

Adsorption of Nickel and Iron Metals by Coke Breeze (CB)

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Abstract— The adsorption of nickel and iron from liquid solutions by coke breeze (CB) has been investigated. The process was studied in a batch system with respect to the initial pH, contact time, temperature, adsorbent dose and metals initial concentration. The results showed that the experimental data was well represented by second order reaction for nickel and first order for iron. Based on the kinetic modeling the values of activation energy were 51.92 KJ/mole for nickel and 8.936 KJ/mole for iron. The equilibrium data for CB indicated that that adsorption of nickel followed Langmuir isotherm and iron followed Freundlich isotherm. The thermodynamic parameters (Δ Ho, Δ So and Δ Go) calculated from the experimental data indicated that the process was endothermic and spontaneous.

Keywords— Adsorption, Coke breeze, Nickel, Iron.

I. INTRODUCTION

The discharge of heavy metal into aquatic ecosystem has become a matter of concern over the last few decades. The presence of heavy metals in such aquatic environments is known to cause severe damage to aquatic life, beside the fact that these metals kill microorganisms during biological treatment of waste water with a consequent delay of the process of water purification. Most of the heavy metals salts are soluble in water and form aqueous solutions and consequently cannot be separated by ordinary physical means of separation.

The effect of heavy metals on man and the environment

Heavy metals are usually classified as the following three categories: toxic metals (such as Hg, Cr, Pb, Zn, Cu, Ni, Cd, As, Co, Sn, etc.), precious metals (such as Pd, Pt, Ag, Au, Ru etc.) and radionuclides (such as U, Th, Ra, Am, etc.), whose specific weight is usually more than 5.0 g/cm3 (Volesky, 1990). With the rapid development of various industries (including mining and smelting of metalliferous, surface finishing industry, energy and fuel production, fertilizer and pesticide industry, metallurgy, iron and steel, electroplating, electrolysis, electro-osmosis, leatherworking, photography, electric appliance manufacturing, metal surface treating, aerospace and atomic energy installation), wastes containing metals are directly or increasingly indirectly discharged into the environment, especially in developing countries, having brought serious environmental pollution, and threatened biolife (Volesky, 1990).

Due to their increasing application and their immutable nature, heavy metal pollution has naturally become one of the

most serious environmental problems today. Metal toxicity may affect all forms of life including microorganisms, plants and animals, but the degree of toxicity varies for different organisms. Physical, chemical and biological processes may combine under certain circumstances to concentrate metals rather than dilute them (Ashraf and Ali, 2007).

Several researchers have demonstrated that heavy metal contamination can cause shifts in microbial populations. Heavy metals may be mobilized by local and global changes in soil conditions i.e., changes in physical and chemical conditions of soil environment, including decrease in pH, redox potential and enhanced decomposition of organic matter. Heavy metals may exert toxic effects on soil microorganism hence resulting in the change of the diversity, population size and overall activity of the soil microbial communities.

Nickel is a toxic heavy metal that is widely used in oil refineries. The chronic toxicity of nickel to humans and the environment has been documented (Aikpokpodion et al., 2010). Nickel (II) is present in the effluents of silver refineries, electroplating, zinc based casting and storage battery industries (Singh et al., 2009). Nickel salts are commonly used in metal plating and its concentration in industrial wastewater varies between 6 to12 mg/L, which is above the safe limit (0.15 mg/L). It is essential to remove nickel from industrial, wastewater before being discharged. High nickel concentration causes gastrointestinal irritation, lung and bone cancers (Kumar and Kirthika, 2009). In animals, the toxic effect of Ni includes "dermatitis nickel-itch" and causes respiratory disorders. Out of all the compounds of nickel, nickel carbonyl is the most toxic. It is a carcinogen and a half an hour exposure to 30 ppm of this compound is lethal to humans.

Iron toxicity is limited to ingested or environmental exposure. Iron does not appear on the ATSDR's "Top 20 List," but it is a heavy metal of concern, particularly because ingesting dietary iron supplements may acutely poison young children (e.g. as few as five to nine 30-mg iron tablets for a 30-lb child). In drinking-water supplies, iron (II) salts are unstable and are precipitated as insoluble iron (III) hydroxide, which settles out as a rust-colored silt. Anaerobic ground waters may contain iron (II) at concentrations of up to several milligrams per liter without discoloration or turbidity in the water when directly pumpe from a well, although turbidity and color may develop in piped systems at iron levels above 0.05–



0.1 mg/L. Staining of laundry and plumbing may occur at concentrations above 0.3 mg/L (Cundy et al., 2008)

Methods for heavy metals removal from solution

Conventional methods for removing metal ions from aqueous solution include chemical precipitation, ion exchange, electrochemical treatment, membrane technologies, adsorption on activated carbon, etc. However, chemical precipitation and electrochemical treatment are ineffective, especially when metal ion concentration in aqueous solution is as low as 1 to 100 mg/L. They also produce large amount of sludge that has to be treated with great difficulties. Ion exchange, membrane technologies and activated carbon adsorption process are extremely expensive, especially when treating a large amount of water and wastewater containing heavy metal in low concentration, so they cannot be used at large scale. As well, most of these processes generate concentrated solutions of the metal, which may require chemical treatment for disposal or reuse.

Adsorption

adsorption is one of the most respective methods that was successfully applied for the removal of metal ions from hazardous wastes due to its low maintenance costs, high efficiency and ease of operation (Volesky, 1990). An alternative process is biosorption, which utilizes various natural materials of biological origin, including bacteria, fungi, yeast, algae, etc. These biosorbents possess metal sequestering properties and can decrease the concentration of heavy metal ions in solution from ppm to ppb level. They can effectively sequester dissolved metal ions out of dilute complex solutions with high efficiency and quickly. Therefore, biosorption is an ideal candidate for the treatment of high volume and low concentration.

II. MATERIALS AND EXPERIMENTAL WORK

Coke Breeze (CB).

Coke breeze is the undersize of metallurgical coke which is used in the production of pig iron. Table 1 shows the chemical compositions of the coke breeze, and the X-ray phase analysis is shown in (Figure 1) and Table 2 shows the chemical analysis of carbon breeze ash and particle size distribution of coke breeze provided in (Figure 2).

haracteristic	Ash content	Volatile mater	Fixed carbon	Sulfur content
w/w%	10.23	0.97	88.80	0.96
Relative Intensity	•		o Carbon	

Fig. 1. X-ray diffractogram of CB

From Figure 1, it is clear that the main phases present in CB are quartz, calcite and carbon.



Fig. 2. Particle size distribution of CB.

TABE 2. Chemical analysis of CB Ash											
Element	SiO ₂	Fe ₂ O ₃	CaO	MgO	Al ₂ O ₃	MnO	K ₂ O	SO ₃	P_2O_5	ZnO	Na ₂ O
w/w%	48.97	13.36	2.72	1.09	27.66	0.13	1.98	0.90	0.24	0.11	0.46

Chemicals and reagents

All chemicals used were of analytical reagent (AR) grade for obtaining reliable, consistent and accurate results. Doubledistilled water for analysis and adsorption experiments was used. Stock solutions for all the tested metal ions were prepared by dissolving the appropriate amount of analytical grade salts in double-distilled water. Working standard metal ion solutions were prepared by appropriate dilution.

Determination of Nickel (Dimethylglyoxime method) (APHA 1985)

Nickel ion Stock Solution 1g/L Ni: dissolve 4.7859g of nickel sulfate hexahydrate in 1000 mL double distilled water and add 1 ml concentrated sulfuric acid to prevent hydrolysis, (1 mL = $1000 \ \mu g \ Ni$).

Standard Nickel solution was prepared by measuring suitable volumes of stock solution of nickel into measuring flask to get 10, 25, 50, 75, 100, 150, 200 and 250 mg/L of nickel.

Dimethyleglyoxime (DMG) was prepared by weight by weighing 0.1 g of dry solid in 100 mL 95% ethanol. Bromine water was prepared by saturation of distilled water by bromine Ethyl alcohol 95%

Standard calibration curve for nickel ion solution

To a series of measuring flasks 100 ml 50, 100, 150, 200, and 250 μ g of nickel, add 25 mL HCl 1N and 5 ml Br₂ water. Cool under cold running water and add 10 ml conc. NH₄OH immediately. Add 20 mL DMG and 20 mL ethanol then dilute to 100 mL. The absorbance is measured at 445 nm after 10



min from adding DMG and a calibration curve (Figure 3) was obtained. According to this procedure, concentration of Ni (II) solution was given by the following equation:

(Concentration of Ni in (mg/L) = [Absorbance/(0.002*10)])



Fig. 3.Calibration curve for nickel ion standard solutions

Determination of Iron (phenanthroline method) (APHA 1985)

Iron (Fe^{2+}) Stock Solution: 7.02 g Fe (NH₄)₂(SO₄)₂. H₂O were dissolved in 50 mL distilled water containing 20 mL conc. sulfuric acid. 0.1M KMnO₄ was added dropwise until a faint pink color and the solution was diluted to 1 L using distilled water (1 mL = $1000 \mu g Fe^{2+}$)

Standard iron solution: was prepared by measuring suitable volumes of stock solution of iron into measuring flask to get 10, 25, 50, 75, 100, 150, 200 and 250 mg/L of iron stock solution.

Hydroxylamine hydrochloride solution: 10 g NH₂OH.HCl in 100 mL distilled water.

Ammonium acetate buffer solution (pH 4): 250 g $NH_4C_2H_3O_2$ was dissolved in 150 mL distilled water and 700 mL concentrated "glacial" acetic acid were added and the solution was completed to 1 L.

Phenanthroline solution: 100 mg 1,10 phenanthroline (C₁₂H₈N₂H₂O) in 100 mL distilled water and 2 drops of conc. HCl was added to the water

Potassium permanganate solution0.1 M: 0.316 g KMnO4 were dissolved in 100 mL

Calibration curve for iron ion solution

In a series of measuring flask (50 mL), add 20, 40, 100, 150 and 200 µgFe.1 ml hydroxyl amine and 5 ml acetate buffer solution and shake well. 2 mL of the phenanthroline solution were added and the volume was completed to 50 ml. the absorbance was measured after 10 minutes at 510 nm and a calibration curve (Figure 4) was obtained. Concentration of Fe (II) solution was given by the following equation:

(Concentration of Fe in (mg/l) = [Absorbance/(0.004*10)])

Methods

Adsorption procedures

Batch equilibration technique was used to determine the optimum sorption conditions pH, shaking time, initial concentration of metal ion, adsorbent weight and temperature. 50 mL of the standard solution of each metal ion was used in all adsorption experiments. The uptake percentage was calculated as follows: (1)

Uptake $\% = (C_o - C/C_o) \times 100$



Fig. 4. Calibration curve for Iron standard solutions

Where C_o and C are the concentration of the metal ion in the initial and final solutions (mg/L). The amount of metal ion uptake qt at time t, was calculated from the mass balance equation (2.).

$$= (C_o - C_t)V/W \tag{2}$$

Where t is the equilibrium contact time, $C_t = C_{o}$, $q_t = q_e$ and the amount of metal ion sorbed at equilibrium, V is the volume of the solution (mL), and W is the weight of the adsorbent (g). a) Effect of Adsorbent Weight

To establish the optimum weight of the adsorbent material for metal ion adsorption, 50 mL of the metal ion solution of known concentration at the suitable pH with different weights of adsorbent material (0.25 - 2.5 g) at room temperature and shaken at optimum time. The system was centrifuged and the metal ion concentration was determined after adsorption process spectrophotometrically. The uptake percentage was calculated using equation (1). A plot of uptake percentage and adsorbent weight was constructed to determine the suitable adsorbent weight for the process.

b) Effect of Initial Concentration of Metal Ion

In order to study the effect of initial metal ion concentration, 50 mL of the metal ion solution with different concentrations at the suitable were shaken with buffer at room temperature for one hour. The system was centrifuged and the concentration of the metal ion was determined spectrophotometrically. The uptake percentage can be calculated from equation (1). A plot of uptake percentage and initial metal ion concentration was constructed to determine the suitable concentration for the adsorption process.

c) Effect of Shaking Time

In order to study the effect of shaking time and obtain time of equilibrium for each metal ion adsorption, a solution of 50 mL containing 25 mg/L of the metal ion at the suitable pH was shaken with optimal adsorbent weight at room temperature for several periods of time. The system was centrifuged. The concentration of the metal ion was determined after words spectrophotometrically and the uptake percentage was calculated from equation (1). A plot of uptake percentage vs. shaking time was constructed to determine the suitable shaking time for the adsorption process.



d) Effect of initial pH

To establish the optimum pH for metal ions adsorption, 50 mL of metal ions solutions containing 25 mg/L of metal ion was shaken with appropriate weight of adsorbent material for 30 min at room temperature at different pH values (3 - 9). The system was centrifuged to separate the liquid phase from the solid one and the concentration of the metal ion was measured after the adsorption process. A plot of uptake percentage and pH was constructed to determine the suitable pH for the adsorption process.

e) Effect of temperature

In order to study the effect of the temperature on the metal ion adsorption, 50 mL containing the suitable concentration of the metal ion at the pH was shaken with weight of adsorbent material at several temperatures for the optimal period of time. The system was centrifuged to separate the liquid phase from the solid one. The concentration of metal ion was determined after adsorption process as mentioned previously. The uptake percentage can be calculated from equation (2). A plot of uptake percentage and temperature was constructed to determine the suitable temperature for the adsorption process.

Sorption Isotherm

Sorption isotherm describes how adsorbate interacts with adsorbents and how equilibrium is established between adsorbed metal ions on the adsorbent and the residual metal ions in the solution during the surface adsorption process. Equilibrium isotherms were measured to determine the capacity of the adsorbent for metal ions. The most common types of models describing the sorption process are Langmuir and Freundlich models. Total sorption capacity of the adsorbent material was determined by shaking 50 mL containing different concentration of metal ion with adsorbent material at the optimum conditions for each metal ion to ensure complete equilibrium. The mixture was centrifuged and the concentration of metal ion was determined as mentioned previously.

Thermodynamic Parameters

The amount of metal ion uptake q_t at time t, was calculated from the mass balance equation (3). The thermodynamic parameters obtained for the adsorption process were calculated using the equations (3).

$$\ln K_d = \Delta S^{o}/R - \Delta H^{o}/RT \tag{3}$$

Where ΔS^{o} is standard entropy (J/mol.K), ΔH^{o} is the standard enthalpy (kJ/mol), T the absolute temperature (K), and R is the universal gas constant (8.314 J/mol.K) equation (4) was used to calculate K_d as follows:

$$K_d = M_{ads} \, V/M_{sol} W \tag{4}$$

Where K_D is the distribution coefficient (mg/g) M_{ads} and M_{sol} are the amount of metal ion on adsorbent and in solution (mg) respectively, V is the volume of the solution (mL), and W is the weight of the adsorbent (g), the experiments were carried out at 298, 308, 318 and 328 K using concentration 25 mg/L. The standard Gibbs free energy ΔG^o values (kJ/mol) were calculated from equation (5):

$$\Delta G^o = \Delta H^o - T \Delta S^o \tag{5}$$

Where the values of ΔH^o and ΔS^o were calculated from the slope and intercept of linear regression of ln K_D versus 1/T.

Sorption Kinetics

To examine the controlling mechanism for the adsorption process, several kinetic models were used to test the experimental data. A simple kinetic analysis of adsorption data was the *Lagergren pseudo first- order* equation in the following form:

$$\frac{dqt}{q} t = k_1 \left(qe - qt \right) \tag{6}$$

Where k_1 is the rate constant of first-order adsorption and q_e denotes the amount of adsorption at equilibrium. After definite integration by applying the initial conditions $q_t = 0$ at t = 0 and $q_t = q_t$ at t = t, equation (7) becomes:

$$\log (q_e - q_t) = \log(q_e) - k_1 t/2.303 \tag{7}$$

The slopes of the plots of log $(q_e - q_t)$ versus t were used to determine the first order rate constant k_1 in order to determine the activation energy of the adsorption process using the Arrhenius equation.

The pseudo second-order equation based may be expressed as the following form:

$$dq_t/d_t = k_2(q_e - q_t)^2$$
(8)

Where k_2 is the rate constant of second-order adsorption. Integrating the equation and applying it to the initial conditions, the equation become:

$$\frac{1}{(q_e - q_t)} = \frac{1}{q_e} + \frac{k_2 t}{k_2 t}$$
Equation (7) can be rearranged to (9)

$$t/q_t = 1/k_2 q_e^2 + t/q_e \tag{10}$$

Similarly, the slopes of the plots of t/q_t versus t were used to determine the second order rate constant k_2 that was used to determine the activation energy of adsorption process using Arrhenius equation.

Equipment used

- Jenway uv-vis 3310 spectrophotmeter

- Jenway 3310 pH-meter: All pH values were determined by pH meter of type connected to a combined single electrode. The pH was calibrated using standard solutions of pH 4, 7 and 10. deviation in the readings of standard buffer with the pH meter was found to be 0.1 at the ambient laboratory conditions.

- Variable mechanical shaker model AHAAM, Japan was used to. The shaking rate was kept constant for the adsorption process of nickel and iron solution during all experiments.

- IEC clinical centrifuge, USA, for the separation of the sample solution containing the remaining metal ions from the solid phase after adsorption. All sample solutions were centrifuged at 4000 rpm for 5 min to achieve complete separation of adsorbent material from the solution.

III. RESULTS AND DISCUSSION

1. Factors affecting the adsorption process

a. Effect of CB weight

From Figure 5, it was observed that Ni (II) and Fe (II) removal percent increased as the weight of CB increased to achieve a maximum value at 1.5 g CB for Ni and Fe.

b. Effect of initial metal ion concentration

Results (Figure 6) revealed that the removal % decreased with the increase in initial concentration of the metal ion. The optimum concentration for adsorption experiments was considered to be 25 ppm.





Fig. 5. The relationship between weight of adsorbent and uptake percentage of Ni^{2+} and Fe^{2+} sorption onto CB. (pH 7 for Ni (II) and 2 for Fe (II), t = 120 min, T = 25°C, volume of solution 50 mL, and Co = 100 mg/L).



Fig. 6. The relationship between the concentration of metal ions and removal percentage of Ni^{2+} and Fe^{2+} sorption onto CB. (pH 7 for Ni(II) and 2 for Fe(II), t = 120 min, T = 25°C, volume of solution 50 mL, and wt = 30 g/L).

c. Effect of agitation

The effect of agitation time upon the adsorption of Ni (II) and Fe (II) using fixed CB weight was studied. The results are shown in Figure 7 which indicate that the maximum adsorbed amount of metal ions reached a maximum for Ni (II) and Fe (II) at 120 min and 90 min, respectively, and remained unchanged due to reaching a point of saturation.



Fig. 7. The relationship between the agitation time and uptake percentage of Ni^{2+} and Fe^{2+} ions sorption onto CB. (pH 7 for Ni(II) and 2 for Fe(II), Co = 25 ppm, T = 25°C,volume of solution 50 mL, and wt = 30 g/L).

d. Effect of initial pH

The results in (Figure 8) indicate that the removal of Ni ion slightly increased as the pH increased to 6 then it decreased to a minimum value at pH 9 while the removal of iron ion did not significantly change as pH increased



Fig. 8. The relationship between pH of solution and uptake percentage of Ni^{2+} and Fe^{2+} ions sorption onto CB. (Co = 25 ppm, T = 25°C, volume of solution 50 mL, and wt = 30 g/L).

e. Effect of temperature

Figure 9 illustrates that the adsorption of metal ions increased with the increase temperature. This indicates that the process of removal was endothermic for both metals and that the rise in temperature favored the adsorbate transport within the pores of the adsorbent. This increase in sorption may be diffusion controlled (Bansal et al., 2009).



Fig. 9. The relationship between the temperature and uptake percentage of Ni^{2+} and Fe^{2+} ions sorption onto CB. (pH 7 for Ni(II) and 2 for Fe(II), Co = 25 ppm, volume of solution 50 mL, and wt = 30 g/L).

2. Sorption Isotherm

Freundlich and Langmuir equations were used for describing adsorption process.

2.1. Freundlich Isotherm

The Freundlich constants relates to the adsorption capacity and the adsorption intensity of adsorbents, respectively. A plot of log q_e versus log C_e resulted in a straight line with the slope of 1/n and intercept of log k as shown in Figure 10. The values of 1/n and log k are presented in Table 3.





Fig. 10. Freundlich sorption isotherms of Ni²⁺ and Fe²⁺ions sorption by CB.

2.2. Langmuir Isotherm

A plot of C_e/q_e versus C_e resulted in a straight line with a slope of $(1/Q_e)$ and intercept of $(1/b \ Q_o)$ as shown in Fig. 11. The value of slope and intercept of the plot are present in Table 3.



Fig. 11. Langmuir sorption isotherm of Ni2+ and Fe2+ions sorption by CB

TABLE 3. Isotherms constants an	nd values of correlation factor (R^2) fo	r
sorption of Ni 2	²⁺ and Fe ²⁺ onto CB.	

Element	Freu	ndlich isot	herm	Langmuir isotherm				
	1/n	Log K	\mathbb{R}^2	Q0(mg/g)	b	\mathbb{R}^2	RL	
Nickel	0.6041	0.8756	0.9873	3.469	0.0879	0.9802	0.3127	
Iron	0.3245	0.1423	0.9867	3.412	0.0162	0.9975	0.7730	



Fig. 12. First order adsorption kinetics of Ni²⁺ adsorption onto CB.

The relative coefficients of these models were calculated using linear-least square fit equation. From the obtained data, it was observed that adsorption of nickel followed Langmuir isotherm and iron followed Freundlich isotherm.

3. Sorption Kinetics

The mechanism of sorption process was studied by the relationship between the quantity of metal ions sorped and time of adsorption. The kinetics of the sorption was studied at different temperatures and the results are presented in Figures 12,13,14 and 15 and in Table 4.







Fig. 14. Second order kinetics of Ni²⁺ adsorption onto CB.



Fig. 15. The second order kinetics of Fe²⁺adsorption onto CB.

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Fig. 16. Arrhenius plot for first order kinetics of $Ni^{2\scriptscriptstyle+}$ and $Fe^{2\scriptscriptstyle+}$ sorption onto CB

TABLE 4. Kinetic parameters calculated for the sorption of Ni and iron ions onto CB at different temperatures (K), where R2 is the correlation coefficient

Adsorbent	Temp (K)	Pseudo-first-order		Pseudo-second-order			
		$\begin{array}{c c} K_1(\min^{-1})x \\ 10^{-2} \end{array} \qquad R^2$		K ₂ (g mg ⁻¹ min ⁻ 1) x10 ⁻¹	\mathbb{R}^2		
	298	3.0	0.8743	2.1	0.9914		
Niekol	308	3.2	0.9702	5.7	0.9931		
INICKEI	318	1.2	0.9972	1.1	0.9932		
	328	1.5	0.9888	1.4	0.9971		
	298	5.9	0.9921	6.3	0.9265		
Iron	308	6.6	0.9950	7.9	0.9505		
non	318	7.4	0.9980	9.9	0.9765		
	328	8.2	0.9954	1.3	0.9907		



Fig. 17. Arrhenius plot for second order kinetics of Ni^{2+} and Fe^{2+} sorption onto CB.

It appears from the results that adsorption of nickel onto CB followed a second order kinetics and that of iron followed first order kinetics. The values of activation energy were 51.92 KJ/mol for nickel and 8.936 KJ/mol for iron (Figure 16 and 17).

4. Thermodynamic Parameters

The thermodynamic parameters were obtained from (Figure 18), and the values are represented in Table 5.



Fig. 18. ln K_D with 1/T for Ni²⁺ and Fe²⁺ sorption onto CB.

TABLE 5. The thermodynamic value for the adsorption of nickel and iron on

uie CB										
Element ΔH^0		ΔS^0	ΔG^0 (kJ/mol)							
Liement	(kJ/mol)	(J/mol.K)	298 K	308 K	318 K	328 K				
Nickel	22.775	0.108	-9.409	-10.489	-11.569	-12.649				
Iron	12.260	0.0611	-5.948	-6.559	-7.170	-7.781				

IV. CONCLUSION

Results and discussion show the following:

- Sorption parameters such as pH, shaking time, initial concentration of metal ions, adsorbent weight and temperature affecting the adsorption process.

- Sorption kinetics of Nickel was second order mechanism with CB with activation energy of 51.92 KJ/mol.

- Sorption kinetics of Iron as first order mechanism with CB with activation energy 8.936 KJ/mol

- Sorption Isotherm of Nickel was found to follow Langmuir isotherm model with Coke breeze adsorbents this was clear from their correlation factor R^2 .

Thermodynamic parameters

The results indicates that the value of enthalpy ΔH_o was found to be a positive for coke breeze that is mean that the endothermic nature of the process and the negative values of Gibb's free energy ΔG^o indicates the feasibility and spontaneous of the process.

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