pH_6 \times D_3 – 0.405pH_6 \times D_4 – 0.004pH_7 \times D_3 – 0.003 pH_7 \times D_4 + 0.003pH_7 \times D_5 + 0.0004pH_8 \times D_5 – 0.0007 pH_8 \times D_5 R^2 = 0.932 \quad (6)$$

$$M_{Zn} = 53.958 – 36.13 pH_3 – 1.811 pH_4 + 1.745 pH_5 + 20.569pH_6 + 23.044pH_7 – 20.337pH_8 + 1.794pH_3 \times D_1 + 1.974pH_4 \times D_1 – 2.701pH_5 \times D_2 + 2.587pH_5 \times D_3 – 1.698pH_6 \times D_1 – 1.395pH_6 \times D_2 + 0.843 pH_6 \times D_3 – 0.396pH_6 \times D_4 – 0.004pH_7 \times D_3 – 0.002pH_7 \times D_4 + 0.003pH_7 \times D_5 + 0.0005pH_8 \times D_5 – 0.0006pH_8 \times D_5 R^2 = 0.928 \quad (7)$$

$$M_{Pb} = 48.958 – 37.98pH_3 – 1.993pH_4 + 1.467pH_5 + 22.114pH_6 + 23.008pH_7 – 20.418pH_8 + 1.526pH_3 \times D_1 + 1.666pH_4 \times D_1 – 2.741pH_5 \times D_2 + 1.822pH_5 \times D_3 – 1.905pH_6 \times D_1 – 1.522pH_6 \times D_2 + 0.618pH_6 \times D_3 – 0.663pH_6 \times D_4 – 0.004pH_7 \times D_3 – 0.006pH_7 \times D_4 + 0.005pH_7 \times D_5 + 0.0003pH_8 \times D_5 – 0.0008pH_8 \times D_5 R^2 = 0.937 \quad (8)$$

$$M_{Cd} = 44.723 – 35.88pH_3 – 1.782pH_4 + 1.639pH_5 + 21.092pH_6 + 22.102pH_7 – 20.337pH_8 + 1.811pH_3 \times D_1 + 1.659pH_4 \times D_1 – 2.833pH_5 \times D_2 + 2.423pH_5 \times D_3 – 1.722pH_6 \times D_1 – 1.408pH_6 \times D_2 + 0.743pH_6 \times D_3 – 0.422pH_6 \times D_4 – 0.003pH_7 \times D_3 - 0.006pH_7 \times D_4 + 0.006pH_7 \times D_5 + 0.0007pH_8 \times D_5 - 0.0008pH_8 \times D_5 R^2 = 0.907 \quad (9)$$

The obtained data strongly agreed with the Ridge multiple regression model, with high confidential accuracy as noted from a high Pearson's correlation coefficient (R^2) value, implying a substantial correspondence with the different developed heavy metal removal efficiency models.

Adsorption isotherm

This study utilized Langmuir isotherm to model the adsorption tests at equilibrium. Langmuir isotherm is mainly based on the assumption that the optimum adsorption corresponds to a saturated monolayer of adsorbate molecules on the adsorbent surface and the adsorption energy is constant. Langmuir Equation is defined as (Fan et al. 2019):

$$q_e = (q_m K_L C_e)/(1 + K_L C_e) \quad (10)$$

The linear expression of the Langmuir adsorption isotherm Equation is presented as:

$$1/q_e = (1/q_m) + (1/q_m K_L)(1/C_e) \quad (11)$$

K_L (l/mg) is the Langmuir constant related to adsorption energy, Ce is the equilibrium concentration in mg/l, and *Qe* is the adsorbate amount adsorbent per unit weight (mg/g). Table 4 shows the Langmuir constants for the sorption of different heavy metals onto FA adsorbent at 27 ± 2 °C. In addition, Figure 5 illustrates the adsorption of various heavy metals onto FA represented in the linear expression of the Langmuir adsorption isotherm.

On the other hand, to ensure the accuracy of the experiment's heavy metal removal efficiency data upon the previously applied Langmuir isotherm model, three statistical measures were selected to evaluate the accuracy of the applied experiment's heavy metals data upon Langmuir isotherm model:

- Relative mean absolute error $(MAE)_{rel}$ – the MAE_{rel} can be estimated as:

$$MAE = \left[\frac{1}{n}\sum_{i=1}^{n}|Y_{Observied} - Y_{Simulated}|\right] \quad (12)$$

$$(MAE)_{rel} = \frac{MAE}{\overline{Y_{Observied}}} \quad (13)$$

- Percent bias (*PBIAS*) – a particular value of zero represent the most optimum value for *PBIAS* and this statistical measures can be calculated as:

$$PBIAS = 100 \times \frac{\sum_{i=1}^{n}(Y_{Observied} - Y_{Simulated})}{\sum_{i=1}^{n} Y_{Observied}} \quad (14)$$

- Nash-Sutcliffe efficiency (*NSE*) – generally, the *NSE* values range from zero to one with an optimum value of one. *NSE* can be calculated as :

$$NSE = 1 - \left[\frac{\sum_{i=1}^{n}(Y_{Observied} - Y_{Simulated})^2}{\sum_{i=1}^{n}(Y_{Observied} - \overline{Y_{Observied}})^2}\right] \quad (15)$$

Table 5 shows the statistical evaluation measures of the observed experiment's heavy metal removal efficiency data compliance upon the

Table 4. Langmuir constants for the sorption of different heavy metals onto FA adsorbent

Heavy metals	Langmuir constants	
	Q_m (mg/g)	K_L(L/mg)
Cu	0.271	0.047
Fe	0.195	0.039
Ni	0.258	0.052
Zn	0.024	0.373
Pb	0.011	0.489
Cd	0.041	1.112

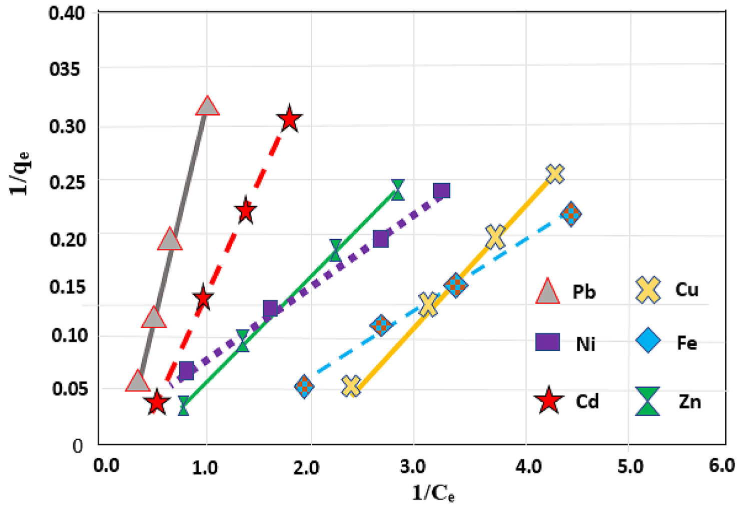

Figure 5. Langmuir isotherm model for various heavy metals onto fly ash

Table 5. The statistical evaluation measures

Heavy metals	MAE_{rel}	PBIAS	NSE
Cu	0.08	0.11	0.95
Fe	0.07	0.13	0.92
Ni	0.09	0.11	0.91
Zn	0.08	0.10	0.93
Pb	0.11	0.12	0.94
Cd	0.06	0.09	0.95

previously applied Langmuir isotherm model. The study's heavy metals removal efficiency data fit well with the Langmuir isotherm model. However, a noted recommended fitness value (zero) for MAPE and PBIAS was obtained. Moreover, NSE results also tend to have the optimum compliance value (1.00). The adsorption data has a high agreement with the Langmuir model, as indicated by a high agreement with various statistical evaluation measures values, suggesting a strong fit with the model. Our results are closest to those produced by Seo et al. (2020) for the fly ash investigation on heavy metal removal.

CONCLUSIONS

In this study, the potential influence evaluation of the various eco-friendly treatment conditions alternatives of fly ash as a low-cost adsorbent for wastewater treatment was experimentally investigated. The study results successfully set the outline determination of wastewater heavy metal treatment optimum equilibrium conditions that can be attained within the optimum pH range for heavy metal adsorption from 5 to 8, two hours of contact time, and a 15 g/l fly ash adsorbent optimum dose. Consequently, at these distinctive conditions, the maximum obtained heavy metals removal efficiencies were 93%, 90%, 85%, 79 %, 75 %, and 70% for Cu, Fe, Ni, Zn, Pb, and Cd respectively. One of the other major outcomes from this study is the proposed heavy metals removal efficiency regression-based model that performs adequately accurate performance with a high Pearson's correlation coefficient (R^2) value of more than 90%. In addition, the experiment's data were utilized using the Langmuir isotherm model, and the results of adsorption data were correlated well with highly satisfactory match providence to Langmuir heavy metals kinetics removal. However, achieving such removal efficiencies within a noted time limit can highly appreciate proposing distinctive future improvement opportunities in applying this eco-environmental system.

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