

PERFORMANCE AND ERROR EVALUATION OF TWO-PARAMETER ADSORPTION MODELS FOR ZINC ION REMOVAL VIA FLY ASH

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ABSTRACT

In the present investigations, a series of batch experiments were conducted to analyse the adsorption behaviour of fly ash using eight two-parameter adsorption isotherm models. Each isotherm model was assessed using eleven distinct error functions to determine the most suitable model that can be used to design the adsorption process. The analysis relied on minimizing error values as the primary metric for model performance evaluation. Based on the analysis of error values, it was concluded that the ranking of the different isotherm models (in terms of accuracy and relative performance) is as follows: Temkin, Freundlich, Frenkel-Halsey-Hill, Frumkin, Elovich, Langmuir, Jovanovic, and Harkins-Jura. Amongst these, the Temkin isotherm emerged as the most reliable model for representing the adsorption process, whereas the Harkins-Jura model showed the least accuracy with the experimental data. These findings highlight the importance of error function analysis in accurately ranking isotherm models and selecting the most appropriate isotherm for specific adsorption studies.

***Keywords:** error matrix, regression analysis, two-parameter adsorption models, zinc ions.*

INTRODUCTION

The usual and the most neglected cause of water pollution due to heavy metals is the discharge from the mining and metal industries. Heavy metals also enter the aquatic system by natural processes, which include the leaching of rocks, minerals, soils, vegetation, etc. [1, 2]. The toxic effects of heavy metal ions have been a concern for researchers and environmentalists working for the health and environment [3]. Metal ions are known to have adverse effects on health and the environment, as well as significant economic implications when their concentrations exceed permissible limits [4].

Zinc is an essential element for the life and growth of plants and animals. However, its excess uptake may result in various health issues. Also, its deficiency may result in loss of appetite, anaemia, depressed growth, diarrhea, hair loss, eczema, etc. The standard given by

the Central Pollution Control Board of India recommends that industrial effluents discharged into inland surface water should not have a zinc concentration of more than 5 mg L⁻¹ [5]. Effective treatment of wastewater effluents is essential to mitigate the harmful effects of toxic metals before their discharge into surface water bodies.

However, in recent years, many chemical and biological treatment processes have been developed and tested successfully for the removal of toxic metals from wastewater [6 - 9]. Among the various available methods, the adsorption of metal ions onto the surface of low-cost adsorbents is regarded as an efficient and cost-effective technique for removing toxic metal ions from wastewater [10, 11]. The findings from multiple studies suggest that fly ash shows promising potential as an adsorbent for the removal of heavy metals from industrial wastewater [12 - 14]. However, it is essential to study the characteristics of fly ash, which was employed

as the adsorbent to probe its equilibrium uptake of Zn from aqueous solutions. In the current study, multiple two-parameter isotherm models were fitted to the adsorption data, and their adsorption constants were evaluated and assessed via a thorough error analysis.

EXPERIMENTAL

Adsorbent used

The fly ash was used as an adsorbent and was collected from a Thermal Power Plant located in central India. Its chemical composition indicates that SiO_2 , Al_2O_3 , and Fe_2O_3 together make up about 91 % of the total fly ash content, while Fe_2O_3 and CaO compose about 4.39 %. The size distribution of fly ash indicates that about 8.88 % of particles are of size above 75 μm and its surface area was measured using the liquid nitrogen adsorption technique, yielding a value of $10.58 \pm 0.143 \text{ m}^2 \text{ g}^{-1}$.

Batch adsorption studies

For designing an adsorption process, it is crucial to analyse isotherm data that accurately represents the results through mathematical models, enabling precise interpretation [15]. These model assumptions play a key role in examining the equilibrium established between the adsorbent and the adsorbate [16].

To determine the adsorption capacity of the adsorbent at equilibrium, it is necessary to study the various adsorption isotherms. The isotherms also establish relationships between adsorbent and adsorbate at equilibrium. These isotherm models describe the empirical relationship to predict the adsorption behaviour of an adsorbent solid surface [17]. They also indicate how efficiently and feasibly an adsorbent can be used for various business applications for the precise substance [18, 19]. Such models are usually formulated based on certain assumptions [20]. The model assumptions are widely used to study the equilibrium established between an adsorbent and adsorbate [16].

To study the adsorption behaviour, batch adsorption tests were carried out by mixing 10 g of fly ash with 100 mL of a 20 mg L^{-1} Zinc solution in glass bottles and agitating over various intervals to identify the equilibrium contact time, which was found to be 3 h. For the isotherm measurements, Zn solutions having concentration of 20, 40, 60, 100, and 200 mg L^{-1} were

prepared, and each solution was stirred with 10 g of fly ash per 100 mL for 3 h. The solution pH was 5.89 and was held constant throughout all experiments.

The zinc uptake by fly ash (in mg g^{-1}) was calculated [11] using the Eq. (1):

$$q_e = \frac{(C_0 - C_e) * V}{W} \quad (1)$$

where: V and W represent the volume of the solution (L) and mass of fly ash used (g), respectively; C_0 and C_e are the initial and equilibrium zinc concentrations (mg L^{-1}).

To study the behaviour of an adsorbent and adsorbate, eight different two-parameter isotherms are analysed. These are briefly discussed below:

Langmuir model

The first two-parameter isotherm was proposed by Langmuir in 1916. In his proposed model he assumed monomolecular adsorption on homogeneous surfaces of adsorbent [21].

This isotherm model is derived assuming that the force of interaction is negligible between different adsorbed molecules and if an adsorption site is occupied by a molecule, no further adsorption takes place. This isotherm can be represented in analytical form as it is presented in Eq. (1).

This equation can also be simplified as given in Eq. (2):

$$q_e = \frac{q_m K_a C_e}{1 + K_a C_e} \quad (2)$$

where K_a (L mg^{-1}) and q_m are empirical constants and depends upon monolayer capacity.

Freundlich model

This adsorption isotherm establishes a relationship between the concentration of an adsorbate and adsorbent [22]. This model is also used for multilayer adsorption on heterogeneous surfaces. The standard form of this model is represented in Eq. (3):

$$q_e = K_f C_e^{1/n} \quad (3)$$

where, K_f is the Freundlich characteristic constant which depends on the relative adsorption capacity, $1/n$ is the heterogeneity factor of sorption and n represents the intensity of adsorption.

Temkin model

This model assumes that adsorption heat is linearly related with coverage interactions between adsorbent-adsorbate for all the molecules in layer [23]. This model ignores the concentrations which are either extremely large or extremely low. The mathematical model of Temkin Adsorption Isotherm in linear form is represented in Eq. (4):

$$q_e = \frac{RT}{b_T} \ln A_T + \frac{RT}{b_T} \ln C_e \quad (4)$$

where, A_T is equilibrium binding constant, T is absolute temperature, R is gas constant and b_T is a constant related to adsorption heat.

Harkins Jura model

This model assumes the possibility of multilayer adsorption on the porous heterogeneous surface of the adsorbent [24]. Mathematically, in its linear form, it is expressed in Eq. (5):

$$\frac{1}{q_e^2} = \frac{B}{A} - \frac{1}{A} \ln C_e \quad (5)$$

where A and B are Harkins-Jura constant and considers multilayer adsorption and heterogeneous pore distribution. The parameters A and B are obtained from the plots of $1/q_e^2$ against $\ln C_e$.

Elovich model

According to this isotherm, the count of adsorption sites grows exponentially as adsorption proceeds, signifying the formation of multiple layers [25]. The relationship is given by Eq. (6):

$$\frac{q_e}{q_m} = K_E C_e \left(e^{-\frac{q_e}{q_m}} \right) \quad (6)$$

Frenkel-Halsey-Hill model

This isotherm is suitable for multilayer adsorption and can be expressed as it is presented in Eq. (7):

$$q_e = e^{\left(\frac{h K_H - h C_e}{n} \right)} \quad (7)$$

where isotherm constants are represented by K_H and n . To verify this isotherm, a graph is plotted between $\ln q_e$ versus $\ln C_e$. The adsorbent is assumed to be heterogeneous if this plot is found to be linear [26].

Jovanovic model

This model assumes the possibility of some mechanical contacts between adsorbate and adsorbent molecules [27]. Mathematically, this model can be represented as it is presented in Eq. (8):

$$q_e = q_m (1 - e^{-K_J C_e}) \quad (8)$$

where K_J ($L g^{-1}$) is Jovanovic adsorption constant. The value of K_J and q_m is obtained from a plot of $\ln q_e$ and C_e .

Frumkin model

This adsorption isotherm model assumes that an interaction in the adsorption layer obeys Frumkin's isotherm if adsorption takes place on a homogeneous surface [28]. The equation of Frumkin isotherm can be expressed in Eq. (9):

$$\left(\frac{q_e}{1 - q_e} \right) e^{2aq_e} = K C_e \quad (9)$$

where a describes the interaction in the adsorbed layer and its value can be positive or negative. The negative value of ' a ' means the force between the molecules in the adsorbed layer is negative and its reverse is true for its positive value.

Error functions

After laboratory investigations and analytical calculations, the primary challenge lies in analysing the errors present in the results. To address this issue, this paper contributes an extensive evaluation of error metrics. A total of eleven different error functions has been detailed in Table 1. These error functions are employed to signify the performance of output received from laboratory investigations and isotherm models, indicating the best isotherm that should be used to design the adsorption process.

RESULTS AND DISCUSSION**Batch studies**

A set of adsorption tests was carried out by agitating 10 g of fly ash in 100 mL of a 20 mg L^{-1} Zn solution at 150 rpm. Samples were taken over intervals from 5 minutes up to 4 h to determine the equilibrium time. Following each experimental run, the adsorbent was carefully separated from the solution, and the residual solution was analysed to quantify the concentration of

Table 1. Error functions name and equation.

Sr. no.	Error function name (Abbreviation), Range and preferred value	Error function equation
1	Max Error (ME), Smaller is better (Best = 0) [29]	$ME(y_t, y_p) = (y_t - y_p)$
2	Mean Absolute Error (MAE), Smaller is better (Best = 0) [30]	$MAE(y_t, y_p) = \frac{\sum_{i=0}^{N-1} y_t^i - y_p^i }{N}$
3	Root Mean Squared Error (RMSE), Smaller is better (Best = 0) [31]	$RMSE(y_t, y_p) = \sqrt{\frac{\sum_{i=0}^{N-1} (y_t^i - y_p^i)^2}{N}}$
4	Mean Squared Error (MSE), Smaller is better (Best = 0) [32]	$MSE(y_t, y_p) = \frac{\sum_{i=0}^{N-1} (y_t^i - y_p^i)^2}{N}$
5	Mean Relative Error (MRE), Smaller is better (Best = 0) [33]	$MRE(y_t, y_p) = \frac{1}{N} \sum_{i=0}^{N-1} \frac{ y_t^i - y_p^i }{ y_t^i }$
6	Confidence Index (CI), Bigger is better (Best = 1) [34]	$CI(y_t, y_p) = AR(y_t, y_p) \times WI(y_t, y_p)$
7	Efficiency Coefficient (EC), Bigger is better (Best = 1) [35]	$EC(y_t, y_p) = 1 - \frac{\sum_{i=1}^N (y_t, y_p)^2}{\sum_{i=1}^N (y_t^i - \text{mean}(y_t))^2}$
8	Explained Variance Score (EVS), Bigger is better (Best = 1) [36]	$EVS(y_t, y_p) = 1 - \left\{ \frac{\text{Var}\{y_t - y_p\}}{\text{Var}\{y_t\}} \right\}$
9	Coefficient of Determination (R^2), Bigger is better (Best = 1) [37]	$R^2(y_t, y_p) = 1 - \frac{\sum_{i=1}^N (y_t^i - y_p^i)^2}{\sum_{i=1}^N (y_t^i - \text{mean}(y_t))^2}$
10	Relative Absolute Error (RAE), Smaller is better (Best = 0) [38]	$RAE(y_t, y_p) = \frac{\sqrt{\sum_{i=1}^N (y_p^i - y_t^i)^2}}{\sqrt{\sum_{i=1}^N (y_t^i)^2}}$
11	Correlation (COR), Bigger is better (Best = 1) [35]	$COR(y_t, y_p) = \frac{COV(y_t, y_p)}{std(y_t) \times std(y_p)}$

zinc ions using an Atomic Absorption Spectrophotometer (AAS) (Model-GBC 932 AA). The collected data were subsequently assessed, culminating in the construction of a graph that delineates the correlation between the adsorption efficiency of fly ash and the contact time, as depicted in Fig. 1.

Fig. 1 provides a comprehensive depiction of the time-dependent behaviour of zinc ion adsorption onto fly ash. The initial phase exhibits a rapid adsorption rate, which gradually declines as the contact time increases. This trend is evident from the observation that the adsorption capacity of fly ash reached 53.2 %

within the first 30 min of contact and further increased to approximately 70 % after 3 h of continuous stirring under constant conditions. Beyond this point, no significant improvement in adsorption capacity was observed, indicating that the system had achieved equilibrium.

The rapid initial adsorption rate can be attributed to the high availability of vacant adsorption sites on the surface of the fly ash at the start of the process. These sites readily interact with zinc ions in the aqueous solution, leading to a steep increase in adsorption efficiency. However, as the process progresses, the number of unoccupied sites diminishes, resulting in a slower rate of adsorption. Over time, a dynamic equilibrium is established, wherein the rate of zinc ion adsorption becomes equal to the rate of desorption. This equilibrium state reflects the saturation of adsorption sites and the stabilization of the adsorption process. Based on the findings of this study, the equilibrium time for zinc ion adsorption onto fly ash was determined to be 3 h.

Adsorption isotherm study

Adsorption isotherms delineate the intricate relationship between the properties of the adsorbate and the adsorbent. To examine the behaviour of adsorption isotherms, aqueous solutions with a pH of 5.89, containing zinc ions at various concentrations were transferred into a glass flask containing 10 g of fly ash per 100 mL of solution and subjected to uniform agitation for three h (the equilibrium time for adsorption) under ambient conditions ($25^{\circ}\text{C} \pm 1$).

Following the designated period, the adsorbent was carefully separated, and the residual solution was analysed to ascertain the final zinc ion concentration (C_e). Subsequently, the adsorption capacity (q_e) was computed, with the corresponding values of C_0 , as summarized in Table 2.

To study the adsorption behaviour, eight different two-parameter adsorption isotherms are plotted and are shown in Fig. 2 to Fig. 9.

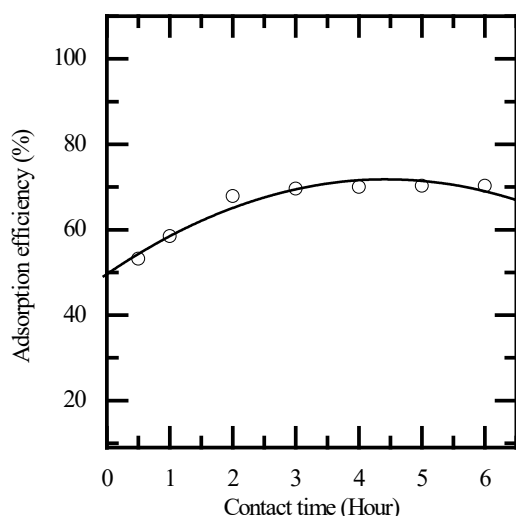


Fig. 1. Effect of contact time on adsorption efficiency.

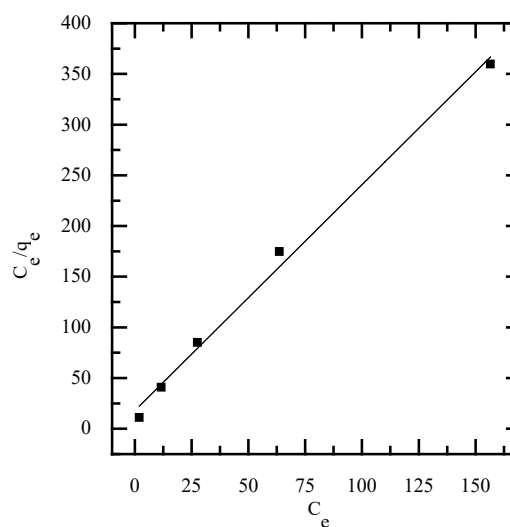


Fig. 2. Plot of Langmuir adsorption isotherm.

Table 2. Zinc ion concentration at $t = 0$ and at $t = 180$ min (equilibrium time).

Initial ion conc., $t = 0$	C_0 , mg L ⁻¹	20	40	60	100	200
Ion conc. at $t = 180$ min	C_e , mg L ⁻¹	2	11.6	27.6	63.6	156.5
Adsorption capacity at equilibrium time, $t = 180$ min	q_e , mg g ⁻¹	0.18	0.284	0.324	0.364	0.435

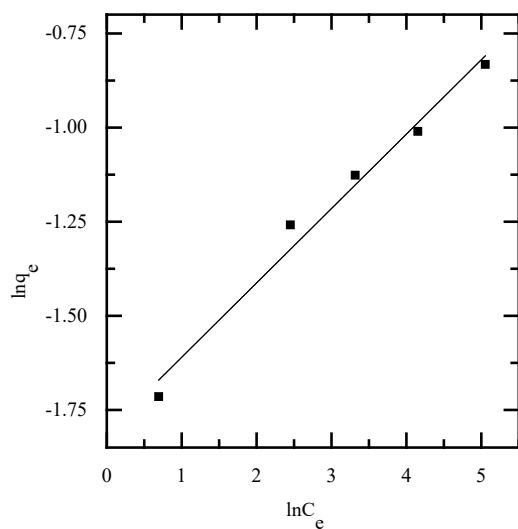


Fig. 3. Plot of Freundlich adsorption isotherm.

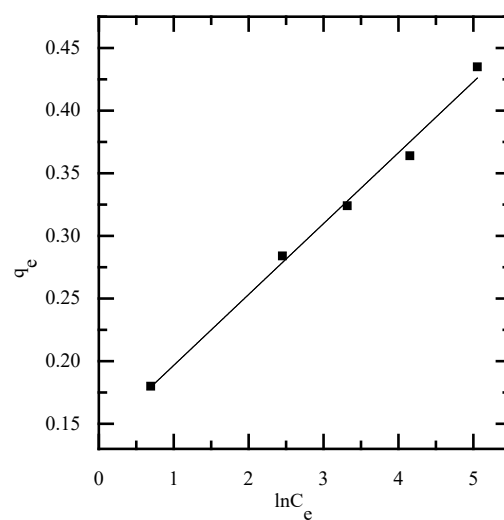


Fig. 4. Plot of Temkin adsorption isotherm.

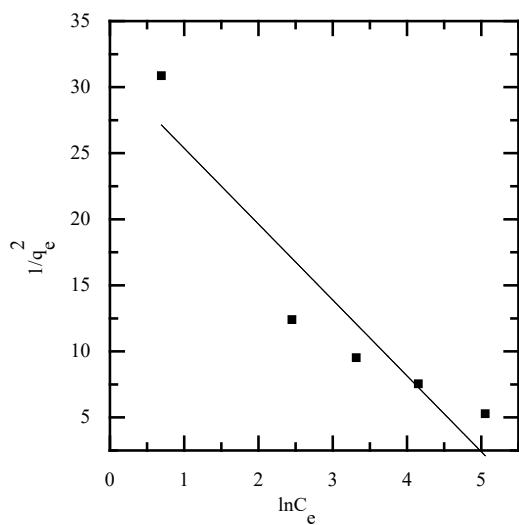


Fig. 5. Plot of Harkins Jura adsorption isotherm.

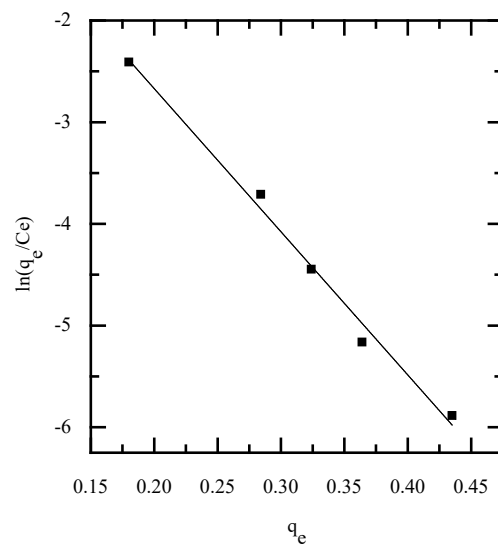


Fig. 6. Plot of Elovich adsorption isotherm.

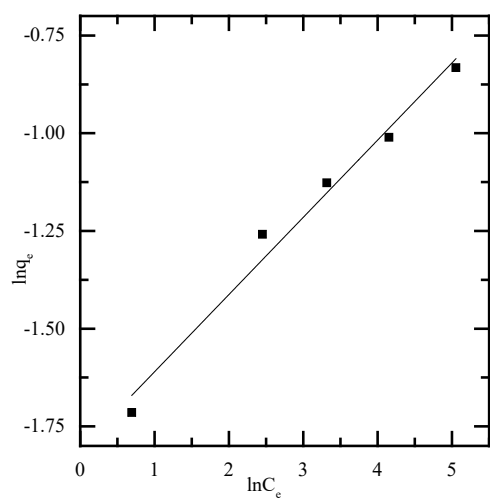


Fig. 7. Plot of Frenkel-Halsey-Hill adsorption isotherm.

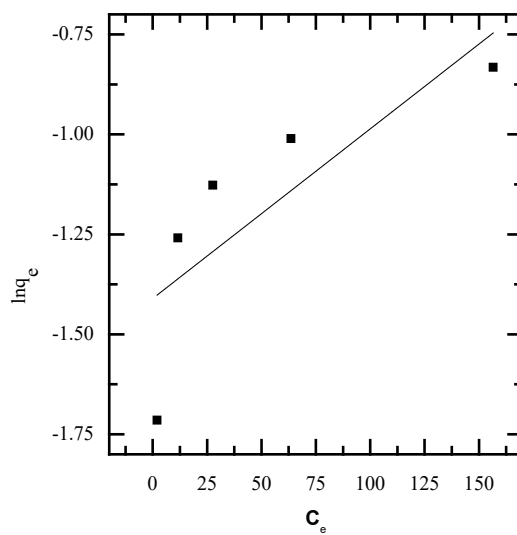


Fig. 8. Plot of Jovanovic adsorption isotherm.

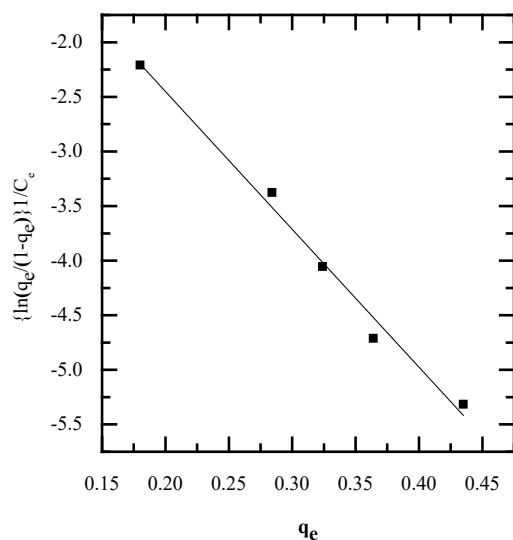


Fig. 9. Plot of Frumkin adsorption isotherm.

By fitting a straight line to each isotherm plot, a regression equation can be obtained whose slope and intercept correspond directly to the model parameters like the adsorption constant. In practice, the slope yields information about the strength of adsorbate–adsorbent interactions, while the intercept is related to the equilibrium constant for adsorption. The isotherm constants and the value of q_e can thus be derived from the regression equation obtained from each plot. These regression equations are presented in Table 3.

Error analysis

The values of q_e , as shown in Table 2, were determined from laboratory investigations for different initial zinc ion concentrations. For evaluating the applicability and reliability of various isotherm models,

Table 3. Regression equation for different isotherm models.

Sr. No.	Adsorption isotherm model	Regression equation
1	Langmuir	$y = 2.2281x + 17.887$
2	Freundlich	$y = 0.1976x - 1.8078$
3	Temkin	$y = 0.0566x + 0.1401$
4	Harkins Jura	$y = -5.7425x + 31.118$
5	Frenkel - Halsey - Hill	$y = 0.2013x - 1.7926$
6	Elovich	$y = -0.8408x - 1.7997$
7	Frumkin	$y = -0.7545x - 1.6692$
8	Jovanovic	$y = 0.2013x - 1.7926$

Table 4. Value of q_e obtained from regression equations.

S. No.	Name of Isotherm	Value of q_e as calculated from the isotherm equation at $t = 180$ min				
		$C_0 = 20$	$C_0 = 40$	$C_0 = 60$	$C_0 = 100$	$C_0 = 200$
1	Langmuir adsorption isotherm	0.08954	0.2653	0.34773	0.39854	0.42694
2	Freundlich adsorption isotherm	0.18816	0.26603	0.31557	0.37197	0.44417
3	Temkin adsorption isotherm	0.17882	0.27726	0.3258	0.37255	0.42297
4	Harkins Jura adsorption isotherm	0.19199	0.24228	0.28797	0.37099	0.69083
5	Frenkel-Halsey-Hill adsorption isotherm	0.18809	0.26619	0.31591	0.37256	0.44509
6	Elovich adsorption isotherm	0.18361	0.24633	0.33376	0.43797	0.39668
7	Frumkin adsorption isotherm	0.18163	0.25724	0.33214	0.40886	0.40986
8	Jovanovic adsorption isotherm	0.24614	0.25636	0.27436	0.3196	0.47389

Table 5. Error function values for different adsorption isotherms.

Sr. no.	Error function name	Name of adsorption isotherm							
		Langmuir	Freundlich	Temkin	Harkins Jura	Frenkel-Halsey Hill	Elovich	Frumkin	Jovanovic
1	ME	0.09046	0.01791	0.01203	0.25583	0.01793	0.07397	0.04486	0.06614
2	MAE	0.03510	0.01034	0.00606	0.07051	0.01053	0.03267	0.02131	0.04534
3	RMSE	0.04551	0.01103	0.00732	0.11720	0.01116	0.04115	0.02619	0.04709
4	MSE	0.00207	0.00012	0.00005	0.01374	0.00012	0.00169	0.00069	0.00222
5	MRE	0.15101	0.03552	0.01740	0.18641	0.03587	0.09483	0.06189	0.16587
6	CI	0.92232	0.98826	0.99471	0.69664	0.98814	0.84884	0.93298	0.77426
7	EC	0.71271	0.98312	0.99257	- 0.906	0.98271	0.76505	0.90484	0.69238
8	EVS	0.73199	0.98313	0.99308	- 0.690	0.98271	0.76577	0.90488	0.69392
9	COD	0.71271	0.98312	0.99257	- 0.906	0.98271	0.76505	0.90484	0.69238
10	RAE	0.13850	0.03357	0.02228	0.35671	0.03398	0.12525	0.07971	0.14332
11	COR	0.97015	0.99233	0.99659	0.87745	0.99229	0.89903	0.95526	0.84500

the values of q_e (adsorption capacity at equilibrium) derived from various isotherm equations across varying initial ion concentrations have been compiled and presented in Table 4.

To obtain the most suitable isotherm model that can be used to design the adsorption process, an extensive error analysis employing eleven distinct error functions (as elaborated in Table 1) has been conducted. The calculated values of these error functions corresponding to different isotherm models are systematically indicated in Table 5.

By analysing the values of the respective error functions outlined in Table 5, the isotherm models have been ranked according to accuracy and relative performance as compared to other models for all the error function values. This comprehensive analysis ensures a robust and nuanced understanding of the adsorption behaviour under varying conditions. In this regard, it can be observed from Table 5 that with respect to all the error functions used in these investigations, the Temkin isotherm always performs the best. It exhibits minimum error for all the eleven error functions as compared to all the other isotherm models. Thus, it has been designated as the best model for the design of adsorption process. Following this, the second-best isotherm model is Freundlich, since for all the error

functions, it gives the second lowest error values in comparison to the remaining models. Thus, with respect to accuracy and relative performance, the isotherm models discussed in this paper are ranked as follows: Temkin, Freundlich, Frenkel-Halsey-Hill, Frumkin, Elovich, Langmuir, Jovanovic and Harkins Jura.

CONCLUSIONS

Eight different two-parameter isotherm models evaluated the adsorption characteristics and equilibrium data, and the accuracy of these models was assessed by using eleven different error functions. These error functions are used to compare the value of q_e as determined from laboratory investigations and isotherm models. From the analysis, it was concluded that:

- Temkin isotherm always performs the best and produces minimum error for all the eleven different error functions.
- The Freundlich isotherm is ranked as the second-best model in terms of accuracy.
- Harkins Jura isotherm shows the least accuracy as inferred from the experimental data.
- Based on the accuracy and relative performance, the different isotherms are ranked as follows: Temkin,

Freundlich, Frenkel-Halsey-Hill, Frumkin, Elovich, Langmuir, Jovanovic and Harkins Jura.

- The results emphasize the critical role of error function analysis in accurately ranking isotherm models and identifying the optimal isotherm for adsorption studies.

Authors' contributions: *A.K.A, affirm that this research paper is the result of my independent work. The content, including ideas, analysis, and conclusions, reflects my own efforts and understanding of the topic. This statement serves as a declaration of my authorship and commitment to upholding scholarly standards.*

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