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Adsorption of Aspirin from Aqueous Solution onto Expanded Vermiculite: Preparation, Characterization, and Adsorption Behavior

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This study investigates the adsorption of aspirin from aqueous solution using expanded vermiculite as a low-cost adsorbent. Expanded vermiculite was prepared via hydrogen peroxide-assisted treatment combined with microwave irradiation, resulting in an exfoliated structure with enhanced surface area. Characterization by SEM, XRD, FT-IR, and BET confirmed significant structural and textural modifications after expansion. Batch adsorption experiments showed that the equilibrium data are well described by the Langmuir isotherm model, with a maximum adsorption capacity of 42.84 mg/g, indicating monolayer adsorption on relatively homogeneous active sites. Kinetic analysis revealed that the adsorption process follows the pseudo-second-order model, suggesting that chemisorption may play an important role. Intraparticle diffusion analysis further indicated that the adsorption proceeds through multiple steps. These findings demonstrate that expanded vermiculite is a promising and cost-effective adsorbent for the removal of aspirin from aqueous environments.

Keywords: expanded vermiculite, aspirin, adsorption, isotherm modeling, Kinetic study.

Introduction

The occurrence of pharmaceutical residues in aquatic environments has raised increasing environmental concerns due to their persistence, potential bioaccumulation, and adverse impacts on ecosystems and human health (González-González et al., 2022; Sánchez et al., 2025). Among these contaminants, aspirin (acetylsalicylic acid) is extensively consumed worldwide and is frequently detected in wastewater systems due to its incomplete removal during conventional treatment processes (De Oro-Carretero et al., 2026; Pires et al., 2024). Adsorption is considered a promising approach for the removal of pharmaceutical pollutants owing to its operational simplicity, efficiency, and cost-effectiveness (Fraiha et al., 2024). Various adsorbents, including activated carbon, biochar (Narloch et al., 2025; Wong et al., 2021), and engineered nanomaterials (Bawazeer, 2026; Mphahlele et al., 2015), have been investigated for aspirin removal. However, some of these materials present limitations, including high cost, complex preparation processes, or limited availability, which may restrict their large-scale application (Sahu et al., 2024).

Vermiculite, a naturally occurring layered silicate mineral, has attracted attention as a low-cost adsorbent owing to its high cation-exchange capacity and expandable layered structure. Thermal or chemical expansion can significantly improve its surface area and adsorption performance by exfoliating the layered structure and enhancing pore accessibility (Dao et al., 2025). Although expanded vermiculite has been extensively investigated for the removal of inorganic contaminants such as heavy metals and metalloids, its application for the adsorption of pharmaceutical pollutants remains largely unexplored. In particular, information regarding the adsorption behavior of aspirin on expanded vermiculite is still scarce. Furthermore, limited studies have examined how the structural modification induced by microwave-assisted expansion influences the adsorption performance of vermiculite toward pharmaceutical compounds. Therefore, investigating aspirin adsorption using expanded vermiculite not only broadens the application scope of this low-cost mineral adsorbent but also provides insights into its potential for the removal of emerging contaminants from aquatic environments.

In this study, expanded vermiculite was prepared via hydrogen peroxide-assisted treatment combined with microwave irradiation and evaluated as a low-cost adsorbent for aspirin removal from aqueous solution. The adsorption performance, equilibrium behavior, and adsorption kinetics were systematically investigated to assess the feasibility of using expanded vermiculite for the treatment of pharmaceutical contaminants.

Materials and Methods

Materials

Aspirin (analytical grade) was used as received from Sigma-Aldrich. Vermiculite (particle size 3–5 mm) was supplied by Shijiazhuang Haoges (China). Hydrogen peroxide (H_2O_2 , 30 wt%) was obtained from Macklin (China).

Preparation of expanded vermiculite

The preparation procedure of expanded vermiculite has been reported in our earlier work with slight modifications (Dao et al., 2025). In brief, a total of 1 g of vermiculite was placed in a Petri dish ($d = 120$ mm) containing 7 mL of H_2O_2 solution. The covered Petri dish was then kept in an oven at 60°C for 60 min. Subsequently, the Petri dish was transferred to a microwave oven (Sharp R-21A1SVN, 800 W, Japan) and irradiated for 60 s. After irradiation, the sample was allowed to cool to room temperature, facilitating the evaporation of residual solvent. This heating-cooling cycle was repeated five times until the solution was nearly completely evaporated. Finally, the obtained expanded vermiculite was dried at 120°C for 1 h and stored in a desiccator for further use.

Characterization techniques

The crystalline structure of the samples was characterized by X-ray diffraction (XRD) using a D8 Advance diffractometer (Bruker) over a 2θ range of 5 – 80° at a scanning rate of $2^\circ/\text{min}$. Surface morphology and elemental distribution were examined using a scanning electron microscope (SEM, Hitachi S-4600) equipped with an energy-dispersive X-ray spectroscopy (EDS) system. The specific surface area was determined using the Brunauer–Emmett–Teller (BET) method using a NOVA touch 2LX analyzer (Quantachrome). Fourier-transform infrared (FT-IR) spectra were recorded in the range of 4000 – 400 cm^{-1} using a TENSOR II

spectrometer (Bruker). The zeta potential of expanded vermiculite was measured at different pH values using a SZ-100 analyzer (HORIBA Scientific, Japan).

Aspirin adsorption experiments

A 100 mg/L aspirin stock solution was prepared by dissolving 20 mg of aspirin in distilled water and making up the volume to 200 mL. All adsorption experiments were conducted at pH 6. This pH was selected as a representative condition for aqueous environments and to evaluate the adsorption behavior of aspirin under conditions where it predominantly exists in its deprotonated form.

In a typical experiment, 0.1 g of adsorbent was added to 50 mL of aspirin solution with initial concentrations ranging from 10 to 80 mg/L in 100 mL conical flasks, followed by shaking at 120 rpm. At predetermined time intervals, the suspensions were centrifuged to separate the solid and liquid phases. The residual concentration of aspirin in the supernatant was determined using a UV-Vis spectrophotometer (Smart Thermostatted 7-Cell Rotary Changer, Thermo Scientific) at a wavelength of 280 nm, based on a previously established calibration curve. The adsorption capacity (q_e) of aspirin was calculated using the following equation:

$$q_e = \frac{(C_o - C_e) \cdot V}{m} \quad (1)$$

where C_o and C_e (mg/L) – the initial and equilibrium concentrations of aspirin in the solution, respectively; V (L) – the solution volume, and m (g) corresponds to the mass of the adsorbent.

Adsorption isotherms

Typically, 0.1 g of expanded vermiculite was added to 50 mL of aspirin solution with initial concentrations ranging from 10 to 80 mg/L, followed by shaking at 120 rpm for 180 min. To investigate the adsorption behavior, the experimental data were fitted using the Langmuir and Freundlich isotherm models, and their linear forms are expressed by the following equations (Narloch et al., 2025), respectively:

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

where q_e and q_m (mg/g) – the equilibrium and maximum adsorption capacities, respectively; C_e (mg/L) –

the equilibrium concentration of aspirin in solution; and K_L (L/mg) is the Langmuir constant.

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (3)$$

where K_F (mg/g) $\times$ (L/mg)^{1/n} – the Freundlich constant and n denotes the heterogeneity factor.

Adsorption kinetics

In a typical experiment, 0.1 g of expanded vermiculite was introduced into 50 mL of aspirin solution (initial concentration of 40 mg/L) and shaken at 120 rpm for 180 min. At selected time intervals, 5 mL samples were collected, centrifuged, and analyzed to determine the residual aspirin concentration. Kinetic data were evaluated using pseudo-first-order, pseudo-second-order, and intraparticle diffusion models, and their linearized forms are presented as follows (Narloch et al., 2025):

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \quad (4)$$

where q_e and q_t (mg/g) – the amounts of adsorbate adsorbed at equilibrium and at time t , respectively; t (min) – the contact time; k_1 (1/min) – the pseudo-first-order rate constant.

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (5)$$

where: k_2 – the rate constant of the pseudo-second-order (g/mg·min).

$$q_t = k_i t^{0.5} + C \quad (6)$$

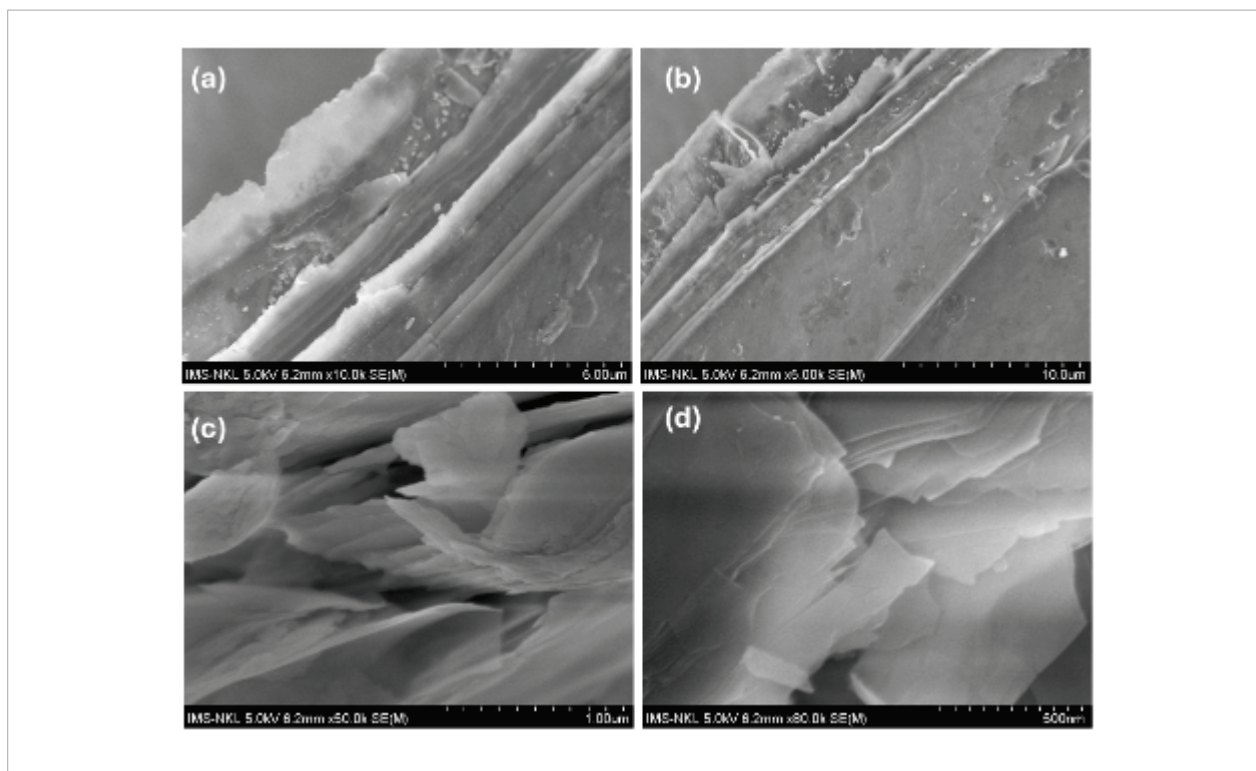
where k_i (mg/g·min) – the intraparticle diffusion rate constant; C (mg/g) – constant related to the thickness of the boundary layer.

Results and Discussion

Characterization of vermiculite materials

The physicochemical characteristics of raw vermiculite and expanded vermiculite were comprehensively investigated using SEM, EDS, XRD, FT-IR, and BET analyses. SEM images at different magnifications (Fig. 1a–d) reveal that raw vermiculite exhibits a typical lamellar morphology with compactly stacked plate-like layers, which is characteristic of vermiculite minerals containing interlayer water and exchangeable cations within a phyllosilicate framework (Wang, 2026).

Fig. 1. SEM images of (a,b) raw vermiculite, and (c, d) expanded vermiculite



After the expansion treatment, a pronounced morphological transformation is observed. The expanded vermiculite displays a clearly exfoliated and accordion-like structure, with enlarged interlayer spacing and a loosely packed architecture. This exfoliation behavior is attributed to the rapid release of interlayer water, which generates internal pressure and forces the silicate layers apart. This mechanism has been widely reported as the primary driving force responsible for vermiculite expansion and layer exfoliation (Assis Neto et al., 2023). The resulting open structure is expected to enhance surface accessibility and mass transfer.

The elemental compositions of raw and expanded vermiculite obtained from EDS analysis are presented in Fig. 2. Both samples mainly consist of O, Si, Mg, Al, Fe, and K, which are characteristic elements of vermiculite (Wang, 2026). The relatively stable contents of Si, Mg, and Al after expansion indicate that the fundamental silicate framework remains largely preserved during the treatment process. A slight increase in oxygen content may be associated with the hydrogen peroxide-assisted expansion procedure, whereas the decrease in Fe and Ca contents could be attributed to

local compositional heterogeneity and partial redistribution of exchangeable species during expansion. Importantly, no additional impurity elements were detected after treatment, confirming that the expansion process primarily induces physical exfoliation rather than significant chemical modification of the vermiculite structure.

The phase structures of raw and expanded vermiculite were further examined by XRD, as shown in Fig. 3a. Raw vermiculite exhibits characteristic basal reflections associated with its layered silicate structure (Lahchich et al., 2025). After expansion, the basal reflections show noticeable broadening and reduced intensity, accompanied by slight shifts in diffraction positions. These changes indicate an increase in interlayer spacing and a partial loss of long-range order along the c-axis, which are typical features of expanded vermiculite formed via exfoliation rather than complete structural collapse (Chen et al., 2023; Marcos, 2021). The retention of the characteristic diffraction peaks confirms that the layered vermiculite framework remains largely preserved after expansion.

Fig. 2. EDS spectra of (a) raw vermiculite, and (b) expanded vermiculite

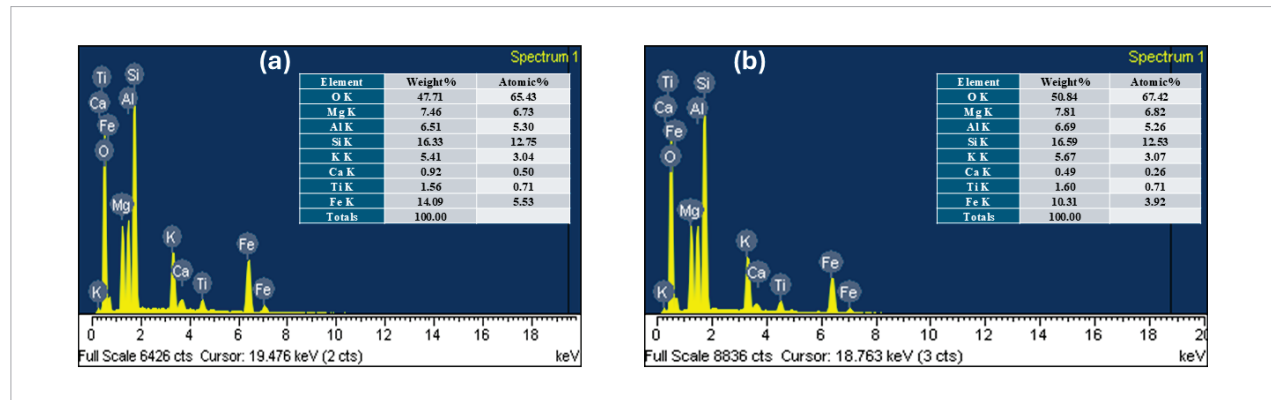
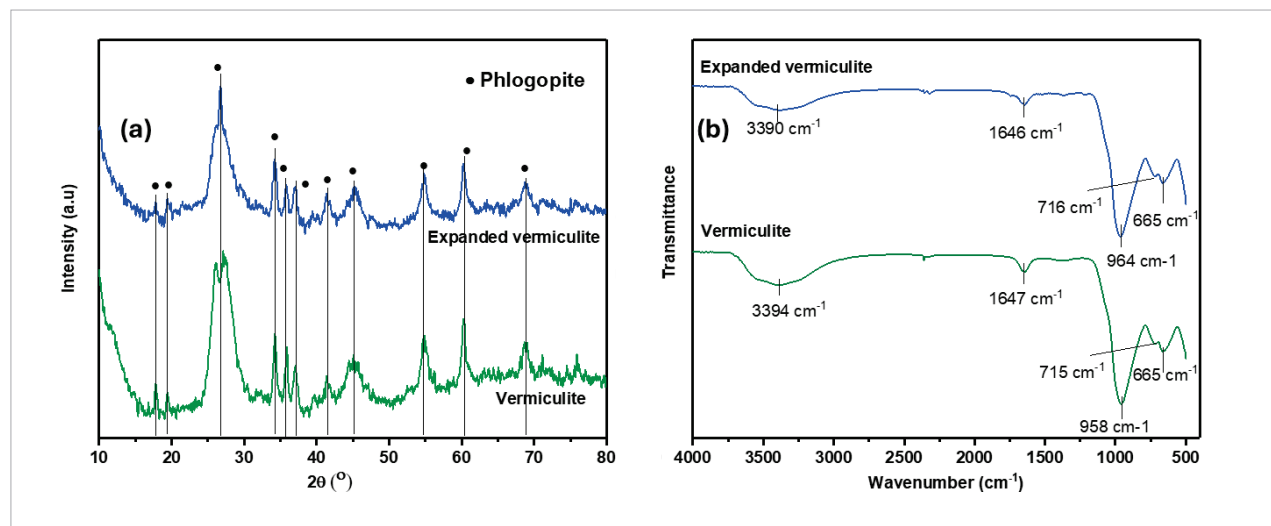


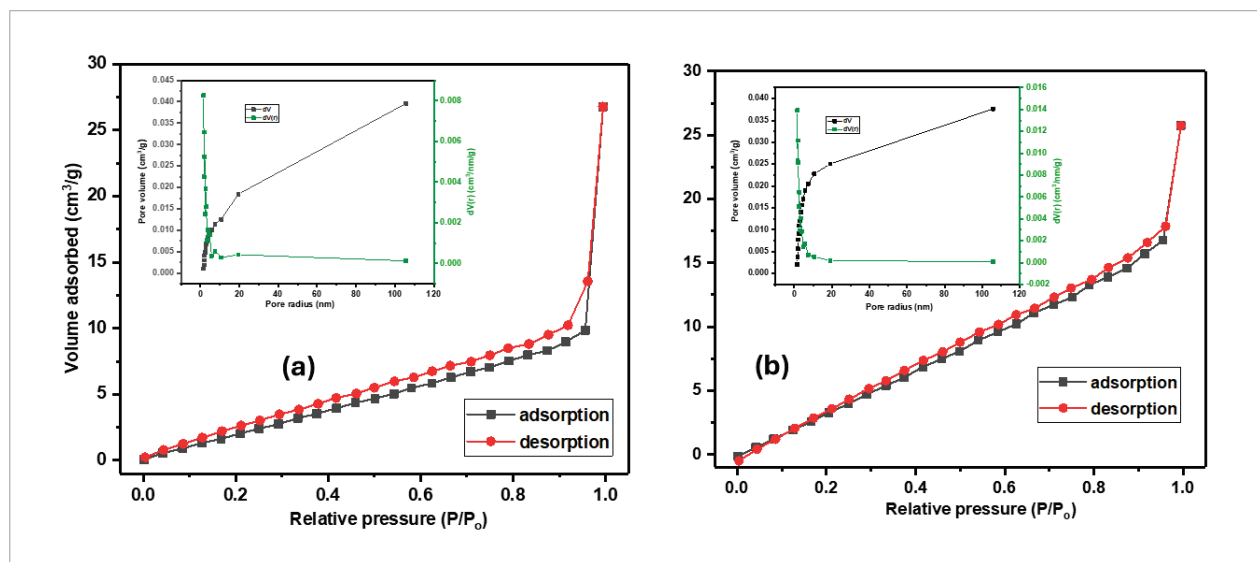
Fig. 3. (a) XRD patterns, (b) FT-IR spectra of raw and expanded vermiculite



FT-IR spectra of raw and expanded vermiculite are presented in Fig. 3b. Both spectra display broad bands in the range of 3330–3350 cm^{-1} , corresponding to the stretching vibrations of hydroxyl groups and interlayer water molecules, as well as bands near 1646–647 cm^{-1} attributed to H–O–H bending vibrations (Długosz et al., 2018). Strong absorption bands around 950–960 cm^{-1} are assigned to Si–O stretching vibrations of the tetrahedral silicate sheets, while bands in the range of 665–715 cm^{-1} are related to metal–oxygen vibrations (Mg–O and Al–O) in the octahedral layers (Onutai et al., 2023). After expansion, slight shifts and intensity changes of these bands are observed, suggesting modifications in hydrogen bonding environments and local structural order induced by layer exfoliation.

The textural properties of vermiculite before and after expansion were evaluated using BET analysis (Fig. 4). The specific surface area increases significantly from 14.97 m^2/g for raw vermiculite to 30.62 m^2/g after expansion, while the total pore volume shows a slight increase from 0.0384 to 0.0395 cm^3/g . The average pore radius remains nearly constant at approximately 1.60 nm. The substantial increase in surface area is attributed to layer exfoliation during expansion, which generates additional accessible surface sites. Meanwhile, the nearly unchanged pore radius suggests that the expansion treatment primarily enhances surface accessibility without significantly altering the pore structure. This behavior is consistent with the exfoliation mechanism of vermiculite reported in the literature (Valášková et al., 2012).

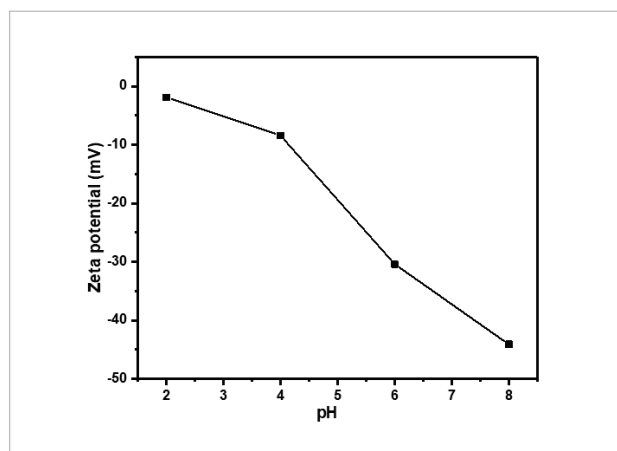
Fig. 4. N_2 adsorption–desorption isotherms and pore size distribution of (a) vermiculite and (b) expanded vermiculite



The zeta potential of expanded vermiculite was measured at different pH values to evaluate the surface charge characteristics of the adsorbent (Fig. 5).

As the pH increased from 2 to 8, the zeta potential decreased from -1.9 to -44.1 mV, indicating that the surface became increasingly negatively charged. This behavior can be attributed to the progressive deprotonation of surface hydroxyl groups on the vermiculite structure, leading to a higher density of negatively charged sites at higher pH values. The results suggest that expanded vermiculite possesses a predominantly negative surface charge over the investigated pH range, with the isoelectric point estimated to be below

Fig. 5. Effect of pH on the zeta potential of expanded vermiculite



pH 2. The increasing negative surface charge may influence the interaction between the adsorbent surface and dissolved species in aqueous environments.

Adsorption isotherms

The equilibrium adsorption data of aspirin onto expanded vermiculite were analyzed using the Langmuir and Freundlich isotherm models to elucidate the adsorption behavior and surface characteristics of the adsorbent. As shown in Fig. 6 and Table 1, both isotherm models adequately described the experimental data. However, the Langmuir model exhibited a higher correlation coefficient ($R^2 = 0.992$) than the Freundlich model ($R^2 = 0.976$). This result indicates that the adsorption of aspirin onto expanded vermiculite is more appropriately described by the Langmuir equation.

The superior fit of the Langmuir model suggests that aspirin adsorption predominantly occurs through monolayer adsorption on relatively homogeneous sites distributed over the expanded vermiculite surface. The maximum adsorption capacity (q_m) obtained from the Langmuir model was 42.84 mg/g, indicating favorable adsorption of aspirin onto the adsorbent. Furthermore, the Langmuir constant ($K_L = 0.609$ L/mg) suggests a strong interaction between aspirin molecules and the expanded vermiculite surface. This adsorption behavior is consistent with the structural characteristics of expanded vermiculite, particularly its exfoliated morphology and increased specific surface area, which

improve the accessibility of adsorption sites and facilitate aspirin uptake.

Although the Freundlich model showed a slightly lower fitting quality, its applicability remains meaningful for interpreting the surface heterogeneity of expanded vermiculite. The Freundlich constant $K_F = 13.388$ further confirms the appreciable adsorption capacity of the material, while the heterogeneity factor $n = 1.867$ indicates a favorable adsorption process, since values of $n > 1$ are generally associated with strong adsorbate–adsorbent interactions and non-uniform surface energies. Therefore, the Freundlich fitting suggests that, besides the dominant monolayer adsorption tendency indicated by the Langmuir model, the expanded vermiculite surface still retains a certain degree of heterogeneity, which is reasonable for a naturally derived mineral material subjected to thermal expansion.

The adsorption of aspirin onto expanded vermiculite may involve multiple interactions, including hydrogen

bonding, surface complexation, and electrostatic interactions. As shown in Fig. 5, the zeta potential of expanded vermiculite decreased from -1.9 mV at pH 2 to -44.1 mV at pH 8, indicating that the surface became increasingly negatively charged with increasing pH. Considering that aspirin has a pKa of approximately 3.5, it predominantly exists in its deprotonated form at pH 6, the pH employed in the adsorption experiments. Under these conditions, both the adsorbent surface and aspirin molecules are negatively charged, suggesting that electrostatic attraction is unlikely to be the dominant adsorption mechanism. Therefore, the observed adsorption performance is more likely associated with hydrogen bonding and surface-related interactions involving hydroxyl groups, exchangeable cations, and the layered silicate structure of vermiculite. In addition, the expansion process not only increases the external surface area but also improves pore accessibility, thereby promoting the diffusion and retention of aspirin within the adsorbent structure.

Fig. 6. Linear fitting of adsorption isotherms for aspirin onto expanded vermiculite (a) Langmuir model (C_e/q_e vs. C_e), and (b) Freundlich model ($\log q_e$ vs. $\log C_e$)

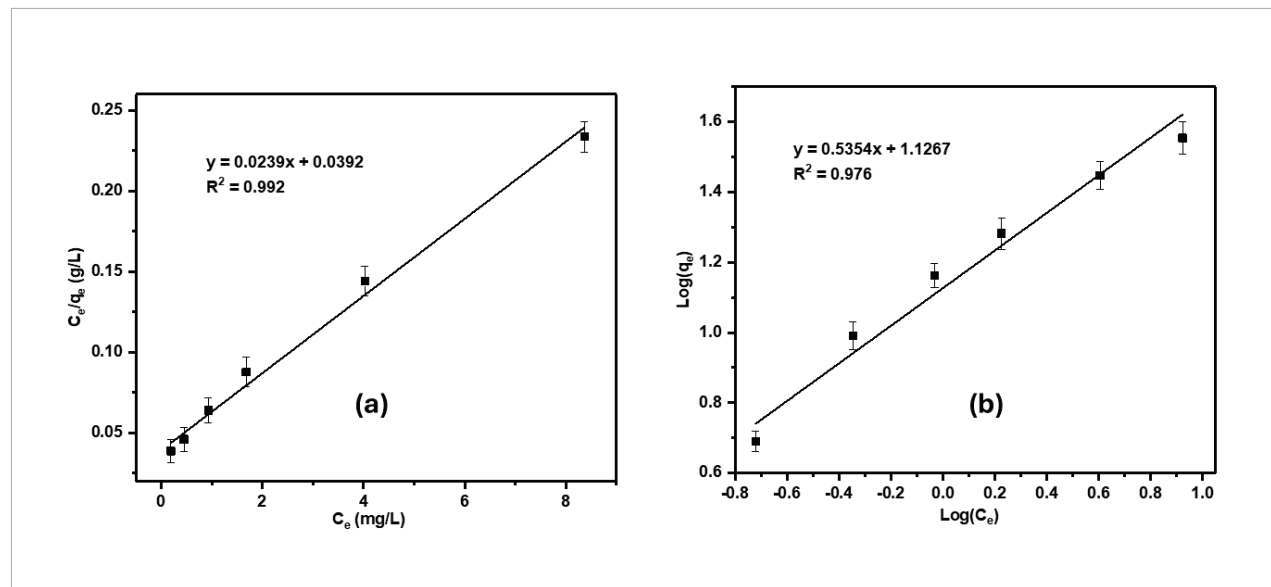


Table 1. Adsorption isotherm parameters of aspirin onto expanded vermiculite.

	Langmuir model			Freundlich model		
	q_m (mg/g)	K_L (L/mg)	R^2	$K_F [(mg/g) \cdot (L/mg)^{1/n}]$	n	R^2
Aspirin	42.84	0.609	0.992	13.388	1.867	0.976

The adsorption performance of expanded vermiculite was further compared with various reported adsorbents, including clay- and mineral-based materials, as summarized in *Table 2*. The results show that expanded vermiculite exhibits a relatively high adsorption capacity (42.84 mg/g), which is substantially higher than those reported for natural clay and mineral adsorbents such as zeolite, organobentonite, and organokaolin. In addition, its performance is comparable to that of more advanced materials, including tyre waste-derived adsorbents and functionalized carbon nanotubes. These findings suggest that expanded vermiculite can achieve competitive adsorption performance while maintaining the advantages of low cost, natural abundance, and simple preparation.

Adsorption kinetics

The adsorption kinetics of aspirin onto expanded vermiculite were analyzed using pseudo-first-order, pseudo-second-order, and intra-particle diffusion models (*Fig. 7*), and the corresponding parameters are summarized in *Table 3*. As shown in *Fig. 7a and 7b*, the pseudo-second-order model exhibited a better fit to the experimental data ($R^2 = 0.982$) compared to the pseudo-first-order model ($R^2 = 0.876$), indicating that the adsorption process may be governed by surface interactions involving valence forces. The calculated adsorption capacity obtained from the pseudo-second-order model was in reasonable agreement with the experimental value, further supporting the applicability

of this model. These results suggest that chemisorption may play an important role in the adsorption process, likely involving valence forces through electron sharing or exchange between aspirin molecules and the active sites of the adsorbent (Narloch, 2025).

The kinetic profile presented in *Fig. 7c* further confirms the suitability of the pseudo-second-order model, as evidenced by the close agreement between the experimental and predicted adsorption capacities throughout the adsorption process. The rapid initial uptake of aspirin is attributed to the abundance of available adsorption sites on the expanded vermiculite surface. As these sites become progressively occupied, the adsorption rate decreases and the system gradually approaches equilibrium.

The intraparticle diffusion plot (*Fig. 7d*) exhibits a multilinear pattern, indicating that the adsorption process is governed by multiple mass-transfer steps rather than solely by intraparticle diffusion. The first stage is characterized by a relatively high intraparticle diffusion rate constant ($k_i = 1.882 \text{ mg/g}\cdot\text{min}^{1/2}$), which can be attributed to rapid external mass transfer (film diffusion) and adsorption on readily accessible surface sites. In contrast, the second stage exhibits a much lower diffusion rate constant ($k_i = 0.112 \text{ mg/g}\cdot\text{min}^{1/2}$), corresponding to the gradual diffusion of aspirin molecules into less accessible regions of the adsorbent. Furthermore, the non-zero intercept values and the fact that the fitted lines do not pass through the origin indicate that intraparticle diffusion is not the sole rate-controlling mechanism and that film diffusion also contributes to the overall adsorption process.

Table 2. Comparison of aspirin adsorption capacities of expanded vermiculite with various adsorbents

Adsorbents	q_m (mg/g)	Reference
Crushed knotweed (<i>Fallopia x bohemica</i>) leaves	17.3	Mucha (2017)
Tyre Waste Adsorbent	40.4	Azman et al. (2019)
Pristine functionalized multiwall carbon nanotubes (PMCNTs)	41	Elamin et al. (2019)
Walnut shell biochar	20.92	Narloch (2025)
Natural zeolitic tuff (Z)	1.98	Rakić et al. (2013)
Organobentonite (OB)	3.78	Rakić et al. (2013)
Organokaolin (OK-90)	3.78	Rakić et al. (2013)
Expanded vermiculite	42.84	This work

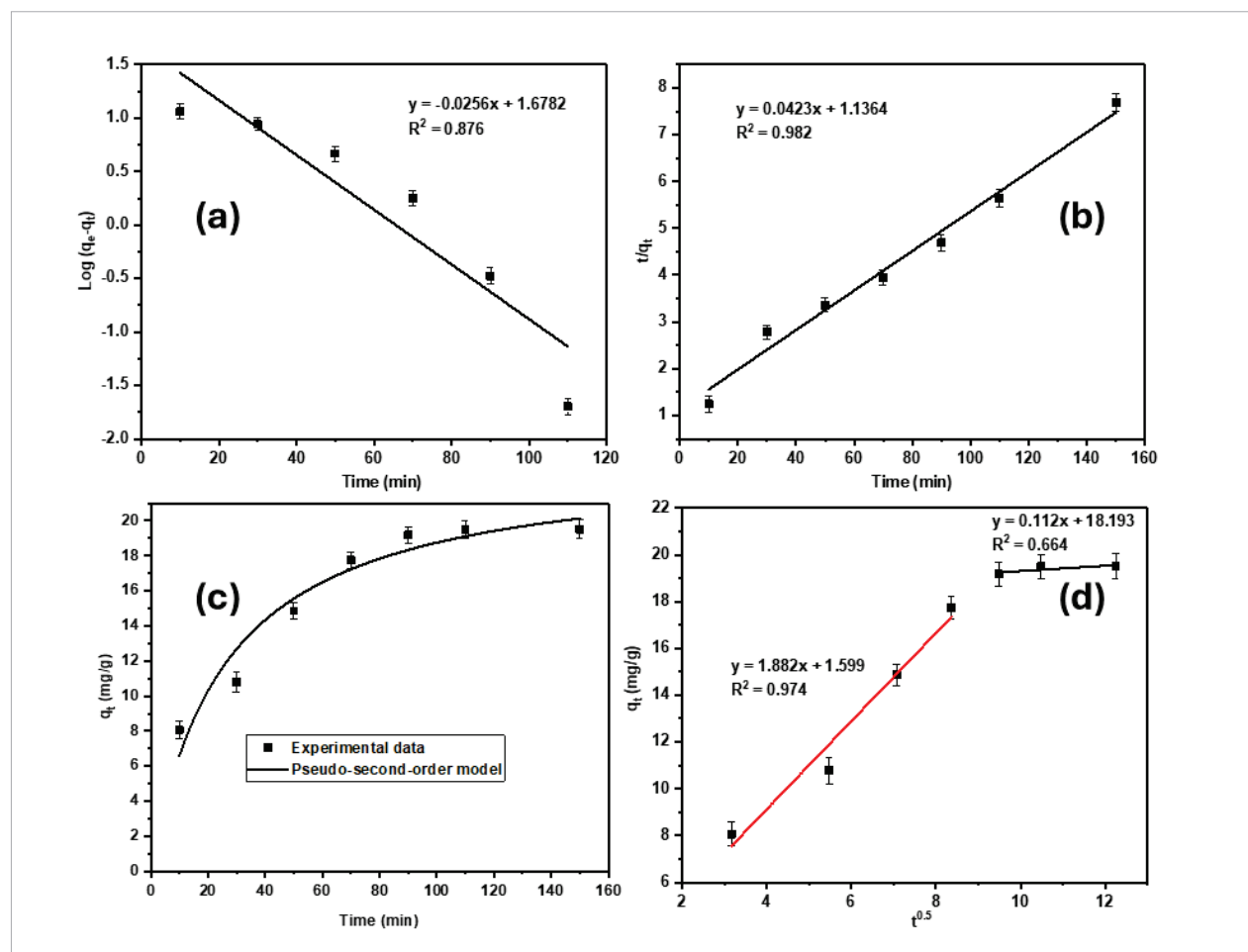
Table 3. Parameters for the kinetic models

Models	Parameters	Unit	Values	
Pseudo-first-order model	q_e	mg/g	47.665	
	k_1	1/min	0.059	
	R^2	-	0.876	
Pseudo-second-order model	q_e	mg/g	23.641	
	k_2	g/mg·min	0.002	
	R^2	-	0.982	
Intraparticle diffusion model	-	-	Step 1	Step 2
	C	mg/g	1.599	18.193
	k_i	mg/g·min ^{1/2}	1.882	0.112
	R^2	-	0.974	0.664

Overall, the kinetic analysis demonstrates that aspirin adsorption onto expanded vermiculite is dominated by pseudo-second-order kinetics, with contributions from both surface adsorption and intra-particle diffusion

processes. This behavior is consistent with the structural characteristics of expanded vermiculite, which provides accessible adsorption sites along with a porous network facilitating mass transfer.

Fig. 7. Adsorption kinetics of aspirin onto expanded vermiculite: (a) pseudo-first-order model, (b) pseudo-second-order model, (c) comparison between experimental data and the pseudo-second-order kinetic model, and (d) intra-particle diffusion model



Conclusions

Expanded vermiculite was successfully prepared through a combined hydrogen peroxide-assisted and microwave expansion process, resulting in a highly exfoliated structure with enhanced surface properties. The material exhibited effective adsorption of aspirin from aqueous solution, and the adsorption behavior was well described by the Langmuir isotherm and pseudo-second-order kinetic models. The improved adsorption performance can be attributed to the

structural modification induced by the expansion treatment, which increased the accessibility of adsorption sites and facilitated mass transfer. Compared with other reported clay- and mineral-based adsorbents, expanded vermiculite demonstrated competitive adsorption performance while retaining the advantages of low cost, natural abundance, and simple preparation. These findings highlight the potential of expanded vermiculite as a sustainable adsorbent for the removal of pharmaceutical contaminants from water. Future studies may focus on evaluating adsorption performance under a

wider range of operating conditions and exploring the practical application potential of expanded vermiculite in water treatment systems.

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