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ISOTHERM ADSORPTION STUDIES OF Cd (II) ION REMOVAL FROM AQUEOUS SOLUTIONS BY MODIFIED RUBBER-BASED ADSORBENT

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Abstract: *This work is dedicated to adsorption study of Cd (II) ions by 1-[1-Methyl-2-(methylamino) ethyl] thiourea modified synthetic rubber. The impact of pH, contact time, initial metal ion concentration were among the several parameters influencing the adsorption process that was studied throughout the research. The highest adsorption capacity was achieved with 593.47 mg/g at pH=3 after modification with new thiourea derivative. Five adsorption isotherm models were examined to explain the adsorption process, and it was found that results are in agreement with the Redlich–Peterson Isotherm. The Temkin model was examined and found that there is chemical adsorption occurs in the adsorbent-adsorbate system. Desorption study are also included in this paper to study regeneration property of the adsorbent. So, 0.5 mol/l H_3PO_4 has demonstrated the maximum desorption capability over Cd (II) ions.*
Keywords: *adsorption, Cd (II), modification, isotherm, modification, thiourea.*

INTRODUCTION

Water is a vital natural resource required for the existence of all living species and the proper functioning of ecosystems [1,2]. However, growing urbanization, expansion of industry, and agricultural intensification have caused extensive pollution of water bodies across the world. Heavy metals are particularly concerning among contaminants due to their non-biodegradability and severe toxicity at extremely low doses [3]. Cadmium is a major representative of this group element since it is both non-essential and hazardous [4].

The high toxicity and bioaccumulation potential of cadmium make it one of the most hazardous heavy metals [5]. Despite being extremely rare (0.1–0.5 ppm) in the Earth's crust, cadmium is frequently released into the environment by human activities such the smelting of copper, lead, and zinc [6]. Additionally, it is commonly used in industrial processes including electroplating, the manufacturing of pigments and plastics, PVC stabilizers, phosphate fertilizers, nickel-cadmium batteries, and other alloys [7]. The main sources of cadmium contamination in surface and groundwater are

wastewater discharges from these businesses and improper disposal of objects containing cadmium [8]. Because of its high environmental persistence and ability to pass through food chains, cadmium can have long-term ecological and biological consequences [9].

Cadmium has no recognized physiological use in the human body, in contrast to several other metals that are useful as trace elements. Rather, even at very low quantities, it is very hazardous [8]. Inhaling industrial pollutants, consuming tainted food, or drinking water can all expose one to cadmium [10]. The buildup of this substance in the body mostly impacts the kidneys [11], liver [12] and bones [13], resulting in skeletal deterioration [14], hepatotoxicity [15], and nephrotoxicity [16]. Prolonged exposure is linked to reproductive damage [17], cardiovascular disorders [18], pulmonary dysfunction [19], and an elevated risk of cancer [20]. Clinical symptoms include bone softening [21], joint pain [22], respiratory distress [23], anemia [24], hypertension [25], and gastrointestinal disorders [26]. Owing to its high toxicity, the World Health Organization (WHO) has set the maximum permissible limit of cadmium in drinking water at only 0.003 mg/L, reflecting its potential threat to human health even at trace levels [27].

Given its persistence, toxicity, and widespread occurrence, cadmium removal from aqueous environments has become an urgent research priority in ecological and analytical chemistry [28]. Traditional treatment methods, such as chemical precipitation, ion exchange, membrane filtration, and electrochemical techniques have a number of disadvantages, including high operational costs, incomplete removal at low concentrations, and secondary waste generation [29]. In comparison, adsorption has emerged as one of the most efficient, cost-effective, and versatile methods for cadmium remediation. This technique offers simplicity, high efficiency at trace levels, reusability of adsorbents, and adaptability to large-scale applications[30].

Recent advances in materials science have focused on the development of novel adsorbents with enhanced affinity and selectivity for cadmium ions [31]. Among them, functionalized polymers and polymer-based composites have gained increasing attention due to their stability, tunable chemical properties, and potential for modification with active functional groups [32,33].

The present study aims to investigate the adsorption behavior of Cd (II) ions from aqueous solutions using a specially designed resin-based sorbent and it was modified with a thiourea derivative to improve its sorption capacity. The research focuses on evaluating the effects of pH, contact time, ionic strength, and initial metal ion concentration. In addition, desorption studies were performed to identify suitable regenerating agents. All studies was done in both static and dynamic conditions. To gain a better understanding of the sorption process, theoretical calculations were also done.

EXPERIMENTAL PART

Devices

The optical densities of the solutions were measured with a BK-UV 1600 Biobase spectrophotometer. The pH values of the solutions were measured using PHS-3DW digital pH meter. Distilled water was obtained through Biobase (WD-A10) water distiller device. The dynamic experiments were leaded by using SK-O180-Pro Digital Orbital Shaker.

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Preparation of solutions

The stock solution of cadmium (10^{-2} M) was prepared by dissolving appropriate amount of $Cd(NO_3)_2 \times 4\ H_2O$ in 100 ml of distilled water. The equilibrium concentrations of Cd (II) ions in the solution were determined with the help of the xylenol orange (R).

Buffer solutions with pH values ranging from 3 to 8 were prepared by mixing appropriate proportions of 0.1 M acetic acid (CH_3COOH) and ammonium hydroxide ($NH_3 \times H_2O$) and for pH 2, a hydrochloric acid (HCl) solution was employed as the acidic medium.

All the reagents that used were analytical grade pure.

Synthesis and identification of sorbent

For the experiment, we have used already synthesized rubber and for modification the novel thiourea derivative, the synthesis carried out according to the literature [34].

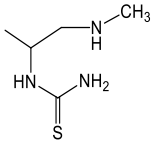
Synthesis of 1-[1-Methyl-2-(methylamino)ethyl]thiourea

The synthesis of N-substituted thiourea derivative was performed according to the literature [34,35].

For the reaction 1-(methylamino) propan-2-ol and thiourea were taken in the same mol ratio with 0.1 mol. So, a three-necked flask is filled with 10.3 g of 1-(methylamino)propan-2-ol and 7.6 g of thiourea then 10 ml of trifluoroacetic acid is added while stirring quickly, then the liquid is heated to 40–50^0 C for completely dissolve the thiourea. The stirring is ceased after four to five hours. Distillation is used to eliminate the trifluoroacetic acid. A water pump is used to remove the organic material from the solution. Precipitation in methyl alcohol yields 72% of 1-[1-methyl-2-(methylamino)ethyl]-thiourea.

Table 1

Summary of analysis results

Name	Structure	Brutto formula	Element Analsis	Spectral analysis	
1-[1-Methyl-2-(methylamino)ethyl] thiourea		$C_5H_{13}N_3S$	C 40.82%, H 8.84%, N 28.57%, S 21.77%.	NMR	^{1}H NMR (300 MHz, $CDCl_3$, δ, ppm): 9.53 (s, 2H, NH_2), 7.31 (s, ^{1}H, NH), 2.47 (d, 3H, CH_3), 2.0 (m, ^{1}H, NH), 2.64–2.89 (t, 2H, CH_2), 3.03 (d, ^{1}H, CH), 1.13 (s, 3H, CH_3). 13C NMR (75 MHz, $CDCl_3$, δ, ppm): 18.6, 53.3, 57.6, 36.2, 184.7.
				IR	$\nu(NH_2)$ = 665, 711, 742, 847, 878 sm^{-1}, $\nu(R_2NH)$ = 1562 sm^{-1}, ν (CH_2) = 2925, 2855, 1462 sm^{-1}, ν(C=S) = 1129, 1168, 1243, 1313, 1365, 1377 sm^{-1}, ν (CH_3) = 1462, 1377 sm^{-1}.

As it is seen from the table 1, all desired functional groups are found in new thiourea derivative.We could predict from this data that the metal will be strongly bounded with this functional groups based on previous theoretical and experimental analysis.

Sorption experiments

Batch sorption experiments for Cd (II) were carried out at ambient temperature (25 ºC). In each run, 2 mL 5 × 10^{-3} mol·L^{-1} concentration of cadmium solution was transferred into 100 mL glass reagent bottle with screw cap. Subsequently, 30 mg of the sorbent was added and the pH of the suspension was adjusted to the desired value. The mixtures were left for 24 h to attain equilibrium, then the solid phase was separated from the aqueous phase by using filter paper. Dynamic sorption experiments were led by same order by using automatic orbital shaker.

The equilibrium concentration of metal ions in the solution was determined by known spectrophotometric method using xylenol orange as organic reagent (pH 6; λ=575 nm) [36].

The degree of sorption and removal percentage of metal ions are calculated by the following formulas:

$$R_{\%}=\frac{C_0-C_e}{C_e}100 \quad \text{(I)}$$

$$q_e=\frac{(C_0-C_e)V}{m} \quad \text{(II)}$$

Here, C_0 is the initial concentration of the metal ion (mol·L^{-1}), C_e is the equilibrium concentration of the metal ion (mol·L^{-1}), V is the volume of the solution (L), and m is the mass of the sorbent (mg).

Regeneration studies

The preparation process of the synthetic adsorbents generally involves multi-step procedures that are both time and energy-intensive [37]. Except from that this process often requiring expensive and scarcely available reagents. For this reason, in the research of novel sorbents investigation of regeneration potential of them has great importance. In the literature, there are approaches exist for adsorbent regeneration, including biological techniques employing microorganisms, thermal methods via oxidative treatment, and chemical regeneration in which the retained adsorbate is released by treatment with suitable solvents [38]. In the present work, we used chemical regeneration method by investigating the influence of different acids.

Desorption experiments were performed using 0.5 mol·L^{-1} aqueous solutions of HNO_3, HCl, H_2SO_4, H_3PO_4 and CH_3COOH. The first step of experiment is the preparation of sorbents. For this 30 mg of the sorbent was weighted and added to each 5 experiment bottles then 2 ml 10^{-2} M metal solution then 18 mL of a pH 3 buffer solution was added onto them. The suspensions were maintained for 24 h to reach equilibrium then the solid phase was separated from the liquid by filtration.

In the next step, for the controlling desorption potential of each acids, 20 mL of 0.5 mol·L^{-1} solutions of acids were added onto dry sorbent samples and kept for 24 hours. After it, each sample measured and desorption capacity calculated.

RESULTS AND DISCUSSION

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Effect of pH on the sorption of Cd (II)

The pH of the solution is one of the most critical factors affecting the sorption of Cd (II) ions, since it controls both the ionization state of functional groups on the sorbent surface and the stability of cadmium in aqueous systems. At low pH values (pH < 4), the high concentration of H^+ ions compete strongly with Cd^{2+} for the active sorption sites, resulting in reduced uptake. In the range of pH 4–8, Cd^{2+} remains the predominant soluble species and electrostatic repulsion is minimized, thereby favoring sorption. However, at higher pH values (pH > 8), cadmium undergoes hydrolysis, leading to the formation of $Cd(OH)^+$, $Cd(OH)_2^-$, and $Cd(OH)_3^-$, $Cd(OH)_4$,$Cd_4(OH)_4$ which can precipitate from solution [39]. To avoid precipitation effects and ensure that the removal process is attributable solely to surface sorption, the initial solution pH was adjusted between 2.0 and 8.0.

The experiment was carried out by weighing and collecting 30 mg of sorbent in different reagent bottles, adding 2 ml of 10^{-2} M metal ion solution and 18 ml of the proper pH, and then letting it remain for 24 hours.

The effect of the pH of the solution on the sorption of cadmium in static and dynamic condition is shown in the figure below (fig 1). As a result of the measurement, it was found that the capture of Cd (II) ions with the highest percentage from the solution was at pH 3 in all three conditions but with different sorption capacity. Therefore, this value of pH was used in all subsequent experiments.

Effect of time on the sorption of metal ions

To investigate the time-dependent nature of sorption, 30 mg of sorbent was mixed with 2 ml of 10^{-2} M metal solution, followed by 18 ml of pH 3. The rate of metal ion sorption in the solution was then monitored for 30 to 270 minutes. It was found that the sorption has already became stable and attained the equilibrium condition after 60 minutes, as shown in figure 2.

One may say that the metal ion was no longer drawn out of the solution after this point. When 1-[1-Methyl-2-(methylamino)ethyl] thiourea was absorbed into the sorbent, the same thing happened but with higher sorption capacity. In other words, the sorption hit the plateau after 60 minutes in both static/dynamic condition for pure sorbent and after modification. The picture below provides a graphical depiction of the experiment's result (fig.2).

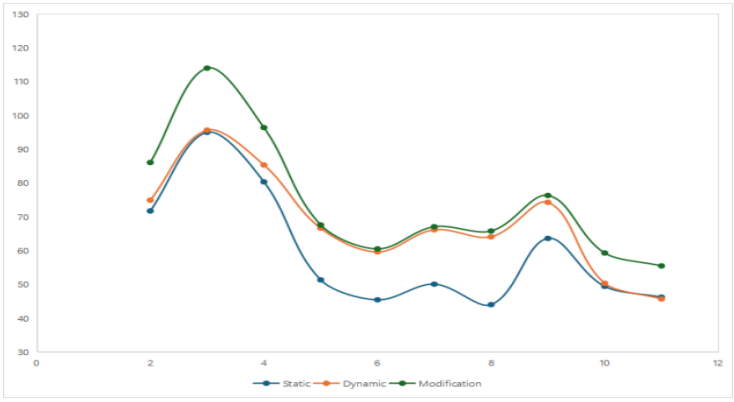

Fig.1. Effect of pH

Effect of initial concentration of Cd (II) ions on adsorption capacity.

The impact of concentration on the degree of adsorption by the synthesized sorbent was investigated throughout the experiment. Cd (II) ion concentrations ranging from 2 $x10^{-4}$ mol/L to 8 $x10^{-3}$ mol/L were employed for this purpose. This was accomplished by weighing 30 mg of sorbent and adding the proper amount of metal ion solution and pH 3.0. The experiment's outcome is showen in figure 3 below.

Desorption process.

The study of the opposing process, desorption, is also included in the present work. Having the eluents needed to desorb the metal ion is a crucial task. So, by employing several inorganic acids of the same concentration as 0.5 mol/L solutions of HNO_3, HCl, H_2SO_4, CH_3COOH and H_3PO_4 to carry out this procedure. According to the study's findings, 0.5 mol/L H_3PO_4 solution has the highest desorption capability for Cd (II) ions.

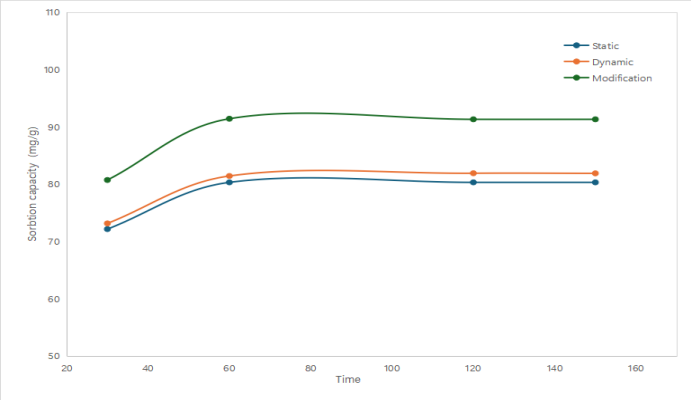

Fig.2. Effect of time

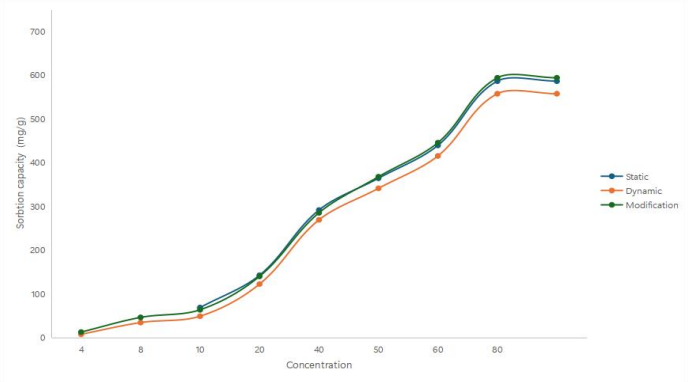

Fig.3. Effect of initial concentration

Adsorption isotherm

In these types of studies, it is necessary to have a deeper comprehension of the adsorption process. For that aim, utilizing the appropriate adsorption equilibrium reaches our help. Adsorption equilibrium describes the nature of the interaction between the adsorbent and the adsorbate. The Langmuir, Freundlich, Dubinin-Radushkevich, Temkin, Redlich–Peterson isotherms were used to examine the equilibrium data in this study.

Langmuir Isotherm

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The Langmuir isotherm is one of the most widely applied models for describing adsorption equilibrium. It is based on the fundamental assumption that the surface of the adsorbent is homogeneous, meaning all adsorption sites are energetically equivalent. According to this model, adsorption takes place at specific sites on the surface in a monolayer fashion, with no lateral interactions between adjacent adsorbed molecules. Furthermore, the probability of adsorption at a given site is independent of the occupancy of neighboring sites [40].

The mathematical representation of the Langmuir isotherm is given as:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e}$$

where C_e (mmol/L) is the equilibrium concentration of the adsorbate in the solution, q_e (mmol/g) is the equilibrium adsorption capacity, q_m represents the maximum adsorption capacity corresponding to complete monolayer coverage, and K_L is the Langmuir constant related to the affinity of the binding sites (L/mmol).

Experimentally, the model can be linearized by plotting 1/ q_e against 1/ C_e. From the slope and intercept of the resulting straight line, the values of q_m and K_L can be determined, respectively.

An additional parameter used to characterize the Langmuir isotherm is the dimensionless equilibrium factor, R_L expressed as:

$$R_L = \frac{1}{1 + bC_0}$$

where b is the Langmuir constant (L/mmol) and C_0 is the initial concentration of the adsorbate (mmol/L). The value of R_L provides insight into the favorability of the adsorption process. Specifically, $0 < R_L < 1$ indicates favorable adsorption, while values outside this range signify unfavorable, linear, or irreversible adsorption.

Calculations show the correlation coefficients (R^2) are 0.0282, 0.6619 and 0.4996, respectively for static, dynamic and modification cases. The results indicate that the separation factor (R_L) ranges from 0 to 1 is favorable but in our case the values are minus for all cases. So, it shows that this model doesn't fit (fig. 4).

Freundlich Isotherm

The Freundlich isotherm is an empirical model that describes adsorption on heterogeneous surfaces with non-uniform distribution of adsorption heat and affinities. Unlike the Langmuir model, which assumes surface homogeneity, the Freundlich model accounts for the presence of multiple adsorption sites of varying energies [41].

The isotherm is expressed by the logarithmic form:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e$$

where q_e (mmol/g) represents the adsorption capacity at equilibrium, C_e (mmol/L) is the equilibrium adsorbate concentration, K_F is the Freundlich adsorption constant indicative of adsorption capacity, and 1/n is the heterogeneity factor that reflects the adsorption intensity.

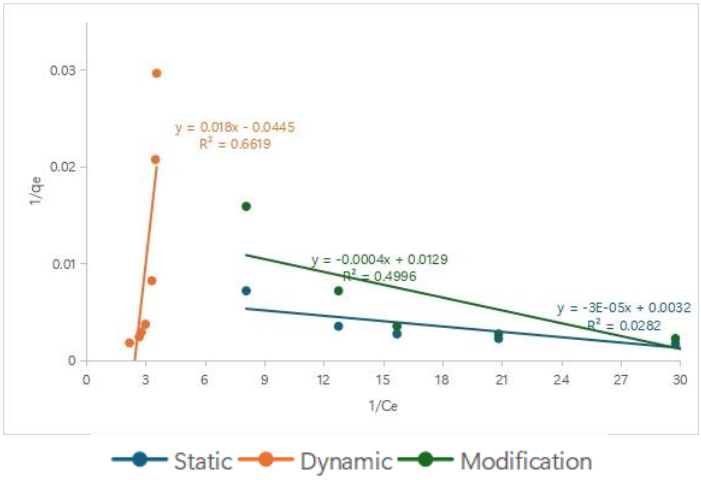

Fig. 4. Langmuir isotherm model

The parameter n provides important insight into the favorability of adsorption. Values of 1 < n < 10 generally indicate favorable adsorption conditions. A higher n value (corresponding to a smaller 1/n) suggests stronger binding interactions between the adsorbent and adsorbate, while 1/n =1 corresponds to linear adsorption, which implies uniform adsorption energies across the surface. Linear adsorption is typically associated with low solute concentrations and minimal adsorbent coverage. In our the values of 1/n is -1.545, 5.782, 2.790 for static, dynamic and modification cases, respectively (fig.5).

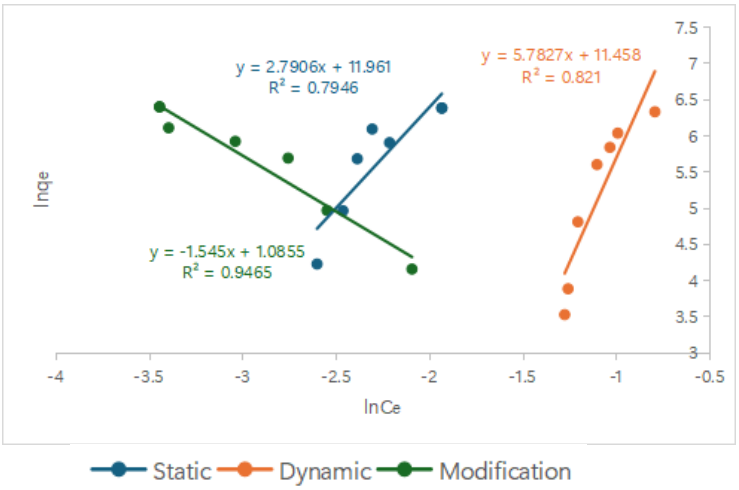

Fig. 5.Freundlich isotherm model

Dubinin–Radushkevich (D–R) Isotherm

The Dubinin–Radushkevich (D–R) isotherm model is frequently employed to evaluate adsorption properties such as porosity, sorption energy, and the mechanism of interaction. Unlike the Langmuir model, it does not assume a uniform surface or constant adsorption potential, making it more suitable for describing adsorption on heterogeneous surfaces [42]. The general form of the D–R isotherm is expressed as:

$$\ln_e = \ln q_s - k_{D\text{-}R}\varepsilon^2$$

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where q_e (mmol/g) is the equilibrium adsorption capacity, q_s represents the theoretical maximum adsorption capacity (mmol/g), $k_{D\text{-}R}$ is the D–R constant related to adsorption energy, and ε is the Polanyi potential.

The Polanyi potential is defined as:

$$\varepsilon = RT \ln\left(1+\frac{1}{C_e}\right)$$

where R is the universal gas constant (kJ·mol^{-1}·K^{-1}), T is the absolute temperature (K), and C_e is the equilibrium concentration of the adsorbate (mmol/L).

A significant feature of the D–R isotherm is its ability to distinguish between physical and chemical adsorption processes. The mean free energy of adsorption (E) can be calculated from the constant $k_{D\text{-}R}$ obtained from the slope of the plot, according to:

$$E = \frac{1}{\sqrt{2k_{D\text{-}R}}}$$

where E (kJ/mol) indicates the type of sorption mechanism. Typically, values of E < 8 kJ/mol suggest physical adsorption driven by weak Van der Waals forces, whereas values of E > 8 kJ/mol are indicative of chemisorption involving charge transfer or chemical bonding.

The obtained R^2 values are 0.8275, 0.8634,0.917 and the calculated adsorption energy (E) is 1 and 5 kJ/mol for static and dynamic studies. According to the established criteria, an E value in the range of 8–16 kJ/mol suggests chemisorption, whereas values below 8 kJ/mol are indicative of physisorption. Therefore, in both cases, the E value being less than 8 kJ/mol suggests that the adsorption process is predominantly physical in nature (fig.6).

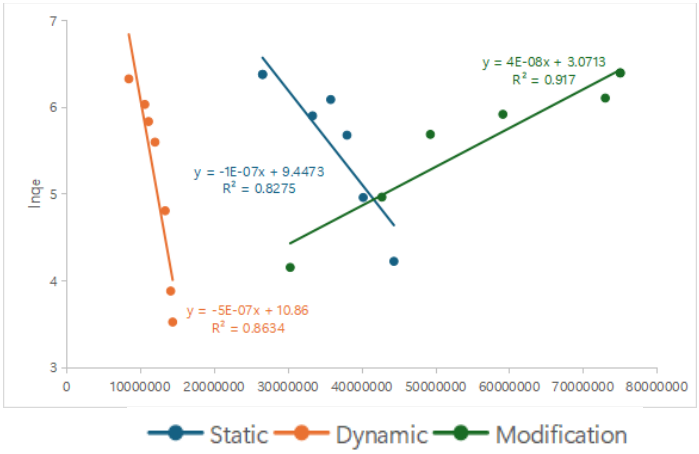

Fig. 6. Dubinin-Radushkevich (D-R) isotherm

Temkin Isotherm

The Temkin isotherm model considers the effects of indirect adsorbate–adsorbate interactions on the adsorption process. Unlike the Langmuir model, which assumes constant adsorption energy, the Temkin model proposes that the heat of adsorption decreases linearly with increasing surface coverage due to these interactions [43]. This assumption provides a more realistic description of adsorption behavior, particularly at intermediate concentrations.

The Temkin equation is expressed in both its general (I) and linearized forms (II):

$$q_e=\frac{RT}{b_T}\ln(K_T C_e) \quad (I)$$

$$q_e=\frac{RT}{b_T}\ln(K_T)+\frac{RT}{b_T}\ln(C_e) \quad (II)$$

where q_e (mmol/g) is the adsorption capacity at equilibrium, C_e (mmol/L) is the equilibrium adsorbate concentration, K_T (L/g) is the Temkin isotherm constant, R (8.314 $J \cdot mol^{-1} \cdot K^{-1}$) is the universal gas constant, T (K) is the absolute temperature, and b_T (J/mol) is a constant related to the heat of adsorption.

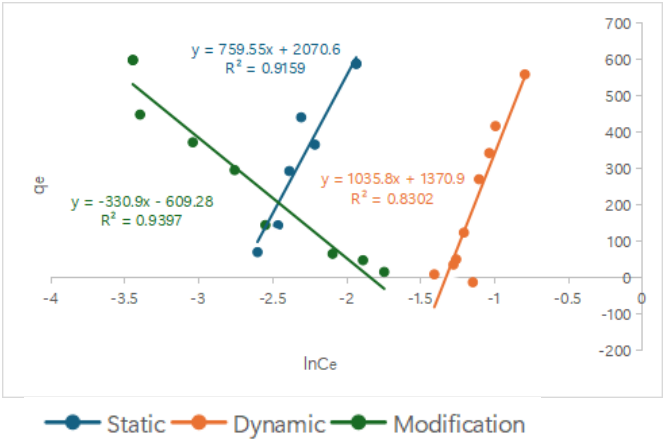

Fig.7. Temkin isotherm model

As shown in figure 7, static condition B_T value is 759.55 kJ/mol ; following in the dynamic case , this value rises to 1035.8 kJ/mol. Since B_T values below 8 kJ/mol are generally linked to physisorption, this suggests that the adsorption process is primarily physical before functionalization. In both cases, B_T value marginally surpasses 8 kJ/mol that indicating chemisorption. In terms of the K_T value, it changes from 15.27 to 3.757 in static and dynamic absorption conditions and 6.305 after modification.From this values we can say that in the static condition adsorbant surface has highest affinity.

Redlich–Peterson Isotherm

The Redlich–Peterson (R–P) isotherm is a hybrid model that incorporates features of both the Langmuir and Freundlich isotherms. It is widely applied because of its ability to describe adsorption over a broad concentration range, including both homogeneous and heterogeneous surface conditions [44]. Unlike strictly empirical models, the R–P isotherm provides a semi-empirical framework that bridges the assumptions of ideal monolayer adsorption and multilayer adsorption on heterogeneous sites.

The non-linear form of the R–P equation is expressed as:

$$q_e = \frac{K_R C_e}{1+a_R C_e^{\beta}}$$

where q_e (mmol/g) is the adsorption capacity at equilibrium, C_e (mmol/L) is the

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equilibrium concentration of the adsorbate, K_R (L/g) is the R–P constant related to adsorption capacity, a_R (L/mmol) is a constant, and β is an exponent with a value between 0 and 1.

The parameter β plays a crucial role in determining the model's behavior. When $\beta = 1$, the R–P equation reduces to the Langmuir form, suggesting adsorption on a homogeneous surface. Conversely, when $\beta < 1$, the equation resembles the Freundlich model, indicating adsorption on heterogeneous surfaces.

Due to this flexibility, the R–P isotherm is particularly valuable for systems where neither the Langmuir nor the Freundlich models alone adequately describe adsorption, thus making it a powerful tool for interpreting experimental sorption data.

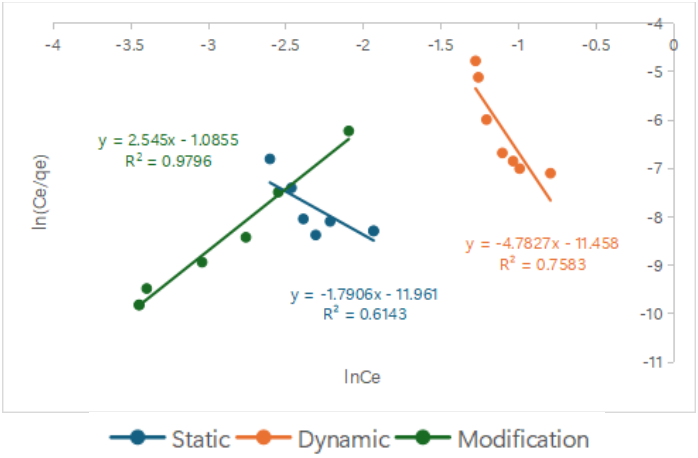

Fig.8. Redlich–Peterson Isotherm

These results show that (fig.8) the three systems differ from one another. Stronger interactions under altered conditions were observed by the modified cases with larger a/K ratio and greater slope g = 2.545. On the other hand, both the dynamic and static systems showed negative values of g, which are beyond the range of $0 < g < 1$ that theoretically acceptable.

CONCLUSION

To sum up, our research yielded encouraging and significant findings that demonstrate the effectiveness of this innovative functionalized sorbent in removing cadmium from water. Adsorption capacity rises by around 20% after sorbent's modification with novel thiourea derivative in static coundition and almost same results were observed in dynamic condition. Several isoterm models were used to study adsorption process and the Redlich–Peterson Isotherm model was determined to provide the greatest match. From the Temkin isoterm, it was calculated that there is chemisorption.

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