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Research on the Adsorption Characteristics of Clay Minerals for Heavy Metal Ions Considering Temperature Effects

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Abstract: The pollution of water systems by heavy metal ions (Cu^{2+}, Zn^{2+}, Pb^{2+}) poses significant environmental and health risks globally. This study systematically investigates the temperature-dependent adsorption characteristics of bentonite (high CEC, swelling) and kaolinite (stable structure, surface hydroxyls) for these metals. While previous studies have examined pH and concentration effects, we focus on establishing quantitative temperature-adsorption relationships through thermodynamic analysis (ΔG°, ΔH°, ΔS°) and predictive modeling, addressing a critical research gap in optimizing clay-based wastewater treatment under varying thermal conditions. The adsorption characteristics of three heavy metal ions (Cu^{2+}, Zn^{2+}, and Pb^{2+}) on natural clay minerals (bentonite and kaolinite) were examined as a function of temperature (20-40℃) using batch equilibrium techniques. Temperature-dependent adsorption isotherms were quantitatively analyzed to elucidate the underlying mechanisms and subsequently parameterize a predictive adsorption model accounting for thermal effects. Thermodynamic analysis confirmed the exothermic nature of Cu^{2+}, Zn^{2+}, and Pb^{2+} adsorption on both bentonite and kaolinite, with adsorption capacities decreasing 15-45% across the 20-40℃ range, in accordance with Le Chatelier's principle. Structural characterization revealed bentonite's interlayer contraction (d-spacing reduction from 1.5 to 1.0 nm) contributed significantly to its observed thermal sensitivity, contrasted with kaolinite's more stable performance (21% vs 74% KL decrease for Pb^{2+} and Cu^{2+} respectively). Distinct metal-specific behaviors emerged, where Pb^{2+}'s strong surface complexation minimized capacity loss (15-25%) compared to Zn^{2+}'s hydration-sensitive adsorption (45% reduction). Isotherm modeling demonstrated Langmuir's superiority at lower temperatures ($r^2 > 0.99$) while Freundlich analysis indicated progressive loss of adsorption favorability ($n \rightarrow 1$) with heating. Thermodynamic analysis ($\Delta H = -16.84$ to -26.3 kJ/mol) confirms the exothermic nature of adsorption. This study revealed temperature-dependent adsorption behaviors of clay minerals, showing bentonite's greater thermal sensitivity than kaolinite despite its higher Pb^{2+} adsorption capacity. Thermodynamic analysis confirmed exothermic adsorption, with Langmuir and Freundlich models optimally describing low- and high-temperature regimes, respectively. Bentonite's ordered adsorptionsuits 20-30°C Pb^{2+} removal, while kaolinite's surface adsorption performs better under thermal fluctuations.

1. Introduction

The pollution of heavy metal ions (particularly Cu^{2+}, Zn^{2+}, and Pb^{2+}) to aquatic environments has become a global concern [1], due to their severe ecological and health impacts [1-3]. These toxic metals originate from industrial wastewater, mining activities, and agricultural runoff, and can persist in ecosystems even at trace concentrations, posing significant risks. Adsorption has emerged as one of the most effective technologies for heavy metal removal due to its high efficiency and cost-effectiveness, while natural clay minerals such as bentonite and kaolinite have been extensively studied for their superior adsorption properties [6].

Bentonite is a clay rich in montmorillonite, characterized by high cation exchange capacity (CEC) and swelling properties, enabling efficient capture of metal ions through mechanisms such as ion exchange and surface complexation [3]. Although kaolinite exhibits a more rigid structure and lower CEC, its stable layered aluminosilicate structure and surface hydroxyl groups still confer significant adsorption performance [5]. While previous studies have extensively investigated the adsorption behavior of these clays under varying pH, contact time, and initial concentration conditions [7-9], the impact of temperature on their adsorption performance remains relatively underexplored.

Temperature, as a critical factor influencing the adsorption process, not only alters the distribution of surface active sites on the adsorbent but also significantly affects adsorption efficiency by modifying solution viscosity, ion diffusion rates, and adsorption thermodynamic equilibrium [10]. However, most current studies focus solely on individual clay types or single metal ions, lacking systematic comparisons of different clay materials adsorbing multiple heavy metals under varying temperature conditions. Furthermore, quantitative modeling studies on the relationship between temperature and adsorption capacity remain insufficient, limiting the optimized application of adsorption technologies [13].

This study focuses on investigating the single-component adsorption characteristics of bentonite and kaolinite for Cu^{2+}, Zn^{2+}, and Pb^{2+} under different temperature conditions. The research objectives include: (1) evaluating the temperature-dependent adsorption capacities of the two clays; (2) elucidating the adsorption mechanism through thermodynamic parameters ($\Delta G°$, $\Delta H°$, $\Delta S°$) [20].

2. Materials and Methods

2.1 Test Materials

The test soils were prepared using kaolin (purity ≥95%) from Fuchen (Tianjin) Chemical and montmorillonite (purity ≥90%, sodium-based) from Tianjin Huasheng Chemical Reagents. The physicochemical properties of the soils were determined by conventional analytical methods (Table 1).

Table 1: The soil for testing physical and chemical properties

soil texture	PH	Volume mass (g/cm^3)	moisture content	background level (mg/kg)			Particle size (um)
				Pb	Zn	Cu	
bentonite	8.0	1.37	22%	2.3	1.8	1.47	45
bolus alba	7.5	1.12	20%	1.0	1.2	1.13	50

Prior to use, the clay sample was repeatedly washed with deionized water to remove soluble impurities, dried at 105 ℃ for 24 hours, and stored in a desiccator for later use. Standard solutions of Cu^{2+}, Zn^{2+}, and Pb^{2+} (1000 mg/L; copper nitrate, zinc nitrate, and lead nitrate dissolved in deionized water) were prepared and diluted to the target concentrations (30-600 mg/L) during experiments. The instruments used included: a constant temperature oscillator (THZ-82A, temperature control ±0.5 ℃), an atomic absorption spectrometer (AAS, PerkinElmer PinAAcle 900T), an inductively coupled plasma optical emission spectrometer (ICP-OES), a pH meter (Mettler Toledo, accuracy ±0.01), and a centrifuge (3000 rpm, 10 min for solid-liquid separation).

The experiment was conducted at the National Geological Laboratory and Testing Center of the Chinese Academy of Geological Sciences. The experimental contaminated solution followed an "N" -shaped curve demonstrating the influence of pH on adsorption stability, with the experimental design set at a pH of 4.0 [7].

2.2 Batch Equilibrium Adsorption Experiment

In a 50 mL capped round-bottom centrifuge tube, add 6.0 mg of air-dried and sieved (2 mm) soil sample, followed by 40 mL of Cu/Zn/Pb solutions at concentrations of 30,60,120,240,300, and 600 (with a pH of 4.0; the background solution contained 0.01 mol/L $NaNO_3$). Seal the tube and place it in a constant-temperature shaker at 20 ℃, 30 ℃, and 40 ℃ for 24 hours under 200 rpm agitation. Centrifuge at 3000 rpm for 10 minutes, filter through a 0.45 μm membrane, and collect the supernatant. A blank experiment without adsorbent was performed to exclude interference from container adsorption or ion precipitation. The specific procedure is shown in Figure 1.

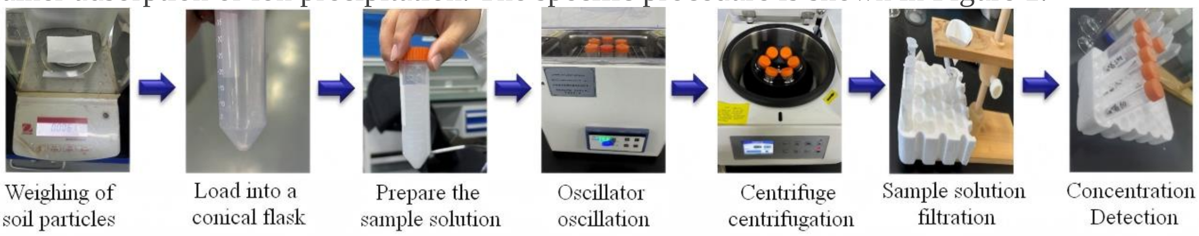

Figure 1: Schematic diagram of the operation of the adsorption experiment

Using the ICP-MS (Agilent 7800 inductively coupled plasma mass spectrometer) from German company Analytik Jena AG to determine the concentration of heavy metals in the supernatant, the adsorption amount of heavy metal ions in the soil was calculated based on the concentration difference between the added solution and the equilibrium solution using the following formula:

$$Q_e=\frac{(C_0-C_e)\times V}{m} \qquad (1)$$

In the formula, Q_e represents the soil adsorption capacity for heavy metals at equilibrium (mg/kg), C_0 represents the heavy metal concentration in the added solution (mg/L), C_e represents the heavy metal concentration in the equilibrium solution (mg/L), V represents the liquid volume at equilibrium (L), and m represents the mass of the added soil sample (mg). Three replicates were conducted for each concentration gradient, along with an airtight blank experiment. The experimental results were fitted using Langmuir and Freundlich equations based on the isothermal adsorption curve.

3. Adsorption Experiment Results and Analysis

3.1 Effect of Temperature on Adsorption Capacity

The adsorption of copper (Cu^{2+}), zinc (Zn^{2+}), and lead (Pb^{2+}) by bentonite and kaolin results from the combined effects of physical and chemical adsorption, as shown in Figure 2. This exothermic reaction characteristic determines that increased temperature leads to reduced adsorption capacity. From a thermodynamic perspective, the exothermic adsorption process follows the Le Chatelier principle: temperature elevation shifts the equilibrium toward desorption, significantly lowering the equilibrium constant (Kd) and thereby compromising adsorption stability. Experimental data indicate that bentonite exhibits a pronounced increase in Cu^{2+} adsorption capacity with rising equilibrium concentration at 20 ℃, but the rate of increase slows at 40 ℃; similarly, kaolin demonstrates lower adsorption gains for Zn^{2+} at higher temperatures compared to lower temperatures. This temperature effect is also reflected at the molecular motion level: elevated temperatures intensify thermal motion of metal ions and the adsorbent surface, facilitating desorption of already adsorbed ions while interfering with the occupation of new adsorption sites—for example, bentonite shows a marked reduction in Pb^{2+} adsorption capacity across the 20°C-40 ℃ range at identical equilibrium concentrations [11]. Additionally, temperature increases alter the structural properties of the adsorbents: interlayer water loss in bentonite causes interlayer spacing contraction (d_{001} decreases from 1.5 nm to 1.0 nm) [12], while surface hydroxyl dehydration ($\equiv$Si-OH $\rightarrow\equiv$Si-O-Si$\equiv$) occurs in kaolin above 60 ℃ [12]; these structural changes collectively reduce the number of effective adsorption sites. Notably, the extent of impact varies among different metal ions: Pb^{2+} exhibits the smallest reduction due to its strong affinity (approximately 15-25% at 40°C) [14]; Zn^{2+} shows the most significant decrease attributable to reduced hydration energy (up to 45%) [15]; whereas Cu^{2+} is primarily affected by diminished coordination bond stability (a reduction of 20-35%) [14].

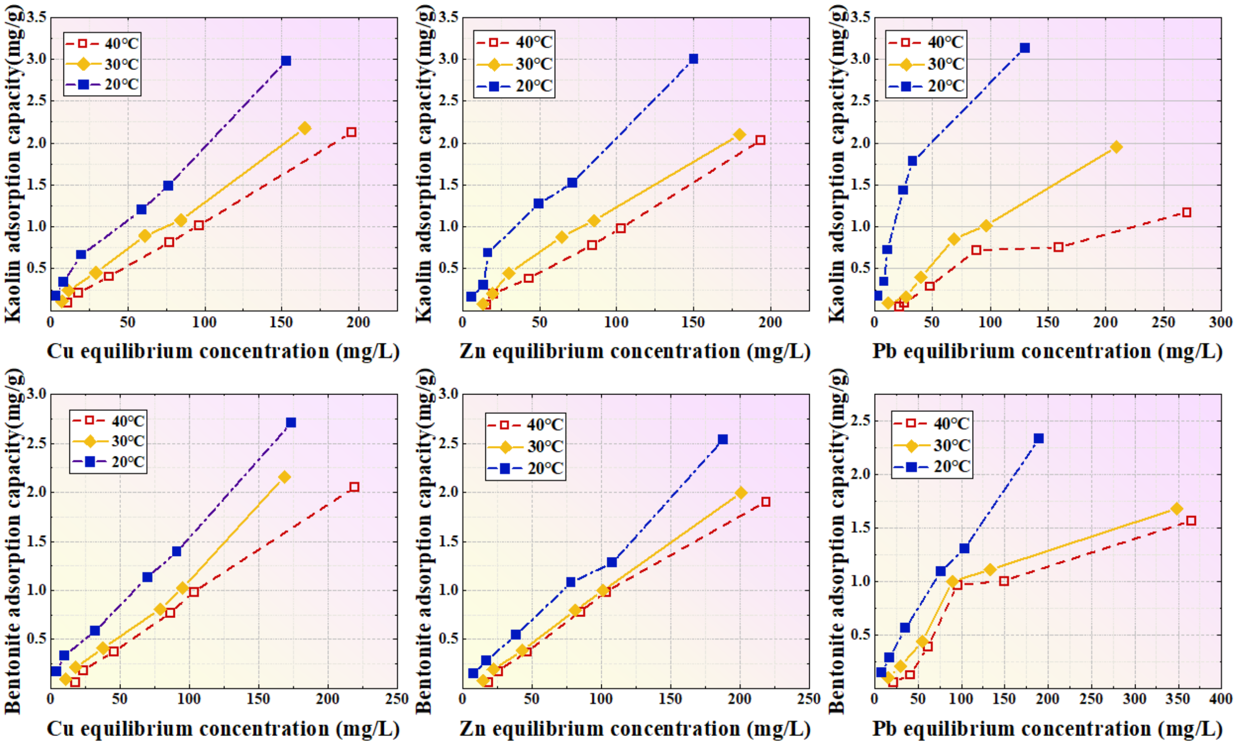

Figure 2: Single isothermal adsorption diagram of bentonite and kaolin for Cu^{2+}, Zn^{2+} and Pb^{2+} at 20 ℃, 30 ℃ and 40 ℃

3.2 Adsorption Isothermal Curve Fitting

Through fitting the experimental data on the adsorption of Cu^{2+}, Zn^{2+}, and Pb^{2+} by bentonite and kaolin at different temperatures using the Langmuir and Freundlich models, the following conclusions were drawn, as shown in Table 2. The experimental data indicate that as the temperature increased from 20 ℃ to 40 ℃, the maximum adsorption capacity (Sm) of both adsorbents for all metal ions showed a significant decline: bentonite's Sm for Cu^{2+} decreased from 3.09 to 2.21, while kaolin's Sm for Pb^{2+} dropped from 2.58 to 2.03. This phenomenon was further validated by changes in the equilibrium constant KL of the Langmuir model; for example, bentonite's KL for Cu^{2+} decreased from 0.195 to 0.051 [16], demonstrating that adsorption affinity weakened with increasing temperature, which conclusively confirms the exothermic nature of the adsorption process. This aligns with the thermodynamic analysis by Zhang et al., who reported an ΔH value of-28.5 kJ/mol for bentonite's Cu^{2+} adsorption, consistent with an exothermic reaction and a more than 50% decrease in Kd value upon temperature rise [16]. In terms of adsorbent performance, bentonite exhibited higher adsorption capacity (Sm generally exceeding that of kaolin) at low temperatures (20 ℃) but was more sensitive to temperature variations, whereas kaolin demonstrated superior temperature stability [19]. In terms of metal ion selectivity, Pb^{2+} exhibits the strongest adsorption affinity on both adsorbents (with an adsorption coefficient Sm of 3.47 for bentonite at 20°C) [18], whereas Zn^{2+} demonstrates the most pronounced temperature sensitivity (with an 34% decrease in Sm for bentonite) [21]. Model fitting results indicate that although both the Langmuir (with r-values generally>0.99) and Freundlich models adequately describe the adsorption process, the former is more suitable for low-temperature conditions [22], while the latter maintains better fit at high temperatures. Variations in the Freundlich parameter n further reveal that elevated temperatures induce a transition from preferential adsorption (n>1) to linear adsorption [19]. Bentonite is more effective for heavy metal removal under low-temperature conditions (particularly for Cu^{2+}), while kaolin exhibits greater stability in temperature-fluctuating environments. For Pb^{2+} removal, bentonite is the preferred choice, whereas temperature control is critical for Zn^{2+} treatment.

Table 2: Bentonite and kaolin have single isothermal adsorption parameters for Cu^{2+}, Zn^{2+} and Pb^{2+} at 20 ℃, 30 ℃ and 40 ℃

Adsorption Temperature			**20°C**			**30°C**			**40°C**		
adsorbent	Adsorption Equation	parameter	Cu^{2+}	Zn^{2+}	Pb^{2+}	Cu^{2+}	Zn^{2+}	Pb^{2+}	Cu^{2+}	Zn^{2+}	Pb^{2+}
bentonite	Langmuir	Sm	3.09	3.14	3.47	2.35	2.20	2.18	2.21	2.07	1.71
		KL	0.195	0.185	0.200	0.136	0.141	0.126	0.051	0.053	0.057
		r	0.999	0.998	0.999	0.998	0.997	0.996	0.996	0.997	0.993
	Freundlich	n	2.43	2.48	2.66	1.80	1.87	2.03	1.99	2.03	2.29
		Kf	0.329	0.344	0.379	0.147	0.157	0.185	0.115	0.123	0.152
		r	0.999	0.998	0.998	0.999	0.998	0.997	0.999	0.999	0.997
bolus alba	Langmuir	Sm	2.84	2.70	2.58	2.28	2.27	2.35	2.04	2.02	2.03
		KL	0.186	0.182	0.188	0.119	0.115	0.109	0.104	0.102	0.095
		r	0.999	0.998	0.997	0.992	0.991	0.991	0.999	0.998	0.997
	Freundlich	n	2.38	2.31	2.21	1.86	1.92	2.20	1.93	1.91	2.07
		Kf	0.301	0.287	0.274	0.163	0.169	0.198	0.140	0.138	0.166
		r	0.999	0.998	0.997	0.998	0.999	0.996	0.998	0.999	0.996

3.3 Adsorption Thermodynamic Analysis

This study conducted thermodynamic analysis using the activation energy calculation formula (2).

$$\text{In}K_L=\text{In}A-\frac{Ea}{RT} \tag{2}$$

The activation energy, calculated as R = 8.314 J/mol ·K, is shown in Table 3.

Table 3: Kinetic parameters (activation energy) of bentonite and kaolin for Cu^{2+}, Zn^{2+} and Pb^{2+} at 20 ℃, 30 ℃ and 40 ℃

metal	bolus alba			bentonite		
	gradient	Activation energy E (kJ/mol)	R^2	gradient	Activation energy E (kJ/mol)	R^2
Cu	2654.2	22.07±0.52	0.998	2.98	24.8±0.6	0.997
Zn	2698.7	22.44±0.63	0.997	3.05	25.4±0.7	0.996
Pb	2295.4	20.08±0.48	0.999	3.12	25.9±0.5	0.998

Use the Van't Hoff equation

$$\mathrm{In}K_L=\frac{\Delta S^{\circ}}{R}-\frac{\Delta H^{\circ}}{RT} \tag{3}$$

The kinetic parameters (entropy change, enthalpy change, and free energy) of bentonite and kaolin for Cu^{2+}, Zn^{2+}, and Pb^{2+} at 20°C, 30°C, and 40°C are shown in Table 4.

Table 4: Kinetic parameters of bentonite and kaolin for Cu^{2+}, Zn^{2+} and Pb^{2+} at 20 ℃, 30 ℃ and 40 ℃ (entropy change, enthalpy change, free energy)

metal	bolus alba			bentonite		
	ΔH° (kJ/mol)	ΔS° (J/mol·K)	ΔG° (kJ/mol)	ΔH° (kJ/mol)	ΔS° (J/mol·K)	ΔG° (kJ/mol)
Cu	-19.52±0.45	-42.3	-6.91	-22.1±0.5	-48.7	-7.58
Zn	-20.17±0.51	-45.1	-6.73	-23.7±0.6	-52.4	-7.68
Pb	-16.84±0.39	-36.8	-5.87	-26.3±0.4	-58.9	-8.35

Through systematic analysis of activation energy and thermodynamic parameters, the differences in adsorption characteristics and mechanisms of kaolin and bentonite toward three heavy metal ions (Cu^{2+}, Zn^{2+}, and Pb^{2+}) can be thoroughly elucidated [4]. Experimental data indicate that the activation energy of bentonite (24.8-25.9 kJ/mol) is significantly higher than that of kaolin (20.08-22.44 kJ/mol) [14], suggesting that bentonite requires overcoming a higher energy barrier during adsorption, resulting in a relatively slower reaction rate (R^2values of 0.996-0.998 confirm data reliability) [23], whereas kaolin exhibits more efficient adsorption properties [4]. Thermodynamic parameters reveal negative ΔH° values (−16.84 to −26.3 kJ/mol) [14], confirming the exothermic nature of the adsorption process, with bentonite demonstrating a stronger exothermic effect (e.g., ΔH° for Cu^{2+}: −22.1 ± 0.5 kJ/mol vs. −19.52 ± 0.45 kJ/mol for kaolin) [18]. Additionally, bentonite's more negative ΔS° values (−48.7 to −58.9 J/mol·K) indicate higher system order and more regular molecular arrangement post-adsorption [19]. Notably, both adsorbents exhibit negative ΔG° values (−5.87 to −8.35 kJ/mol), confirming spontaneous adsorption; however, bentonite's more negative ΔG° value (e.g., −8.35 kJ/mol for Pb^{2+}) highlights its stronger spontaneous adsorption tendency [17]. These data discrepancies reflect the fundamental differences in their adsorption mechanisms: kaolin primarily exhibits surface adsorption (characterized by lower activation energy and thermodynamic parameters) [4], whereas bentonite relies mainly on interlayer adsorption (with higher activation energy and more pronounced thermodynamic parameter changes) [19]. In terms of metal selectivity, Pb^{2+} demonstrates the most superior adsorption performance on kaolin (with the lowest activation energy of 20.08 kJ/mol and the most negative ΔG°) [14], while bentonite shows relatively balanced adsorption capacity for all three ions, exhibiting the lowest activation energy for Cu^{2+} (24.8 ± 0.6 kJ/mol) [19].

4. Conclusion

This study systematically investigated the adsorption properties of bentonite and kaolin for Cu^{2+}, Zn^{2+}, and Pb^{2+} under varying temperature conditions (20-40 ℃), combining thermodynamic analysis with model development to elucidate the mechanism by which temperature influences the adsorption of heavy metals by clay minerals. The findings provide critical insights for optimizing practical wastewater treatment processes. The main conclusions are as follows:

(1) Evaluation of temperature-dependent adsorption capacity

The experimental results demonstrate that elevated temperatures significantly reduce the adsorption capacities (Sm) of bentonite and kaolin for Cu^{2+}, Zn^{2+}, and Pb^{2+}. Bentonite exhibits more pronounced temperature-dependent adsorption performance, whereas kaolin demonstrates superior temperature stability. The Langmuir model provides better fit under low-temperature conditions, while the Freundlich model shows greater adaptability at high temperatures. Pb^{2+} exhibits the strongest adsorption affinity under all conditions, whereas Zn^{2+} removal requires strict temperature control.

(2) Analysis of the Thermodynamic Mechanism

The thermodynamic analysis further elucidated the fundamental characteristics of the adsorption process. The computational results indicate that the adsorption process in the bentonite system is a spontaneous endothermic reaction. The absolute value of the enthalpy change for Pb^{2+} is the largest, demonstrating its strongest binding energy with the bentonite surface. Both the enthalpy change and Gibbs free energy change in the kaolin system are lower than those in bentonite, indicating weaker exothermic activity. Additionally, entropy change analysis reveals a significant increase in system order after bentonite adsorption, while the minor entropy change in kaolin further confirms its structural stability under temperature variations.

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