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Efficient removal of naphthalene from aqueous solutions using modified kaolin: Optimization and characterization studies

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ABSTRACT

The presence of organic pollutants, particularly polycyclic aromatic hydrocarbons and dyes, poses significant environmental threats, necessitating urgent remedial measures. This study aims to address the critical issue of naphthalene contamination in water by investigating the efficacy of modified kaolin as a low-cost adsorbent for its removal from aqueous solutions. Raw kaolin underwent calcination at 650 °C followed by acid treatment using 1M hydrochloric acid. The treated kaolin was meticulously characterized using Fourier-transform infrared spectroscopy and scanning electron microscopy. A comprehensive optimization study was conducted using the Box-Behnken design, revealing the highest removal efficiency (RE) of approximately 87.9% under the following conditions: pH 7, initial naphthalene concentration 100 mg/L, and contact time 120 minutes. Equilibrium data fitting into the Langmuir isotherm yielded an impressive coefficient of determination (R^2) value of approximately 0.98. Kinetic data were successfully modeled using the pseudo-second-order equation, further validating the efficacy of the adsorption process. Thermodynamic studies confirmed the feasibility of the adsorption process, as evidenced by increasingly negative values of Gibbs' free energy alongside a positive enthalpy change. Notably, naphthalene adsorption onto treated kaolin clay particles exhibited promising results. Optimization of process parameters using response surface methodology elucidated an optimum condition for maximum RE, which was experimentally validated. In conclusion, this study establishes that kaolin represents a viable and cost-effective adsorbent for naphthalene removal in water treatment applications. Its potential to mitigate environmental pollution underscores its significance in addressing contemporary environmental challenges.

Keywords: waste water, pollutants, kaolin, removal efficiency, adsorption

INTRODUCTION

Water pollution remains a persistent and escalating global challenge, presenting substantial health risks and environmental threats (du Plessis, 2022; Tozer, 2023). Despite concerted efforts to mitigate its impact, water pollution persists as a formidable issue (Al-Nuaim et al., 2022; Savaşan, 2017). The World Health Organization reports that contaminated drinking water results in approximately 485,000 diarrheal deaths annually and facilitates the spread of diseases such as cholera, dysentery, hepatitis A, typhoid, and polio (Savaşan, 2017). The presence of harmful substances such as chemical elements, organic compounds, and heavy metals in water sources poses significant health risks emphasizing the urgent need for effective environmental protection strategies (Mitra et al., 2022).

A critical approach involves identifying and removing contaminants such as naphthalene, toluene, and phenol from the environment and treating wastewater to make it safe and reusable (Bala et al., 2022). However, conventional remediation technologies often fall short and can take years to produce effective results (Bhatt et al., 2022). Therefore, innovative and efficient methods are essential to address this pressing issue and ensure sustainable water management.

Water pollution has emerged as a critical global concern in recent years. Factors such as rapid industrialization, agricultural expansion, and environmental changes have collectively contributed to the degradation of water quality (du Plessis, 2022). Among the various pollutants, polycyclic aromatic hydrocarbons (PAHs) pose significant risks, with naphthalene being a prominent example (Patel et al., 2020). The increasing demand for water, driven by both industrial and domestic activities, underscores the urgent need for efficient water management and treatment solutions (Boretti & Rosa, 2019). Unfortunately, the persistent release of PAHs and heavy metals, including nickel, into the environment remains a cause for concern (Sakhiya et al., 2023). Naphthalene, due to its non-polar nature, does not readily dissolve in water, exacerbating the threat to public health and the ecosystem (Arizavi et al., 2020).

Naphthalene, along with other organic pollutants, is commonly found in petroleum products. The U.S. Environmental Protection Agency classifies naphthalene as a group C possible human carcinogen (Kishor et al., 2020; Saleem et al., 2024; Shetty et al., 2023). These pollutants result from the partial burning of organic matter, such as tobacco smoke, coal, oil spills, and refuse combustion (Yaashikaa et al., 2022). The incomplete combustion of organic substances releases PAHs into the environment, particularly in areas with heavy fossil fuel usage (Mitra et al., 2022). The hydrophobicity and low water solubility of PAHs, including naphthalene, make them persistent contaminants in wastewater. This persistence complicates their degradation, as they strongly adsorb to environmental surfaces (Achakulwisut et al., 2023).

Naphthalene's presence in water poses significant health risks, including respiratory problems, skin irritation, and hemolytic anemia (Arizavi et al., 2020) . Its environmental persistence and potential for bioaccumulation raise concerns for both aquatic ecosystems and human health. Moreover, naphthalene's volatility contributes to air pollution, further complicating remediation efforts. Therefore, developing innovative and efficient remediation techniques is essential for sustainable water management (Yost et al., 2021). However, naphthalene is not the only harmful agent present in wastewater. Other hazardous substances include heavy metals (such as lead, copper, and cadmium), PAHs, polychlorinated biphenyls, volatile organic compounds, and various microbial pathogens (Chilakamarry et al., 2023; Ezeonuegbu et al., 2021; Ramutshatsha-Makhwedzha et al., 2022). To effectively cleanse water, a range of adsorbents can be employed, including activated carbon, zeolites, clays, and nanoadsorbents, each with specific capacities for removing different pollutants (Shu et al., 2007).

Kaolin, a naturally occurring clay mineral, has shown promise as an effective adsorbent due to its high surface area, chemical stability, and availability. Kaolin's application in water treatment offers advantages such as low cost, environmental friendliness, and efficiency in adsorbing various contaminants. Enhancing kaolin's adsorptive properties through modifications, such as thermal treatment, acid activation, or intercalation with organic molecules, can increase its capacity to remove naphthalene from wastewater.

In recent years, researchers have explored the use of natural adsorbents for pollutant removal from wastewater. Among these, kaolin–a clay mineral abundant in certain regions–has gained attention. Specifically, kaolin sourced from Gbako Local Government in Niger State, Nigeria, has demonstrated promise as an economic, safe, and effective adsorbent for tannery wastewater treatment (Mustapha et al., 2021). The characterization of this kaolin sample revealed a specific surface area of 17 m^2/g, a pore volume of 0.018 cm^3/g, and a pore diameter of 3.587 nm. These properties contribute to its adsorption capacity.

Additionally, Arizavi et al. (2020) investigated the removal of naphthalene–an organic pollutant–from aqueous solutions using nanoporous kaolin composites derived from wastewater. Their findings indicated a desirability index of 1.00, with optimal conditions achieved at a composite dose of 4.8 g/L, a contact time of 66 minutes, and a solution pH of 6.5. Under these conditions, the composite achieved an impressive 97% removal efficiency (RE) for a solution containing 10 mg/L of naphthalene.

This research aims to develop an effective adsorbent from raw kaolin rock for removing naphthalene from wastewater. The specific objectives include creating an effective kaolin-based adsorbent, conducting batch adsorption studies of naphthalene, investigating suitable isotherm and kinetic models for naphthalene adsorption on kaolin, optimizing adsorption parameters using response surface methodology, and performing thermodynamic studies of naphthalene adsorption using kaolin. A comprehensive review conducted by Uddin (2023) underscored the efficacy of both natural and modified forms of clay minerals, with a particular focus on Kaolin, in removing various toxic aquatic metal pollutants from wastewater. Building upon this, Ismail et al. (2024) conducted a study evaluating the effectiveness of thermally treated kaolin clay as an absorbent and coagulant in wastewater treatment. Their findings revealed that kaolin clay exhibited promising coagulation properties and could efficiently absorb pollutants such as chemical oxygen demand, biological oxygen demand, turbidity, total suspended solids, and various elements from wastewater, achieving significant removal rates. Similarly, Chai et al. (2023) investigated the adsorption of heavy metals from industrial wastewater using a low-cost Malaysian kaolin clay-based adsorbent. Their results highlighted that acid-activated kaolinite adsorbent emerged as a favorable and commercially feasible option for wastewater treatment due to its enhanced adsorption capacity.

In a study by Zubir et al. (2024), the effective removal of phenol using a green kaolin adsorbent was investigated. The research utilized RSM to determine the optimal conditions for phenol removal, which were found to be a pH of 1.6, a contact time of 40 minutes, an initial phenol concentration of 50 mg/L, and an adsorbent dose of 2 g. Under these conditions, a maximum RE of 92.19% was achieved. Additionally, regeneration tests indicated that the kaolin adsorbent retained over 76.76% of its adsorption capacity after reuse.

Similarly, Abdel-Aleem et al. (2023) explored the efficiency of phenol removal using both natural and chemically modified kaolin clays. The optimal conditions identified for phenol removal included a contact time of 300 minutes, an initial phenol concentration of 25 mg/L, a pH of 7, and an adsorbent dose of 2.5 g/L. The study found that the highest phenol removal efficiencies at a concentration of 5 mg/L were 90%, 97%, and 96.2% for crude (natural), acid-modified, and base-modified kaolin clays, respectively. The adsorption capacities for phenol and 4-chlorophenol were reported as follows: crude kaolin had 7.481 mg/g for phenol and 4.195 mg/g for 4-chlorophenol, acid-modified kaolin had 8.2942 mg/g for phenol and 3.211 mg/g for 4-chlorophenol, and base-modified kaolin had 8.05185 mg/g for phenol and 18.565 mg/g for 4-chlorophenol.

This study endeavors to tackle the challenge of organic pollutants in wastewater by harnessing the cost-effectiveness and accessibility of kaolin as an adsorbent. The proposed method holds the potential for efficiently treating wastewater contaminated with naphthalene, thereby offering a substantial solution to environmental pollution. Through this research, significant societal benefits are anticipated, including the alleviation of the adverse impacts of organic pollutants in wastewater and the promotion of kaolin utilization, thereby fostering environmental sustainability.

The research methodology involved using kaolin powder for adsorption to remove naphthalene from wastewater. We systematically examined various factors that influenced adsorption, including contact time, solution pH, and initial concentration. Additionally, the study assessed different isotherm models (Langmuir, Freundlich, and Temkin) and kinetic models (pseudo second order [PSO] and Elovich model) relevant to naphthalene adsorption on kaolin.

By devising an effective technique for naphthalene removal from wastewater utilizing kaolin, this research endeavors to make a significant contribution to the fields of environmental engineering and public health. The ultimate goal is to ensure the availability of cleaner water resources and foster a healthier environment for current and future generations.

MATERIALS

The materials and reagents used in this study included kaolin as the primary material sourced from Alkaleri Local Government area in Bauchi State, Nigeria. Analytical-grade chemical reagents, such as naphthalene, hydrochloric acid (HCl), distilled water, and methanol, were employed in the research process. The apparatus consisted of beakers, measuring cylinders, stirrers, flat-bottom flasks, and foil paper. Equipment utilized included a Shimadzu digital weighing balance for precise measurements, a mechanical shaker for ensuring proper mixing of samples, a Shimadzu UV-visible spectrometer for measuring absorbance efficiency, an electric blender for pulverizing kaolin, a Thermo Fisher Scientific oven for removing moisture content from samples, and a mortar and pestle for breaking down kaolin before laboratory analysis. Additionally, specific materials used included conical flasks sourced from Luco Laboratory, a 100-mesh screen sieve, test tubes, and a 400 nm Shimadzu UV spectrophotometer.

The schematic figure shown in **Figure 1** outlines the process of this research. It starts with the beneficiation of kaolin to improve its quality, followed by the calcination of the treated kaolin material at high temperatures. The next step is the acid treatment of the calcined kaolin sample to further enhance its properties. This is followed by the morphological and structural characterization of the kaolin clay sample using techniques like scanning electron microscopy (SEM) and Fourier-transform infrared spectroscopy (FTIR). Finally, the process examines the effects of varying parameters on the adsorption of naphthalene, focusing on factors such as organic pollutant concentration and contact time. This comprehensive process is crucial for enhancing the properties of kaolin clay, making it suitable for various industrial and environmental applications.

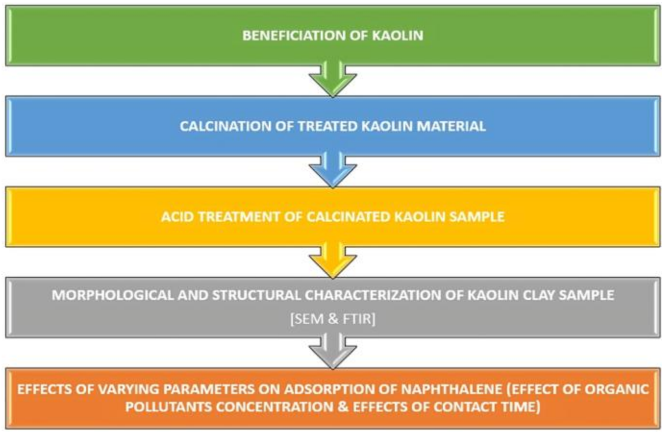

Figure 1. Schematic of the experimental procedures (Source: Authors' own elaboration)

Beneficiation of Kaolin

The kaolin rock clay was crushed with a mechanical crusher at the Central Workshop Faculty of Engineering, University of Lagos. The Crushed samples was then sieved with a manual sieve of 0.099nm diameter so as to obtain fine powder kaolin particles.

To eliminate impurities from the fine powder, a method described by Abdel-Aleem et al. (2023) was employed. Specifically, 500 g of the powdered kaolin was submerged in a container containing 3,000 cm^3 of distilled water for a duration of 72 hours. Subsequently, the resulting slurry underwent filtration to remove any accumulated dirt, facilitated by passing it through a 100-mesh screen sieve.

Calcination of Treated Kaolin Material

The beneficiated kaolin sample underwent the crucial process of calcination to enhance its properties for various applications. The procedure involved placing the treated kaolin material in a precision-controlled furnace, where it was subjected to temperatures reaching approximately 500 °C. This thermal treatment was meticulously conducted over a duration of 6 hours to ensure optimal transformation of the kaolin structure. The calcinated kaolin samples was then crushed for further treatment. This was done to improve adsorption capacity.

Acid Treatment of Calcinated Kaolin Sample

The crushed kaolin sample was subjected to acid treatment using 2M HCl for a duration of 4 hours. This step served several purposes. First, it helped remove impurities and enhance the surface properties of the kaolin particles. Second, acid treatment facilitated the breakdown of any residual minerals or organic matter present in the sample. After acidification, the sample was meticulously rinsed with distilled water until achieving a neutral pH of 7. This ensured that any excess acid was thoroughly removed. Subsequently, the acid-treated sample underwent moisture removal by placing it in an oven at 105 °C for 2 hours. This step was crucial for obtaining a dry, consistent sample for further analysis.

Experimental Design

The Box-Behnken design (BBD) was employed to investigate the impact of variables on the RE, serving as the experimental response. BBD is a type of RSM that is particularly useful for fitting quadratic models without involving extreme combinations of variables. Each factor, namely Initial concentration, pH, and contact time, was denoted by a three-level code: -1, 0, and +1. The RE was determined as a function of the initial concentration (ranging from 100 ppm to 300 ppm), pH (ranging from 5 to 9), and contact time (ranging from 30 to 120 minutes), resulting in a total of 17 experimental runs.

A second-order polynomial quadratic model was utilized to analyze and fit the experimental data collected throughout the reaction process. Analysis of variance (ANOVA) charts were generated using Design Expert software to assess the significance of the model and its parameters. Furthermore, numerical optimization techniques were employed to determine the optimal values for the independent variables, thereby maximizing the RE of the system.

Evaluation of Adsorption Capacity and Removal Efficiency

To assess the RE and adsorption capacity of the treated kaolin material, a stock solution of naphthalene was prepared with a concentration of 500 ppm. This concentration was achieved by dissolving 0.50 g of naphthalene in a mixture comprising 300 ml of methanol and 200 ml of distilled water.

The quantity of naphthalene adsorbed from each solution was determined using Eq. (1). Subsequently, the residual concentration of naphthalene in the solution was ascertained by referencing the standard curve expression, enabling precise quantification of the adsorption process.

$$\%Removal = \frac{Co-Ce}{Co} * 100, \quad (1)$$

$$Adsorption\ capacity\ (q_e) = (Co - Ce) * \frac{V}{M}, \quad (2)$$

where C_o (mg/l) is the initial concentration of solution, C_e (mg/l) is the residual concentration of the solution at equilibrium, V (L) is the volume of the solution, M (g) is the mass of the kaolin adsorbent, q_e (mg/g) is the adsorption capacity of the kaolin.

The RE was determined by Eq. (1), while adsorption capacity will be determined by Eq. (2).

Morphological and Structural Characterization of Kaolin Clay Sample

Surface morphology analysis was conducted through SEM to examine both treated and raw kaolin clay samples. Prior to analysis, the samples were meticulously prepared by mounting them onto SEM stubs and sputter-coating with gold to enhance conductivity. High-resolution imaging using a SEM analyzer revealed distinctive surface textures, showcasing rough surfaces and porous structures. Moreover, the analysis unveiled the presence of minute holes and small openings across the sample surfaces. These morphological features provide valuable insights into the microstructure of kaolin clay, which are essential for understanding its physical properties and potential applications.

In addition to SEM analysis, further characterization of the kaolin clay samples was performed using FTIR technique. FTIR analysis elucidated the chemical composition of the samples by identifying characteristic absorption bands corresponding to various functional groups present in the material.

Batch Adsorption Experiment

Adsorbate solution preparation

A stock solution was prepared with a concentration of 500 mg/L, as outlined by Shankar et al. (2017). Subsequently, the stock solution was diluted to various concentrations including 100 ppm, 200 ppm, and 300 ppm. These dilutions were made to facilitate batch adsorption equilibrium studies.

Adsorption studies

Batch adsorption experiments were conducted using treated kaolin adsorbent powder to evaluate its performance in adsorbing naphthalene from solution. Each experiment involved adding 1 gram of the sample to 100 ml of naphthalene solution with different initial concentrations in flat-bottom flasks. The concentration of naphthalene solution was measured at different time intervals using a UV spectrometer at 275 nm, and the residual concentration was determined. The adsorption capacity (qe) was calculated using Equation 2

Effects of varying parameters on adsorption of naphthalene

Effect of organic pollutants concentration: The impact of the initial concentration of naphthalene solution on adsorption was studied, maintaining other variables such as temperature, pH of the solution, and adsorbent dose constant. Naphthalene concentrations were varied within the range of 200, 150, 100, and 50 mg/L, as described by Arizavi et al. (2020) and Yost et al. (2021).

Effects of contact time: The impact of contact time on the adsorption of naphthalene was studied by preparing organic pollutant solution mixtures with a fixed adsorbent dose and initial concentration. Eight different flasks containing 100 ml of the synthesized wastewater were each added with 1 gram of the adsorbent. The contact time was varied in the range of 15 to 120 minutes, maintaining constant agitation and temperature. The adsorption process was monitored, and the removal rate of organic pollutants was observed to increase with increasing contact time until reaching equilibrium. The equilibrium time reflects the point where the adsorption and desorption of organic pollutants on the adsorbent reach dynamic equilibrium, indicating the maximum adsorption capacity under the given operating conditions.

Adsorption Kinetic Experiments

Adsorption kinetics is a fundamental aspect of study that delineates the pace at which a solute is released from an aqueous environment to a solid-phase interface under specific conditions. This process encompasses two primary mechanisms: physical (physisorption) and chemical (chemisorption). Physical adsorption originates from weak van der Waals forces, while chemisorption involves the establishment of robust interactions between the solute and the adsorbent, typically involving electron transfer. Various

Table 1. Standard calibration table

CONC	ABS
20	0.400
10	0.219
5	0.036
15	0.290
25	0.476
30	0.628

kinetic models are utilized to scrutinize adsorption kinetics, each offering distinct insights into the process.

Pseudo second order model

A PSO kinetic equation is given, as in Eq. (3):

$$\frac{t}{q_t} = \frac{1}{kq_e^2} + \frac{1}{q_e}t, \quad (3)$$

where q_t is the amount of pollutant removed at time t (mg/g), q_e is the adsorption capacity at equilibrium (mg/g), k_1 is the pseudo-first-order rate constant (1/min), and t is the contact time (min). A graph of $\frac{t}{q_t}$ is plotted against t to obtain a slope of k.

RESULTS AND DISCUSSION

Standard Calibration Curve

To establish a quantitative relationship between absorbance and the concentration of the standard solution utilized in the adsorption experiments, a standard calibration curve was plotted. The standard solution, comprising naphthalene in wastewater, was prepared at concentrations ranging from 5 to 30 mg/L. The absorbance of each standard solution was measured spectrophotometrically.

The resulting data were tabulated in **Table 1** and plotted in **Figure 2** to generate the standard calibration curve. The absorbance values corresponding to concentrations of 5, 10, 15, 20, 25, and 30 mg/L were recorded as 0.036, 0.219, 0.29, 0.4, 0.476, and 0.628, respectively.

Figure 2 depicts the linear relationship between absorbance and concentration, affirming the suitability of the chosen method for quantifying naphthalene concentrations in wastewater samples. The calibration curve was fitted to a straight-line equation, enabling the accurate determination of naphthalene concentrations in subsequent adsorption experiments utilizing kaolin clay as the adsorbent.

Subsequent regression analysis was conducted to establish a linear equation representing the correlation between absorbance and concentration. This analysis revealed a coefficient of determination (R^2) of 0.9822, indicating a strong correlation between absorbance and concentration. The derived linear regression equation, $y = 0.0219x - 0.0426$ (where y represents absorbance and x represents concentration), played a crucial role in constructing the standard calibration curve illustrated in **Figure 2**.

The calibration curve not only demonstrates the sensitivity and accuracy of our analytical method but also ensures precise measurements throughout our investigation. The positive slope of 0.0219 highlights a direct relationship between

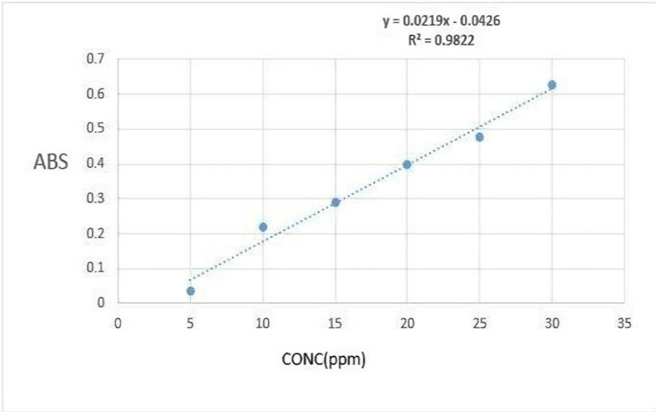

Figure 2. Regression curve of absorbance vs concentration of standard solution (Source: Authors' own elaboration)

absorbance and concentration, facilitating straightforward determination of naphthalene concentrations in complex environmental matrices.

Design Summary for the Optimization Studies

The optimization study aimed to enhance the efficiency of naphthalene removal from wastewater using kaolin clay as the adsorbent. Analysis of experimental data was facilitated by Design Expert software version 13.0.1.0. The BBD, augmented with five center point selections within a block, resulted in seventeen experimental runs. Quadratic interactions served as the model for analysis.

The factors investigated in this experimental design were the initial concentration of the solution, contact time, and pH of the solution, while maintaining the adsorbent dosage at a constant level. RE was the key metric evaluated to assess the effectiveness of the adsorption process, calculated according to Eq. (1).

Table 2 depicts the removal percentages achieved for each experimental run, illustrating various conditions and their corresponding efficiencies in naphthalene removal. Notably, the efficiency ranged from 64.92% to 87.69%, indicating significant variability in performance under different experimental settings.

Table 2. Removal percentage for each experimental run

RUNS	Co	ABS	Ce	Re	% Re
1	200	1.275	60.16438	0.699178	69.91781
2	300	1.382	65.05023	0.783166	78.31659
3	200	1.349	63.54338	0.682283	68.22831
4	200	1.092	51.80822	0.740959	74.09589
5	100	0.556	27.33333	0.726667	72.66667
6	200	1.250	59.02283	0.704886	70.48858
7	300	1.519	71.30594	0.762314	76.23135
8	300	1.533	71.94521	0.760183	76.01826
9	300	1.281	60.43836	0.798539	79.85388
10	200	1.306	61.57991	0.6921	69.21005
11	200	1.292	60.94064	0.695297	69.52968
12	200	1.260	59.47945	0.702603	70.26027
13	100	0.551	27.10502	0.72895	72.89498
14	100	0.449	22.44749	0.775525	77.55251
15	200	1.333	62.81279	0.685936	68.59361
16	200	1.494	70.16438	0.649178	64.91781
17	100	0.227	12.3105	0.876895	87.6895

Table 3. Summary of removal efficiency of naphthalene

Response	Name	Observations	Min	Max	Mean	SD	Ratio
R1	PR	17.00	64.92	87.69	73.32	5.52	1.35

Note. PR: Percent removal & SD: Standard deviation

Summary of removal efficiency of naphthalene

The highest removal percentage of 87.69% was achieved in experimental run 17, characterized by a pH of 7, an initial synthetic wastewater concentration of 200 mg/L, and a contact time of 120 minutes. This outcome underscores the efficacy of the adsorption process under specific conditions, highlighting the potential of kaolin clay as an adsorbent for naphthalene removal.

Table 3 provides a concise overview of the removal efficiencies observed across experimental runs. Notably, the mean RE stood at 73.32%, with a standard deviation of 5.52%, indicating a moderate level of variability in the results.

ANOVA of removal efficiency of naphthalene

The significance of the experimental model was assessed through ANOVA, employing the F-statistical test and p-values. These metrics elucidate the model's adequacy in explaining the variation in RE and the significance of individual factors and interactions.

Table 4 represent the ANOVA table which shows the contribution of each factor and interaction to the model's predictive capability. The model exhibited statistical significance (p-value = 0.0408), indicating its suitability for explaining the variation in naphthalene RE. Notably, the initial concentration of the solution (A), quadratic term of initial concentration (A^2), and quadratic term of pH (B^2) demonstrated significant influences on RE ($p < 0.05$).

Additionally, the lack of fit test indicated the insignificance of the model's lack of fit (p-value = 0.0737), affirming the model's adequacy in capturing the underlying trends in the data. The coefficient of determination ($R^2 = 0.8369$) signifies that approximately 83.69% of the variability in RE can be explained by the model, further supporting its validity.

Table 5 presents the BBD of the experiment showing the actual and predicted values of RE for each experimental run, along with the corresponding residuals. The close alignment between actual and predicted values, with minimal residuals, validates the accuracy of the experimental model in predicting naphthalene RE.

Adsorption Kinetics Models for the Study

In order to delve deeper into the adsorptive capacity of kaolin clay as an adsorbent for naphthalene in wastewater, a kinetics study was conducted. This investigation aimed to elucidate the adsorption behavior at various pollutant concentrations over time. Statistical analysis was performed on the results of each adsorption kinetics experiment, focusing on two models: pseudo-second-order and Elovich kinetics. The model that best described the experimental data for each case was determined to be pseudo-second-order, and subsequently, the constants of each equation and the equilibrium capacity (q) were calculated.

Table 4. ANOVA for quadratic model (percent removal)

Source	SS	df	MS	F-value	p	
Model	408.41	9	45.38	3.9900	0.0408	Significant
A-Initial conc	0.019	1	0.019	0.0016	0.9693	
B-pH	10.37	1	10.37	0.9122	0.3714	
C-Contact time	41.27	1	41.27	3.6300	0.0985	
AB	2.89	1	2.89	0.2541	0.6297	
AC	15.37	1	15.37	1.3500	0.2832	
BC	0.93	1	0.93	0.0819	0.7830	
A^2	272.36	1	272.36	23.9500	0.0018	
B^2	4.17	1	4.17	0.3663	0.5641	
C^2	51.29	1	51.29	4.5100	0.0713	
Residual	79.61	7	11.37			
Lack of fit	63.24	3	21.08	5.1500	0.0737	Not significant
Pure error	16.38	4	4.09			
Cor total	488.02	16				

Note. SS: Sum of squares; MS: Mean square; Standard deviation = 3.37; Mean = 73.32; CV% = 4.60; R^2 = 0.8369; Adjusted R^2 = 0.6271; Predicted R^2 = -1.1257; & Adeq Precision = 6.2802

Table 5. Box-Behnken design of the experiment

Run order	Actual value	Predicted value	Residual
1	69.92	68.36	1.5600
2	78.32	80.16	-1.8400
3	68.23	68.36	-0.1320
4	74.09	74.75	-0.6600
5	72.67	75.17	-2.5000
6	70.49	71.51	-1.0200
7	76.23	73.37	2.8600
8	76.02	79.54	-3.5200
9	79.85	77.35	2.5000
10	69.21	68.36	0.8480
11	69.53	68.36	1.1700
12	70.26	69.24	1.0200
13	72.89	75.75	-2.8600
14	77.55	75.71	1.8400
15	68.59	67.93	0.6600
16	64.92	68.36	-3.4400
17	87.69	84.17	3.5200

Table 6 presents the experimental data obtained from the adsorption kinetics study. Each sample (denoted as t1 through t8) was subjected to different pollutant concentrations (abs1, abs2, abs), initial pollutant concentrations in solution (Ce), and corresponding time intervals (time(min)). The initial pollutant concentration in solution (Co) was maintained at 100 mg/L. The adsorption capacity at each time interval (Qt), equilibrium time (time(min)), ratio of equilibrium time to adsorption capacity (t/Qt), and natural logarithm of time (lnt) are also recorded.

Pseudo second order kinetic model

The pseudo-second-order kinetic model is utilized to elucidate the adsorption process of naphthalene adsorbates onto kaolin adsorbent, wherein the interaction between the adsorbate molecule and functional groups on the surface of adsorbents governs the adsorption capacity of the adsorbent. **Figure 3** illustrates the linear graphs obtained from a plot of t/qt against t at a concentration of 100 mg/L pollutant.

Table 6. Adsorption kinetics table

Sample	abs1	abs2	abs	Ce	Time (min)	co	Qt	Time (min)	t/qt	lnt
t1	0.323	0.323	0.3230	16.69406	15	100	4.165297	15	3.601184	2.708050
t2	0.346	0.346	0.3460	17.74429	30	100	4.112785	30	7.294327	3.401197
t3	0.258	0.257	0.2575	13.7032	45	100	4.314840	45	10.429120	3.806662
t4	0.246	0.246	0.2460	13.17808	60	100	4.341096	60	13.821390	4.094345
t5	0.330	0.330	0.3300	17.01370	75	100	4.149315	75	18.075270	4.317488
t6	0.196	0.195	0.1955	10.87215	90	100	4.456393	90	20.195710	4.499810
t7	0.268	0.268	0.2680	14.18265	105	100	4.290868	105	24.470580	4.653960
t8	0.270	0.270	0.2700	14.27397	120	100	4.286301	120	27.996160	4.787492

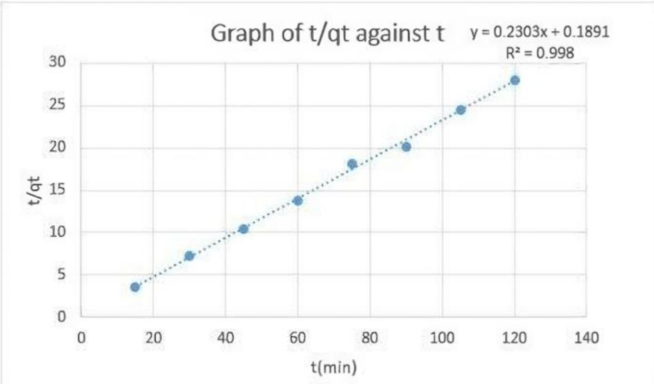

Figure 3. Pseudo second order kinetic plot (Source: Authors' own elaboration)

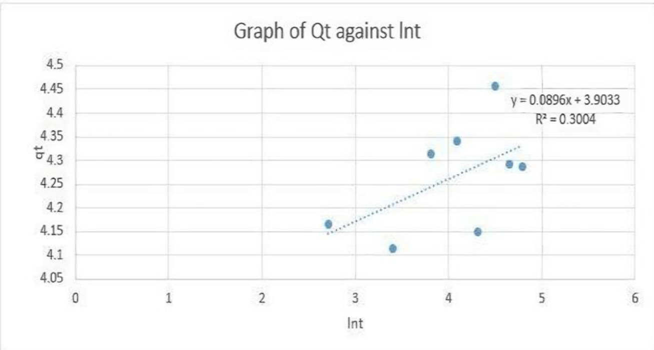

Figure 4. Elovich model plot (Source: Authors' own elaboration)

Elovish model

The Elovich model is employed to characterize adsorption processes that adhere to second-order kinetics, assuming that the surface of the adsorbent is energetically heterogeneous, thereby exhibiting varying activation energies. The experimental data is fitted into the linearized form of the model by plotting a graph of qt against lnt, as depicted in **Figure 4**.

Investigated Isotherms Results and Analysis

The investigation into the adsorption of naphthalene in wastewater utilizing kaolin clay as the adsorbent revealed intriguing insights into the relationship between naphthalene concentrations and adsorptive capacity, as summarized in **Table 7**. To elucidate these relationships, three commonly used isotherm models, namely Langmuir, Freundlich, and Temkin, were employed and analyzed.

Table 7. Investigated isotherm table

Re	Co	abs	ce	qe	lnqe	lnce	Ce/qe
0.902648	50	0.064	4.86758	2.256621	0.353459	0.687313	2.157021
0.921187	100	0.13	7.881279	4.605936	0.663318	0.896597	1.711113
0.876530	150	0.363	18.52055	6.573973	0.817828	1.267654	2.817254
0.692785	200	1.303	61.44292	6.927854	0.840599	1.788472	8.868969

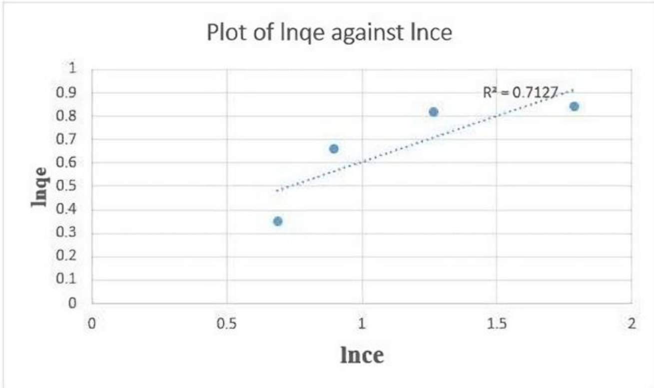

Figure 5. Freundlich model plot (Source: Authors' own elaboration)

The Freundlich isotherm, selected for its suitability for non-uniform or heterogeneous surfaces, provided valuable insights into the variation in surface properties and the distribution of active sites with different energies on the kaolin clay surface, as illustrated in **Figure 5**. The calculated Freundlich adsorption constant (Kf) of 1.0268 indicated highly favorable adsorption behavior, while the adsorptive intensity (n) of 6.188 suggested a robust affinity of naphthalene molecules for the kaolin clay surface. The coefficient of determination (R^2) value of 0.71 indicated a satisfactory fit of the Freundlich model to the experimental data.

In contrast, the Temkin isotherm model, which accounts for indirect adsorbate/adsorbate interactions, demonstrated a comparable adsorption constant (Kf) of 1.0268, consistent with the Freundlich model. The adsorptive intensity (n) remained at 6.188, indicating a strong affinity of naphthalene for the kaolin clay surface. Notably, the Temkin model exhibited a slightly improved fit, with a coefficient of determination (R^2) value of 0.79, as depicted in **Figure 6**.

Additionally, the Langmuir isotherm model was employed to evaluate the adsorption capacity of kaolin clay for naphthalene, as presented in **Figure 7**. The Langmuir model assumes monolayer adsorption onto a homogeneous surface with a finite number of identical sites. The graphical representation of Langmuir 1 showcased a rapid increase in adsorption capacity with increasing naphthalene

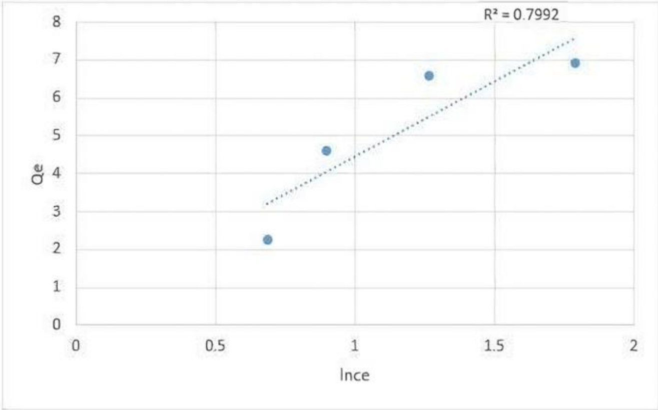

Figure 6. Temkin isotherm plot (Source: Authors' own elaboration)

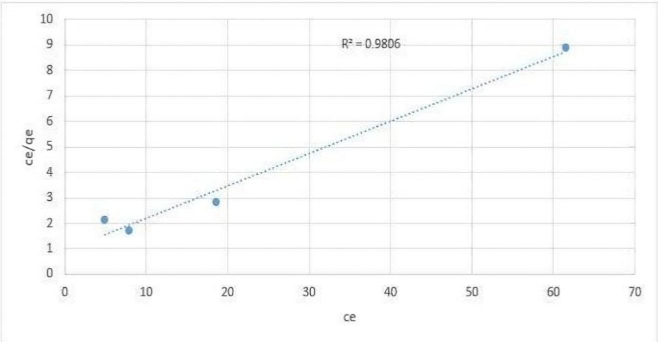

Figure 7. Langmuir isotherm plot (Source: Authors' own elaboration)

concentration, indicative of favorable adsorption behavior. The high coefficient of determination (R^2) value of 0.98 further confirmed the excellent fit of the Langmuir model to the experimental data.

Thermodynamics of Adsorption Process

In this investigation, an in-depth exploration of the thermodynamics governing the adsorption of naphthalene from wastewater onto kaolin clay was undertaken. Critical thermodynamic parameters, including enthalpy (ΔH°, kJ/mol), standard entropy (ΔS°, J/molK), and changes in Gibbs free energy (ΔG°, kJ/mol), were rigorously computed using Eq. (4)-Eq. (7) to elucidate the intricate adsorption mechanism.

$$k_c = \frac{C_0 - C_e}{C_e}. \quad (4)$$

$$\ln kc = -\Delta H0\ RT + \Delta S0\ R. \quad (5)$$

$$\Delta G0 = -RT\ln kc. \quad (6)$$

$$\Delta G0 = \Delta H0 - T\Delta S0. \quad (7)$$

The relationship between the rate constant (k) and temperature (T) was meticulously examined utilizing Equation 5, which enabled the determination of the slope and intercept from the graph of lnk against 1/T. Impressively, the resultant linear correlation, depicted in **Figure 8**, exhibited a notably high coefficient of determination (R^2) value of 0.94, indicating a robust association between the variables.

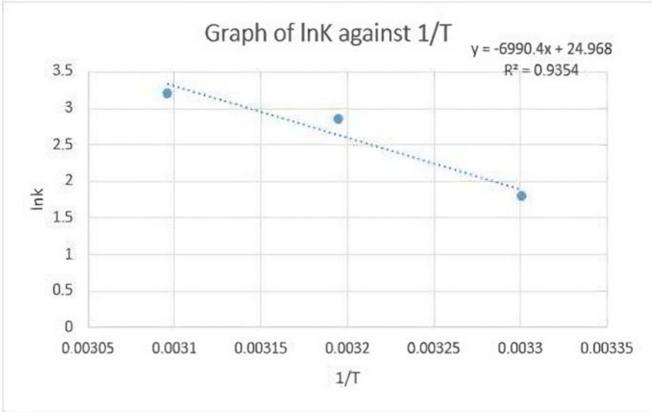

Figure 8. Graph of lnk against 1/T (Source: Authors' own elaboration)

Table 8. Thermodynamics of adsorption process

co	Temp	abs	ce	kc	lnkc	1/T
100	303	0.270	14.273970	6.005758	1.792719	0.003300
100	313	0.077	5.461187	17.311040	2.851344	0.003195
100	323	0.042	3.863014	24.886520	3.214326	0.003096

Analysis of the experimental findings presented in **Table 8** unequivocally illustrates that as the temperature escalates, the equilibrium concentration (Ce) experiences a corresponding reduction, leading to an escalation in the equilibrium constant (Kc). The computed lnKc values offer profound insights into the temperature-dependent behavior of the adsorption process.

By meticulously juxtaposing the slope and intercept with Eq. (6), the enthalpy (ΔH°) and entropy (ΔS°) variations were meticulously deduced. The positive ΔH° value (+58.118 kJ/mol/K) signifies an endothermic adsorption process, implying the requisite energy for the adsorption of naphthalene onto the kaolin clay surface. Additionally, the positive ΔS° value (+207.58 J/molK) suggests an increase in randomness at the solid/liquid interface, indicating consequential structural alterations in both the adsorbent and adsorbate.

Furthermore, the computation of the Gibbs free energy change (ΔG°) via Eq. (7) across different temperatures yielded negative values of ΔG° (-4514.30 J at 303 K, -7419.32 J at 313 K, and -8620.20 J at 323 K), unequivocally affirming the thermodynamic favorability of the adsorption process over the entire temperature range.

In conclusion, the meticulous thermodynamic analysis provides profound insights into the adsorption mechanism of naphthalene onto kaolin clay. The discernibly endothermic nature of the adsorption process, coupled with the positive entropy change and negative Gibbs free energy values, unequivocally highlight the favorable and spontaneous nature of naphthalene adsorption onto kaolin clay, thus advocating its potential as an efficient adsorbent for wastewater treatment endeavors.

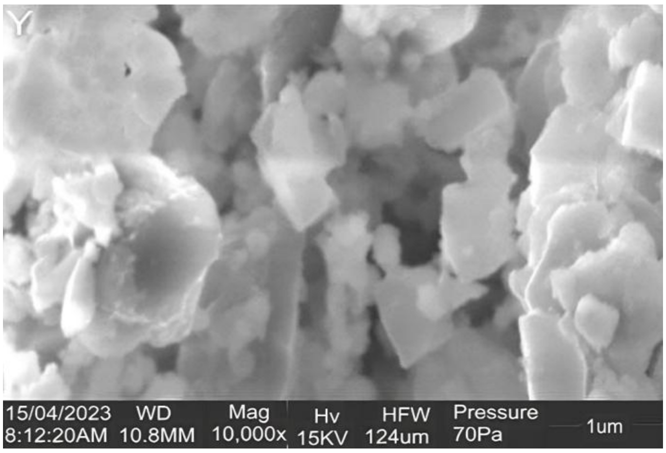

Figure 9. SEM analysis of raw kaolin at Mag 10,000*x*-1 (Source: Authors' own elaboration)

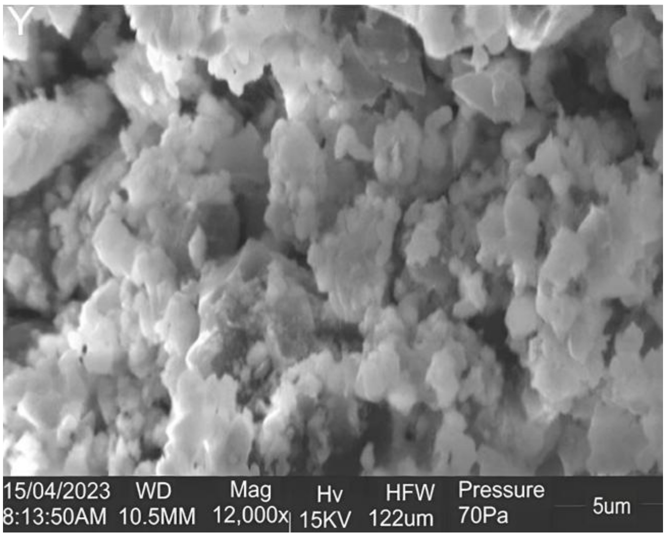

Figure 10. SEM analysis of raw kaolin at Mag 10,000*x*-2 (Source: Authors' own elaboration)

Characterization of Kaolin Adsorbent

Characterization of kaolin was done by the following technique, SEM and FTIR to study the morphology and structure of the Kaolin as a potential adsorbent.

Scanning electron microscopy

In this study, SEM played a crucial role in examining the morphology and surface characteristics of the materials under scrutiny, particularly focusing on the kaolin clay employed as an adsorbent for naphthalene removal from wastewater.

The SEM images, meticulously captured and presented in **Figure 9** and **Figure 10**, offer valuable insights into the inherent structure and morphology of the raw kaolin feed. These images vividly depict the dispersed nature of kaolinite, the predominant constituent of kaolin clay. The morphological examination, as illustrated in **Figure 10**, highlights the prevalent presence of typical kaolin morphology characterized by heterogeneous layered sheets of various sizes. The observed kaolin structure displays a size range of 1-5 µm, underscoring its heterogeneous nature.

Table 9. FTIR spectrum analysis

Frequency range (cm^{-1})	Functional group	Compound class
3,653.69	Hydroxyl (-OH)	Alcohols/phenols
2,976.36	C-H stretching	Alkanes
1,638.80	C=C stretching	Alkenes/amides
1,454.00-1,375.00	C-H bending	Alkanes
1,249.58	C-N stretching	Amines
1,155.00-1,064.00	C-O stretching	Ethers/esters

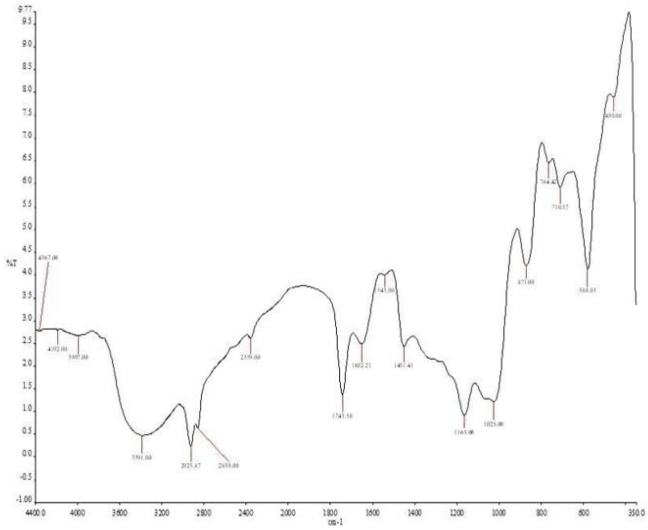

Figure 11. FTIR plot (Source: Authors' own elaboration)

Through SEM imaging, it becomes apparent that the surface of kaolin clay exhibits a complex and multi-layered structure, which is essential for its adsorption capabilities. The presence of heterogeneous layered sheets provides a substantial surface area and numerous active sites for the adsorption of naphthalene molecules from wastewater.

Furthermore, the observed morphology underscores the potential utility of kaolin clay as an effective adsorbent for wastewater treatment applications. The layered structure and heterogeneous surface of kaolin clay facilitate enhanced adsorption kinetics and efficiency, positioning it as a promising candidate for the removal of organic contaminants, such as naphthalene, from aqueous environments (**Table 9**).

Fourier-transform infrared spectral analysis

In this investigation, FTIR emerged as a critical analytical tool to discern the functional groups present in both raw kaolin samples as seen in **Figure 11**. Renowned for its ability to characterize material chemical compositions based on molecular vibrations, FTIR played a fundamental role in this study.

The FTIR spectra obtained for the raw and treated kaolin samples unveiled distinctive peaks corresponding to specific functional groups. Spectral scanning spanned frequencies from 4,400 to 350 cm^{-1} with a resolution of 4 cm^{-1}.

Characteristic peaks representing various functional groups were evident in both raw and treated kaolin FTIR spectra. Notably, the region between 3,600 and 3,200 cm^{-1} typically signifies the presence of hydroxyl (-OH) groups, commonly found in clay minerals like kaolinite.

Table 10. Comparative outlook on produced kaolin absorbent vs. other adsorbents

Absorbent	Removal efficiency	Reference
Modified kaolin	87.90%	Our work
Orange peel	74.81 ± 3.96%	Kalengyo et al. (2024)
Coconut shell	89.00%	Packialakshmi et al. (2023)
Sugarcane bagasse	85.20%	Ezeonuegbu et al. (2021)
Rice husk	80.00%	Mane et al. (2024)
Sawdust	45.00%	

Furthermore, peaks within the 1,700 to 1,600 cm^{-1} range denote the stretching vibrations of carbonyl (C = O) groups, potentially indicating the presence of carbonate or organic contaminants on the kaolin surface.

The spectral range from 1,200 to 1,000 cm^{-1} often exhibits peaks corresponding to Si-O stretching vibrations, characteristic of silicate minerals such as kaolinite. Additionally, peaks within the 800 to 600 cm^{-1} range may be attributed to bending vibrations of Si-O bonds, confirming the presence of silicate groups in the kaolin samples.

Alterations in the FTIR spectra upon treatment may indicate changes in chemical composition or surface functional groups of the kaolin samples. These changes could imply modifications aimed at enhancing adsorption properties or removing impurities from the kaolin surface.

In conclusion, FTIR offers valuable insights into the chemical composition and surface functional groups of both raw and treated kaolin samples, aiding in their characterization and understanding of adsorption behavior in wastewater treatment applications.

Comparative outlook on kaolin absorbent vs. other adsorbents

Table 10 presents a comparative analysis of the removal efficiencies of different adsorbents used for wastewater treatment. Our modified kaolin absorbent demonstrates a high RE of 87%, making it a competitive option among the tested adsorbents. This efficiency is particularly notable when compared to other naturally derived adsorbents such as orange peel and rice husk, which have removal efficiencies of 74.81 ± 3.96% and 80%, respectively. While these natural adsorbents are effective, they fall short of the performance exhibited by modified kaolin.

Coconut shell, with a RE of 89%, shows the highest efficiency among the adsorbents listed. This indicates its strong potential for wastewater treatment. However, the difference in efficiency between coconut shell and modified kaolin is marginal, suggesting that modified kaolin is a viable alternative with nearly comparable performance. Sugarcane bagasse, another natural adsorbent, also shows a high RE of 85.2%, closely matching that of modified kaolin. This further supports the effectiveness of modified kaolin as a competitive adsorbent.

On the other hand, sawdust exhibits the lowest RE at 45%, indicating that it may not be as suitable for high-efficiency wastewater treatment applications. This significant difference highlights the superior performance of modified kaolin and other high-efficiency adsorbents like coconut shell and sugarcane bagasse.

Overall, the modified kaolin prepared in our study demonstrates a strong potential for use in wastewater treatment, offering a balance between high RE and the benefits of being a naturally derived material. Its performance is competitive with other effective adsorbents, making it a promising option for sustainable and efficient wastewater treatment solutions.

CONCLUSION

This study explored the potential of low-cost kaolin clay as an effective adsorbent for the removal of naphthalene, an organic pollutant, from aqueous solutions. The experimental findings highlighted the remarkable efficacy and affordability of kaolin as an adsorbent for environmental remediation applications.

The results demonstrated that the adsorption capacity of kaolin was influenced by various operational parameters, including contact time, temperature, initial pollutant concentration, and pH of the solution. Notably, the pseudo-second-order kinetic model emerged as the most suitable model for describing the adsorption kinetics of naphthalene onto kaolin, indicating a chemisorption mechanism. Optimization studies revealed that under optimal conditions–pH of 7, initial pollutant concentration of 100 mg/L, and a contact time of 120 minutes–the highest RE of 86.69% was achieved. The mean RE across experiments reached a commendable 73.23%. Furthermore, the experimental data were successfully fitted to various adsorption isotherm models, with the Langmuir isotherm exhibiting the best fit ($R^2 = 0.98$). This suggests that the Langmuir model can reliably predict naphthalene adsorption onto kaolin, serving as a valuable tool for design purposes. Thermodynamic investigations revealed favorable conditions for the adsorption process, with negative ΔG values at all temperatures studied, indicative of spontaneous adsorption. Moreover, the positive ΔH values confirmed the endothermic nature of the process, suggesting chemisorption as the predominant mechanism. At the end of the study the following conclusions where drawn:

1. Kaolin clay is a cost-effective and efficient adsorbent for naphthalene removal from wastewater.
2. The adsorption capacity is influenced by contact time, temperature, initial pollutant concentration, and pH.
3. The pseudo-second-order kinetic model best describes the adsorption kinetics, indicating chemisorption.
4. Optimal conditions for maximum RE are pH 7, initial pollutant concentration of 100 mg/L, and a contact time of 120 minutes.
5. The Langmuir isotherm model provides the best fit for the adsorption data, suggesting monolayer adsorption.
6. Thermodynamic parameters indicate that the adsorption process is spontaneous and endothermic.
7. The study provides valuable insights for designing kaolin-based adsorption systems for environmental remediation.

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Ethical statement: The authors stated that ethics committee approval was not required for the work, therefore it was exempted. The study involves data collection using online resources involving information freely available in the public domain that does not collect or store identifiable data. All related laws, rules, and regulations required for the study's implementation have been followed. The authors further stated that the article is the original study of the authors, and it has not been published elsewhere.

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Data sharing statement: Data supporting the findings and conclusions are available upon request from corresponding author.

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