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# Use of Hydrochloric Acid Modified Clay for Adsorption of Mercury(II) Ions from Aqueous Solutions

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Doğal bollukları ve işleme gereksinimlerinin azlığıyla killer, çeşitli ağır metaller için ekonomik adsorbanlar olarak potansiyel taşırlar. Bu araştırmada, hidroklorik asit (HCl) ile modifiye edilmiş kilin, sulu çözeltilerden cıva(II) ($Hg^{2+}$) iyonlarını adsorplama yeteneği incelenmiştir. Adsorpsiyon kapasitesine etki eden parametreleri belirlemek için başlangıç metal iyon konsantrasyonu, temas süresi ve sıcaklık etkileri araştırılmıştır. Çalışmada, doğal kil için uygun başlangıç konsantrasyonun 400 mg/L, temas süresini ise 50 dakika olarak belirlenmiştir. Modifiye edilmiş kil için ise 400 mg/L başlangıç konsantrasyonu ve 60 dakika temas süresi en iyi sonuçları vermiştir. İzoterm verileri analizi, Langmuir izoterm modelinin $Hg^{2+}$ iyonları adsorpsiyonunda her iki materyal için en iyi uyumu gösterdiğini göstermiştir. 298 K, 308 K ve 318 K'de doğal kil ile Hg2+ iyonları adsorpsiyon kapasitesi sırasıyla 4.56, 5.01 ve 5.08 mg/g olarak bulunmuştur. Modifiye edilmiş kilin ise aynı sıcaklıklarda 11.12, 11.37 ve 12.30 mg/g adsorpsiyon kapasitesine sahip olduğu hesaplanmıştır. Ayrıca, kinetik analizi, $Hg^{2+}$ iyonları adsorpsiyonunda her iki materyal için de en iyi uyan denklemin sözde ikinci derece kinetik model olduğu belirlenmiştir. Adsorpsiyon deneyleri, doğal kil ile modifiye edilmiş kilde $Hg^{2+}$ metal iyonlarının adsorpsiyon mekanizmalarını incelemiş ve sonuçlar, modifiye edilmiş kilin metal iyonları için adsorpsiyon kapasitesinin ham kilinkinden daha yüksek olduğunu göstermiştir.

# Sulu Çözeltilerden Civa(II) İyonlarının Adsorpsiyonunda Hidroklorik Asit İle Modifiye Edilmiş Kilin Kullanılması

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With their natural abundance and minimal processing requirements, clays hold the potential to serve as economical adsorbents for various heavy metals. In this research, the adsorption capacity of hydrochloric acid (HCl) modified clay to adsorb mercury(II) ($Hg^{2+}$) ions from aqueous solutions was investigated. The parameters affecting the adsorption capacity were determined by studying the initial metal ion concentration, contact time, and temperature effects. For natural clay, an optimal initial concentration of 400 mg/L and a contact time of 50 minutes were identified. Meanwhile, modified clay showed best results with an initial concentration of 400 mg/L and a contact time of 60 minutes for $Hg^{2+}$ ions. The analysis of isotherm data revealed that the Langmuir isotherm model exhibited the best fit for both materials in $Hg^{2+}$ ion adsorption. At temperatures of 298 K, 308 K, and 318 K, the adsorption capacity for natural clay and $Hg^{2+}$ ions were found to be 4.56, 5.01, and 5.08 mg/g, respectively. Meanwhile, the modified clay displayed adsorption capacities of 11.12, 11.37, and 12.30 mg/g for $Hg^{2+}$ ions at the same temperatures. Additionally, the kinetic analysis determined that the pseudo-second-order kinetic model was the best fit for both materials in $Hg^{2+}$ ion adsorption. The adsorption experiments investigated the adsorption mechanisms of $Hg^{2+}$ metal ions on both natural clay and modified clay, with results indicating that the modified clay had a higher adsorption capacity for metal ions compared to the raw clay.

## 1. INTRODUCTION

In the wake of escalating worldwide industrialization and urbanization, heavy metal pollution poses a significant hazard to human beings and other forms of life. Stringent regulations have been implemented to regulate heavy metal levels across diverse settings, encompassing potable water sources, agricultural fields, food supplies, and wastewater treatment (Altunkaynak et al., 2022). These regulations promote the adoption of pragmatic and cost-effective methods for remediation. Numerous sectors, particularly mining and electronics industries, significantly contribute to the substantial presence of heavy metals in waste materials. These industrial contaminants often trigger heavy metal poisoning within aquatic ecosystems. While some heavy metals like Cu, Ni, Mn, Co, Pb, Cd, and Hg can inflict harmful effects on humans, animals, and plants in excess or insufficient quantities, they are also indispensable for fundamental biochemical processes (Canpolat et al., 2022). Thus, effectively addressing heavy metal pollution and devising sustainable solutions remains imperative. Ongoing advancements in technology and research are actively striving to develop safer and ecologically sound approaches for eliminating or mitigating heavy metal contamination. Throughout this process, the industrial sector should exercise caution, optimizing production practices while prioritizing environmental protection and adhering to principles of sustainability. Mercury ($Hg^{2+}$) pollution presents an imminent global concern, especially due to its hazardous nature. It is deemed one of the most perilous heavy metals, extensively released into the environment through industries like mining, refining, and coal extraction (Rahman et al., 2020; Renu et al., 2020). Its elevated toxicity and potent carcinogenic attributes can result in critical health conditions, affecting the nervous, circulatory, immune, and reproductive systems. Consequently, eradicating $Hg^{2+}$ ions from aqueous solutions holds paramount importance in curbing environmental contamination and enhancing overall quality of life.

Natural adsorbents like clay minerals and agricultural residues play a central role in effectively eliminating heavy metals and inorganic pollutants. These techniques embody promising measures that hold immense significance in safeguarding the environment and the welfare of living organisms (Wang et al., 2021). Likewise, the ongoing advancement of environmentally friendly and natural technologies enables us to progress further in our endeavors to counter the menace of heavy metal pollution. Specifically, clay assumes a pivotal function in extracting heavy metals from aquatic settings. The benefits of clay in adsorption processes hold crucial environmental value: substantial capacity, cation exchange proficiency, sustainability, economic feasibility, strong attraction, and absence of toxicity. The utilization of this natural asset not only contributes positively to the local economy but also mitigates environmental concerns. This marks a significant stride toward a cleaner environment for generations to come.

This study extensively explored the capacity of both natural and acid-modified clay to capture $Hg^{2+}$ ions from aqueous solutions. The efficacy of these clay materials in $Hg^{2+}$ ion removal was assessed through investigations into parameters like initial metal ion concentration, contact time, temperature, as well as isotherm and kinetic influences. Brunauer-Emmett-Teller (BET) and X-Ray Diffraction (XRD) analysis techniques were employed in the research to gain deeper insights into the adsorption mechanism of $Hg^{2+}$ ions. These analyses facilitated the assessment of the physical and chemical attributes of the clay, elucidating the process of sequestering $Hg^{2+}$ ions.

## 2. EXPERIMENTAL

### 2.1. Preparation of Modified Clay

For the adsorption study involving $Hg^{2+}$ ions, an initial stock solution was established with a concentration of 800 mg/L. This stock solution was created by employing deionized water and mercuric (II) nitrate monohydrate [$Hg(NO_3)_2.H_2O$]. Subsequently, this initial stock solution was employed to generate lower-concentration $Hg^{2+}$ solutions essential for the research. Each adsorption experiment was carried out using freshly prepared dilutions. All chemicals employed in this study were sourced from Sigma-Aldrich.

The clay utilized as an adsorbent in this study was sourced from deposits in the Şenkaya district of Erzurum province. The clay minerals underwent initial crushing using a jaw crusher followed by further processing in a ball mill, reducing them to small stone-like pieces. The resulting powdered clay was then sieved to attain particle sizes below 100 microns, and its moisture content was eliminated by subjecting it to 105 °C in an oven for 24 hours. The procedure for preparing the modified clay is as follows: 5 g of natural clay was taken and dispersed in 50 mL of a 10% by weight HCl aqueous solution (Abdullahı et al., 2020). Subsequently, the blend was stirred at ambient temperature for a duration of 24 hours. The resulting altered clay substances were isolated from the blend using centrifugation, followed by thorough rinsing with distilled water. This rinsing process was conducted iteratively to remove any lingering acids or impurities until the pH of the solution after rinsing reached 7. The end product was then subjected to drying at 105 °C and utilized in the ensuing adsorption trials.

### 2.2. Characterization of clay

The XRD (ADX-2500 models) is a technique used to analyze the crystal structure of materials. This technique is based on the principle of scattering X-rays resulting from the regular arrangement of atoms or molecules in the crystal structure. It is used to obtain information such as the crystal structures of the material, the size and orientation of the crystal with XRD analysis. Therefore, XRD analysis was used to confirm the crystal structure of natural clay.

The BET (Micromeritics Gemini VII 2390t models) analysis is a technique used to determine surface area and porosity properties. BET (Brunauer-Emmett-Teller) analysis is usually performed based on the adsorption and desorption properties of solid surfaces. This analysis is used to understand the surface properties of materials and to evaluate the performance of materials for applications such as catalytic, adsorption and gas storage. For both the unaltered and modified clay, the specific surface areas were determined using BET (Micromeritics Gemini VII 2390t models) analysis, which involved $N_2$ sorption conducted at lower temperatures.

### 2.3. Adsorption study

Adsorption experiments were conducted within 250 mL conical bottles at a stirring rate of 100 rpm, utilizing a 25 mL solution with varying concentration ranges (50-500 mg/L for both natural and modified clay). Following the completion of the adsorption process, unadsorbed $Hg^{2+}$ ions in the liquid medium were quantified using an atomic absorption spectrophotometer (AAS).

The optimal conditions for the adsorption studies were determined through calculations involving the initial $Hg^{2+}$ ion concentration and contact time. The impact of temperature on adsorption was examined at three distinct temperatures The amount of $Hg^{2+}$ adsorbed was determined using Equation (1) (Savastru et al., 2022).

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

In this context, W(g) signifies the adsorbent's mass, while V(mL) indicates the solution's volume. The metal ion showcases initial and equilibrium concentrations, represented as $C_o$ and $C_e$ respectively.

## 3. RESULTS AND DISCUSSION

### 3.1. BET analysis

The adsorption capacity of the materials employed as adsorbents is directly correlated with their pore volume and surface area. Application of the BET theory revealed that the pore volume and surface area of natural clay are 0.0146 cc/g and 5.18 $m^2/g$, respectively, while modified clay exhibits a pore volume of 0.0224 cc/g and a surface area of 9.25 $m^2/g$.

### 3.2. XRD analysis

The clay's diffractogram is depicted in Figure 1. Quartz $SiO_2$, a silica variety characterized by interconnected $SiO_2$ tetrahedra forming a robust three-dimensional lattice, is evident. The prominent bands are observed at $2\theta = 21.48$, with strong peaks at 29.52, 36.12, and 61.85. The distinctive shifts at $2\theta = 37.36$, 42.15, and 49.25 are attributed to the clay material.

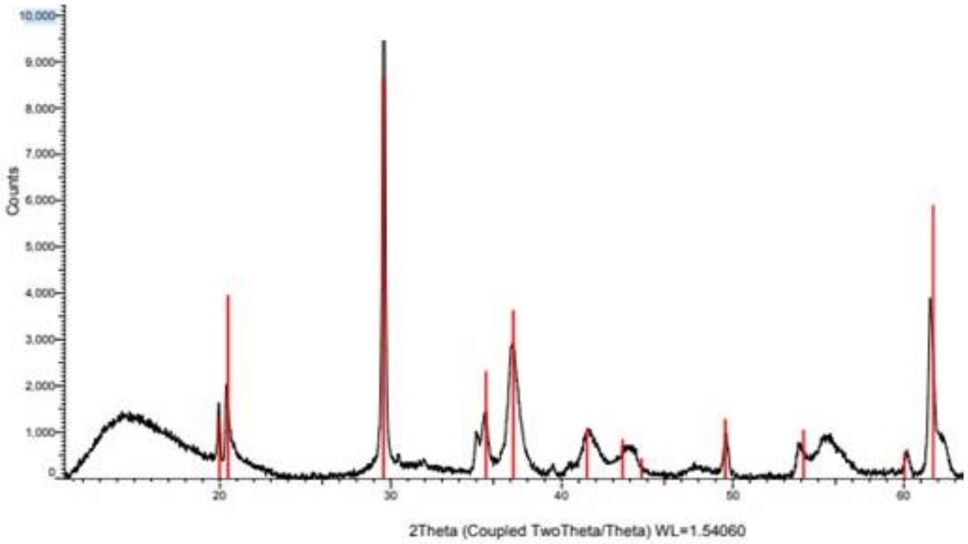

**Figure 1.** XRD pattern of Clay

### 3.3. Effect of initial concentration

The initial solution concentration wields significant influence over the adsorption process. This study's results demonstrate that adsorption rates are subject to variation based on adsorption time and initial concentration (Altunkaynak et al., 2023). In order to examine this effect, 0.5 g of both natural clay and modified clay were mixed with 25 mL of $Hg^{2+}$ metal ion solutions, with temperatures set at 298 K, 308 K, and 318 K for a duration of 120 minutes. The concentration ranges for both materials ranged from 50 to 500 mg/L. Post-adsorption, the remaining metal ion quantities were quantified using AAS. Illustrated in Figure 2 is the impact of the initial concentration on the removal of metal ions from the aqueous solution by both natural and modified clay. It was observed that the adsorption of $Hg^{2+}$ ions in both materials exhibited an increase up to a certain concentration threshold, beyond which the adsorption plateaued as the metal ion solution concentration further increased. This behavior is likely attributed to $Hg^{2+}$ ions saturating the active sites of the materials. As depicted in Figure 2, the concentrations of adsorbed metal ions at 298 K, 308 K, and 318 K exhibit an upward trend with increasing temperature for both materials. According to the data obtained in the later stages of the study, 400 mg/L $Hg^{2+}$ ion solutions were used for both materials.

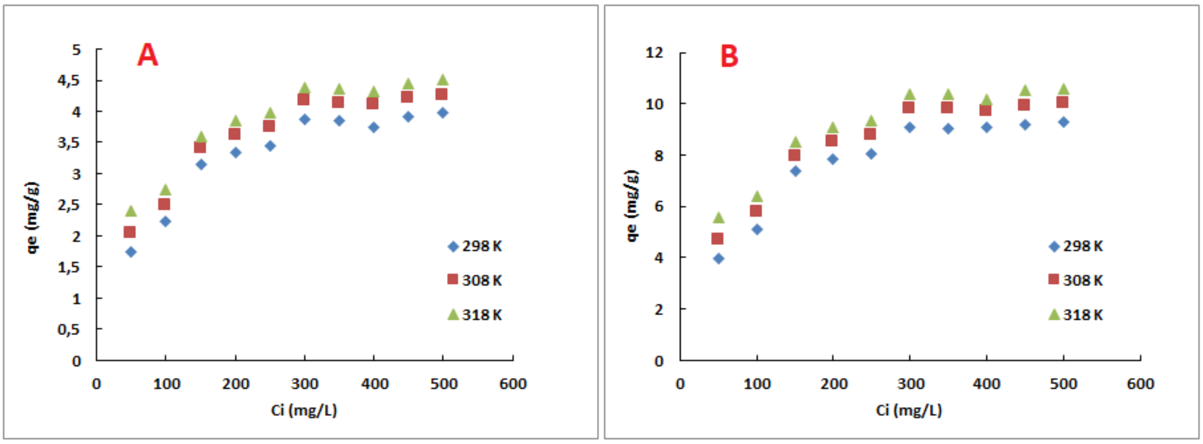

**Figure 2.** The influence of starting concentration on $Hg^{2+}$ removal by natural (A) and modified clay (B) adsorbents (0.5 g adsorbent mass, V = 25 mL).

### 3.4. Effect of contact time

The duration of contact between the clay and the $Hg^{2+}$ ions in the solution is a pivotal factor in adsorption studies (Oh et al.,2019). Figure 3 illustrates the impact of contact time on $Hg^{2+}$ removal using natural and modified clay. Adsorption experiments were performed at 298-318 K using concentrations of 0.5 g adsorbent and 400 mg/L $Hg^{2+}$ solution for both natural and modified clay. The data collected within the 10-120 minute timeframe were employed to plot qt against time, revealing an equilibrium time of 50 minutes for natural clay and 60 minutes for modified clay. As observed in Figure 3, the removal of $Hg^{2+}$ ions exhibited a gradual increase until the 50th and 60th minutes, respectively. Subsequent to these durations, the saturation of accessible sites led to a stabilization in the removal of $Hg^{2+}$ ions, resulting in a tendency for the removal rate to plateau as the adsorption time continued to increase.

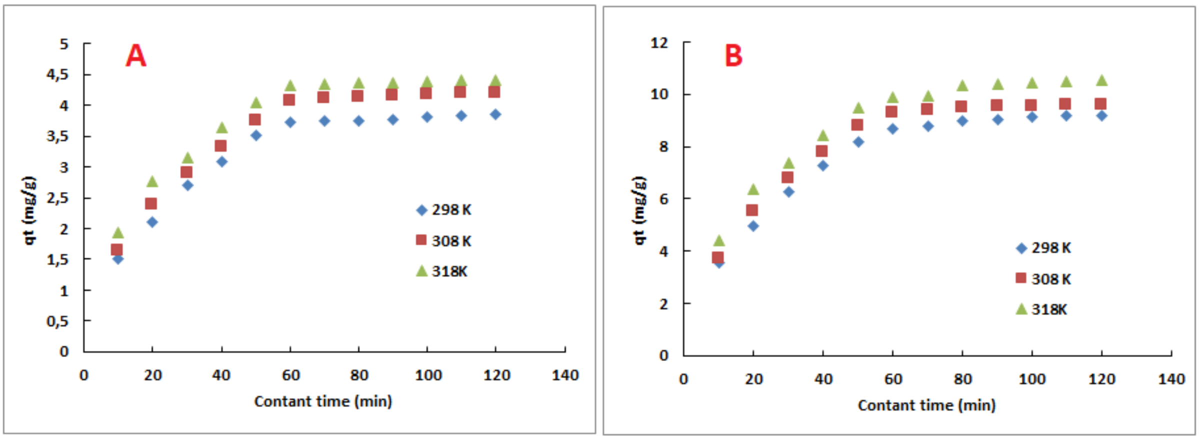

**Figure 3.** Contact time influences Hg2+ ion adsorption in natural (A) and modified clay (B).

### 3.5. Kinetic study

In order to gain insight into the $Hg^{2+}$ ion adsorption mechanisms on natural and modified clay, as well as to ascertain the rate-controlling step of the process, kinetic experiments were conducted. Among the vital factors affecting the adsorption process, contact time plays a crucial role. To delve deeper into the kinetic characteristics, a comprehensive analysis of nonlinear kinetic modeling was performed. The pseudo-first-order (PFO) and pseudo-second-order (PSO) kinetic models were explored, employing experimental data to evaluate the adsorption information. Equations (2) and (3) present the corresponding nonlinear kinetic equations for PFO and PSO (Rahdar et al., 2019; Ghanimati et al., 2017).

$$\ln(q_e - q_t) = ln q_e - k_1 t \tag{2}$$

$$\frac{t}{q_t} = \frac{t}{q_e} + \frac{1}{k_2 q_e^2} \tag{3}$$

In this context, the rate constants corresponding to the PFO and PSO adsorption processes are denoted as $k_1$ and $k_2$, respectively.

The kinetic parameters derived from the nonlinear plots of the models are presented in Figure 4 and Table 1. The qe and qt values, as detailed in Table 1, were determined across temperatures of 298, 308, and 318 K. Notably, the PFO model exhibited relatively low $R^2$ determination coefficients and a discernible variance between $q_{Experimental}$ and $q_{Theoretical}$ values, suggesting its inadequacy in elucidating the adsorption of the cations investigated in this study. In contrast, the PSO model displayed a theoretical adsorption capacity closely aligned with the experimentally ascertained value and relatively high $R^2$ coefficients, affirming its suitability in describing the adsorption kinetics of metal cations on both natural and modified clay. The maximum adsorption capacity of $Hg^{2+}$ ions on natural clay was found to be 4.56 mg/g, 5.01 mg/g, and 5.08 mg/g for temperatures of 298, 308, and 318 K, respectively. Similarly, the maximum adsorption capacity of $Hg^{2+}$ ions on modified clay was 11.12 mg/g, 11.37 mg/g, and 12.30 mg/g for the same temperature range. These results show that the adsorption capacity of the clay increases approximately 2.2 times when modified with HCl compared to its natural state.

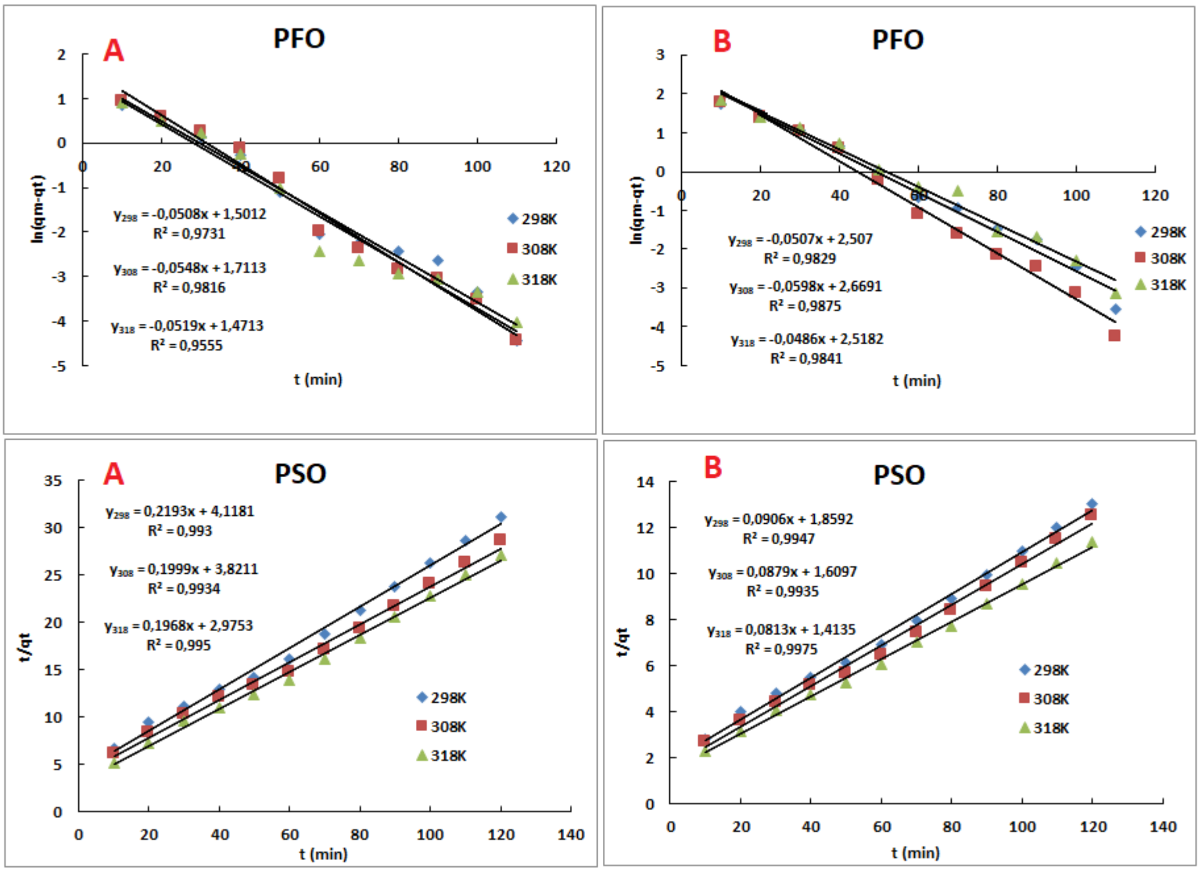

**Figure 4.** For $Hg^{2+}$ adsorption on natural (A) and modified clay (B); PFO and PSO model plots

**Table 1.** Kinetic parameters for the removal of $Hg^{2+}$ on natural and modified clay at disparate temperatures

| | Natural clay | | | Modified clay | | |
|---|---|---|---|---|---|---|
| | **298 K** | **308 K** | **318 K** | **298 K** | **308 K** | **318 K** |
| **PFO** | | | | | | |
| $q_{max}$ (mg/g) | 4.48 | 5.54 | 4.35 | 12.18 | 14.43 | 12.41 |
| $k_1$ ($min^{-1}$) | 0.050 | 0.055 | 0.052 | 0.051 | 0.059 | 0.048 |
| $R^2$ | 0.9731 | 0.9816 | 0.9555 | 0.9829 | 0.9875 | 0.9841 |
| **PSO** | | | | | | |
| $q_{max}$ (mg/g) | 4.56 | 5.01 | 5.08 | 11.12 | 11.37 | 12.30 |
| $k_2$ (g $mg^{-1}$ $min^{-1}$) | 0.0116 | 0.0105 | 0.0130 | 0.0044 | 0.0048 | 0.0047 |
| $R^2$ | 0.9930 | 0.9934 | 0.9950 | 0.9947 | 0.9935 | 0.9975 |

### 3.6. Adsorption isotherms study

To investigate the equilibrium characteristics of both natural and modified clay, Langmuir and Freundlich isotherm models were employed. These models serve to elucidate the connection between the surface attributes of the adsorbent material and the quantity of adsorbate retained from the solution. The Langmuir isotherm model postulates a monolayer adsorption of $Hg^{2+}$ ions onto the surfaces of natural and modified clay. The linear representation of the Langmuir adsorption isotherm is formulated by Equation (4) (Ashouri et al., 2021).

$$\frac{C_e}{q_e} = \frac{1}{qmax.K_L} + \frac{C_e}{qmax} \qquad (4)$$

The constant associated with adsorption is denoted as $K_L$.

In contrast, the Freundlich isotherm model considers multilayer adsorption, accounts for the heterogeneity of the adsorbent surface, and incorporates molecular interactions between the adsorbent and the adsorbate. This model provides insight into the intricate interactions occurring during the adsorption process. The linear rendition of the Freundlich isotherm model is presented through Equation (5) as follows (Sing et al., 2019).

$$logq_e = logK_F + \left({}^{1}/_{n}\right)logC_e \qquad (5)$$

The Freundlich constants $K_F$ and n, respectively, denote the extraction capacity and density.

The graphical representation of the adsorption equilibrium data based on the Langmuir and Freundlich isotherm models is depicted in Figure 5. Furthermore, the comprehensive parameters calculated using these two distinct isotherm models are detailed in Table 2. Upon evaluating the $R^2$ values, it becomes evident that the Langmuir model emerges as the most fitting isotherm model for explaining the adsorption of $Hg^{2+}$ onto both the natural and modified clay. The selection of this model also lends support to the notion that the adsorption process is predominantly chemically driven and potentially irreversible.

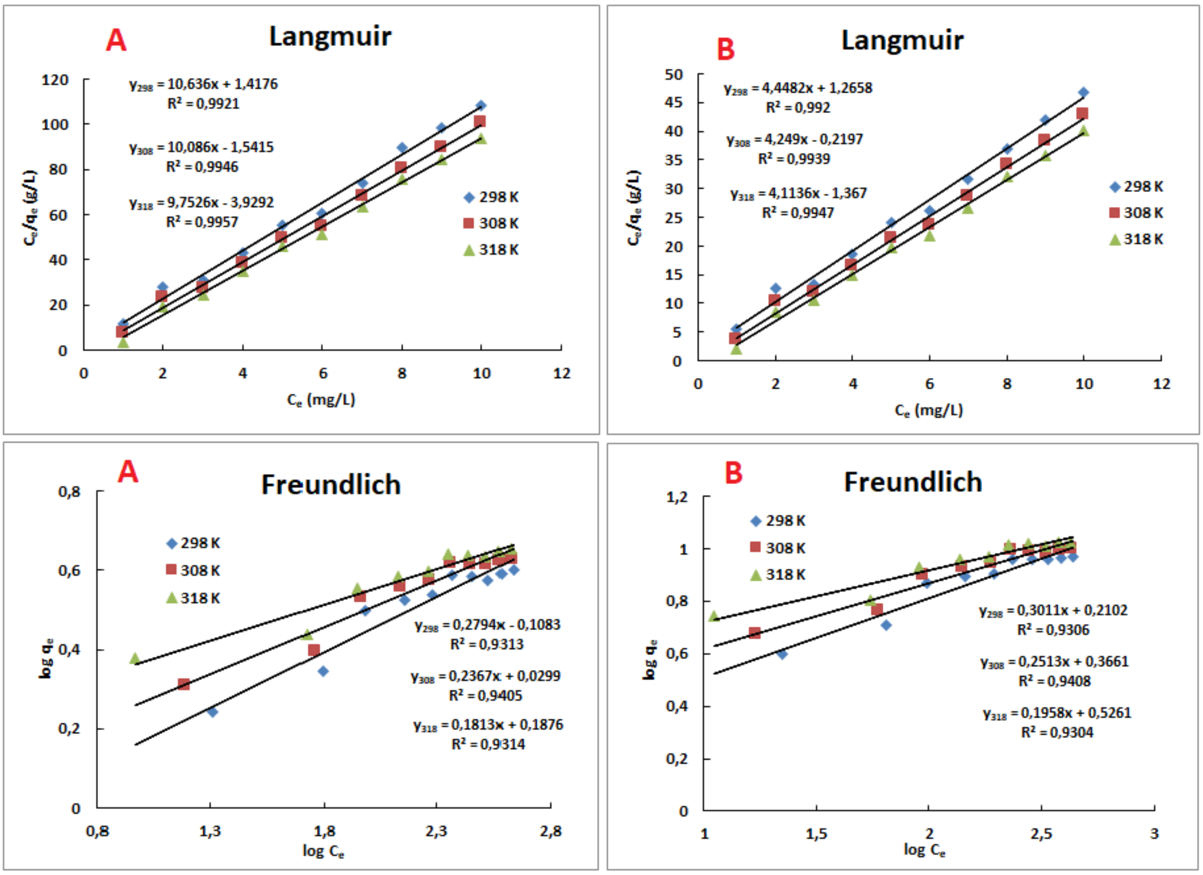

**Figure 5.** Langmuir and Freundlich isotherm plots of Hg2+ ion adsorption on natural (A) and modified clay (A).

**Table 2.** Isotherm variables for $Hg^{2+}$ ion adsorption with natural and modified clay

| | **Natural clay** | | | **Modified clay** | | |
|---|---|---|---|---|---|---|
| | **298 K** | **308 K** | **318 K** | **298 K** | **308 K** | **318 K** |
| **Langmuir** | | | | | | |
| $q_{max}$ (mg/g) | 0.096 | 0.099 | 0.103 | 0.225 | 0.235 | 0.243 |
| $K_L$ (L/mg) | 7.348 | -6.553 | -2.477 | 3.511 | -19.369 | -3.011 |
| $R^2$ | 0.9921 | 0.9946 | 0.9957 | 0.9920 | 0.9939 | 0.9947 |
| **Freundlich** | | | | | | |
| $K_f$ (mg/g) | 3801.9 | 16595.8 | 323593.6 | 2089.3 | 9332.5 | 128824.9 |
| n | 3.58 | 4.22 | 5.51 | 3.32 | 3.97 | 5.11 |
| $R^2$ | 0.9313 | 0.9405 | 0.9314 | 0.9306 | 0.9408 | 0.9304 |

## 4. CONCLUSION

In this investigation, the adsorption of $Hg^{2+}$ ions from aqueous solutions was accomplished using both natural and modified clay as the adsorbents. The adsorption efficacy of natural clay in capturing $Hg^{2+}$ ions (4.56 mg/g at 298 K, 5.01 mg/g at 308 K, and 5.08 mg/g at 318 K) was compared to that of modified clay, which demonstrated notably enhanced adsorption performance for $Hg^{2+}$ ions (11.12 mg/g at 298 K, 11.37 mg/g at 308 K, and 12.30 mg/g at 318 K), outperforming natural clay. Equilibrium analysis indicated that the Langmuir isotherm model was more fitting for both materials compared to the alternative model. Kinetic investigations revealed that the PSO equation accurately describes the $Hg^{2+}$ ion removal process for both materials. The adsorption capacity of the materials exhibited a direct correlation with their surface area and pore volume. The BET analysis confirmed that the modified clay possessed significantly higher adsorption capacity compared to the natural clay. Specifically, the surface area and pore volume of natural clay were found to be 5.18 $m^2/g$ and 0.0146 cc/g, respectively, while those of modified clay were determined to be 9.25 $m^2/g$ and 0.0224 cc/g, respectively. Based on the analyses, it can be concluded that HCl-modified clay offers a practical and cost-effective solution for the removal of $Hg^{2+}$ from aqueous solutions, owing to its accessibility, affordability, and elevated adsorption capacity.

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