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Synthesis and characterization of organoclays based on bentonite and TOAB, HDAB surfactants: application for glyphosate removal

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Abstract

Inappropriate use by agriculture and industry has led to contamination by glyphosate (GPH), which is toxic to human health, life and the environment. GPH is eliminated by the adsorption process of organoclay composites. To synthesize organoclays, the cationic surfactants hexadecyltrimethylammonium bromide (HDAB) and trimethyloctadecylammonium bromide (TOAB) were combined with bentonite clay materials. The structural properties of the materials were studied by FTIR, XRD, SEM-EDX, BET surface and pHpzc. The batch process was carried out with the following parameters: pH, contact time, temperature, initial concentration and comparative doses of bentonite and the two composite materials. However, the X-ray diffraction patterns of the surfactant-modified TOAB- Bent and HDBA-Bent composites revealed an increase in basal spacing. The sorption capacity of GPH in TOAB-Bent and HDBA-Bent increased with the loading level of TOAB or HDAB, while the sorption efficiency of GPH was produced by the hydrophobic phases formed by surfactants, with hydrophobic partitioning being the critical process. Therefore, the addition of bentonite and surfactants can significantly improve glyphosate removal from aqueous solutions.

Keywords: *Bentonite, surfactants, environment, glyphosate, wastewater.*

Introduction

Surface and The world's population relies primarily on surface and groundwater for drinking water. Industria-lization and urbanization have had a significant environmental impact, making water pollution one of the most pressing environmental issues of the twenty-first century. This is due to the introduction of various contaminating pollutants into water sources. With the global increase in herbicide use, numerous scientific studies have been published that describe the negative effects of herbicides and fertilizers. Herbicides are a type of emerging pollutant that is widely used in agriculture due to their efficacy. Glyphosate (Benbrook et al., 2016) is one of the world's most widely used herbicides, discovered by a group of scientists led by Dr. John Franz at Monsanto and marketed in the 1970s under the trade name "Roundup" (Duke et al., 2008).. It is a component of many herbicides used to eliminate weeds (20). Since its introduction to the world market in 1974, its annual global use has ranged between 600 and 750,000 tonnes, with over 1.6 billion kilograms consumed in the United States alone (Maggi et al., 2020). This herbicide is well known for its ability to effectively treat soils. However, it can cause serious environmental problems (molecular contamination of soil, water and air), posing a significant risk to plants, animals and human health. The amount of glyphosate used has increased by approximately 90 times, and studies claim that traces of glyphosate can be found in

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water, soil, food products, and air, as well as breast milk, human serum, and urine (Duke et al., 2008 ; Mañaset al., 2009). In addition, an intriguing study by the International Agency for Research on Cancer (IARC) classified glyphosate as "probably carcinogenic to humans" (Tarone et al., 2018). Because of glyphosate's toxic properties, its removal from the environment has become critical. Several methods have been used to remove glyphosate, including ultrafiltration, coagulation- flocculation, POAs, and sorption. Adsorption with adsorbent materials is one of the most cost-effective methods for removing these pollutants. These materials include activated carbon, zeolites, molecular sieves, and clays. This study aims to investigate clay, in particular bentonite, which is widely used as an adsorbent. It is a widely available and inexpensive natural material composed mainly of montmorillonite, an aluminosilicate mineral. Bentonite stands out among clay minerals for several reasons: it is composed of a variable number of elementary sheets, has a high specific surface area, a high cation exchange capacity (CEC), flexibility in terms of physical and chemical properties, water adsorption capacity, plasticity and swelling. In recent years, the use of bentonite has become an important aspect of research, with an irreplaceable role in water treatment. To achieve the objective of this work, we (i) created two clay-based composites using the surfactants HDAB and TOAB and (ii) studied the interaction of glyphosate with the synthesized materials. The parameters that affect glyphosate adsorption (adsorbent mass, pH, time, and pollutant concentration) were investigated. In addition, isothermal, kinetic, and thermodynamic studies were conducted to investigate the adsorption process.

Materials and methods

Materials

The clay studied originates from Maghnia in the Tlemcen region of Algeria and is referred to as bentonite. Bentonite is a finely ground, untreated substance with a creamy hue. Bentonite was chosen due to its prevalence in Algeria and its propensity to bind with metals and polluting molecules.

Composite preparation

The following procedure was used to exchange Raw-Bent for HDTMA+ and TOAB+: after dissolving 4.5 g of HDTM Br and TOAB Br, which accounted for 100% of the bentonite's CEC value, in 1 L of distilled water, 15 g of bentonite was gradually added and agitated (500 rpm) in various containers for a full day. The solutions were then filtered, recovered and cleaned with distilled water to remove the bromide anions. AgNO3 resulted in a negative test. The solid was subsequently sieved, ground, and dried. Tiller 410, we used a commercial glyphosate formulation (isopropylamine (IPA) salt, 48%) produced by Astrachem (Saudi Arabia).

Characterization methods

Mineralogy, functional groups, morphology, and surface charge were studied using XRD, FTIR, SEM, and BET pHpzc, respectively. The XRD data for the adsorbents were collected using a D/MAX 2200 diffractometer with Cu Kα radiation (Rigaku, Japan). FTIR spectra were obtained using a Perkin Elmer ATR spectrophotometer. Surface measurements were performed using an ASAP 2020 surface and porosity analyzer (Micromeritics, USA). The morphological properties of clay and organoclays were determined using a scanning electron microscope.

Adsorption study

To obtain adsorption isotherms, 50 ml volumes of glyphosate solutions ranging from 10 to 150 mg/L were mixed with masses of HDBA-Bent, TOAB-Bent, and Raw-Bent in 100 ml Erlenmeyer flasks. The pH of the samples was adjusted with HCl and NaOH solutions (0.1N). Adsorbate/adsorbent mixtures were stirred for 25 minutes for HDBA-Bent and TOAB-Bent and 30 minutes for Raw-Bent before being centrifuged for 30 minutes at 6000 rpm. The concentration of residual glyphosate was determined using a UV-vis spectrophotometer (Perkin Elmer) at 222 nm. The effects of glyphosate adsorption on the three materials were investigated to describe the adsorption mechanism, and the amount of glyphosate adsorbed was calculated using the following equation [1]:

$$qe = \frac{C_0 - C_{eq}}{m}.V \qquad [1]$$

where, q_e is the quantity adsorbed at equilibrium in (mg/ g), C_0 (mg/ L) is the initial glyphosate concentration, C_e (mg/ L) is the residual glyphosate concentration, V (ml) is the volume of glyphosate solution, m is the quantity of adsorbent in solution in (g).

Modeling adsorption isotherms and kinetics

The kinetic study is important for determining the speed of the adsorption process and providing additional information about the reaction mechanism. Two types of kinetic models were used in this study: pseudo-first-order model (Vaccari et al., 1999):

$$(q_e - q\ln_t) = \ln q_e - K_1 \,.\, t \qquad [2]$$

pseudo-second-order (Aurich et al., 2017) :

$$\frac{t}{qt} = \frac{1}{K2.\,q2} \cdot \frac{t}{qe} \qquad [3]$$

where K1 (min -1) and K2 (g/ mg.min) are the pseudo-first-order and pseudo-second-order rate constants respectively.

Three models of adsorption isotherms, Langmuir (Langmuir., 1917) [Eq. 4], Freundlich (hai Yang et al., 1998) [Eq. 5] and Temkin [Eq. 6] (Foo et al., 2009) were applied:

$$\frac{1}{q_e} = \frac{1}{C_e}\frac{1}{q_m K_L} + \frac{1}{q_m} \qquad [4]$$

$$\text{In } q = \text{In } K + .\text{In } C \qquad [5]$$

$$q_e = b.\ \text{In } K_T + b.\ \text{In } C_e \qquad [6]$$

where, Ce (mg/g) is the equilibrium concentration of the glyphosate solution, KL (L/mg) and KF ((mg/g)/(mg /L) n) are the Langmuir and Freundlich isotherm constants respectively; n is the Freundlich constant which correlates with adsorption capacity and adsorption intensity, KT (L/g) is the equilibrium binding constant, and the constant b (J /mol) is related to the heat of adsorption.

Thermodynamic study

The thermodynamic study of adsorption improves our understanding of the nature and operation of the adsorption mechanism. Adsorption relies heavily on thermodynamic parameters like free energy change (ΔG°), enthalpy change (ΔH°), and entropy change (ΔS°). Enthalpy variations determine whether an adsorption process is endothermic or exothermic. The Gibbs energy change, ΔG°, is calculated as follows:

$$\ln K = -\frac{\Delta H^{\circ}}{RT} + \frac{\Delta S^{\circ}}{R} \qquad [7]$$

Results and discussion

Sorbent characterization

Fluorescence X-ray spectrometry (FRX). Table 1 shows the elemental composition of Raw-Bent as determined by Fleurance-X. The XRF analysis of the raw clay revealed that silica and alumina were the most abundant elements in the samples examined, which was confirmed by the XRD analysis (Fig. 2). The results confirm that our bentonite is a calcic clay (%Ca >% Na).

Table 1. *Elemental composition of Raw-Bent as determined by X-ray fluorescence. M = mass percentage*

Element	SiO_2	AL_2O_3	$F_{e2}O_3$	CaO	MgO	SO_3	K_2O	Na_2O	P_2O_5	TiO_2	Mn_2O_3	**Cl**
M (%)	63	17.06	4.86	2.53	3.7	0.148	1.13	1.8	0.12	0.66	0.04	0.129

Infrared spectroscopy (FTIR). Figure 1 depicts the FTIR spectra of Raw-Bent, TOAB-Bent, and HDBA-Bent in the 4000–400 cm-¹ range. The raw clay spectrum reveals a peak at 3650 cm-¹, indicating OH bonds. This peak is caused by hydroxyl elongation vibration bands in the Al-Al-OH species found in the phyllosilicate's octahedral layer. A low-intensity band at 1650 cm-¹ indicates the presence of inter-lamellar water, while peaks at 1010, 900, and 780 cm-¹ indicate Si-O stretching vibrations and bending vibrations of Si-O-Al and Si- O-S in the tetrahedral layer, respectively. The intense peaks at 1650 and 3650 cm-¹ in all three materials were attributed to the deformation of water-OH bonds. Only TOAB-Bent and HDBA-Bent exhibited bands at 2920 and 2850 cm-¹. They can be attributed to symmetrical and asymmetrical elongation vibrations of the methylene groups (CH_2), which are slightly more intense in TOAB than HDTM. Methylene group deformation vibrations can be seen at 1450 cm-¹.

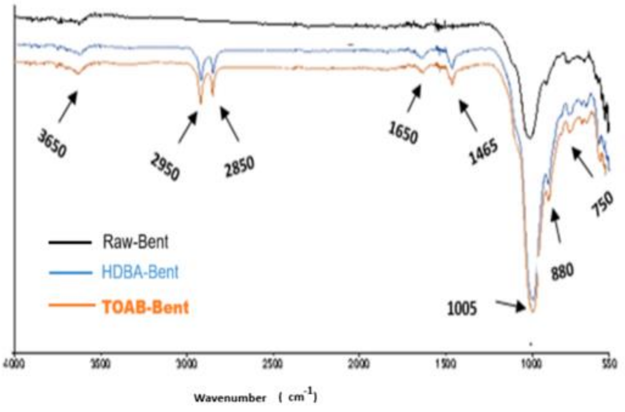

Figure 1. *FTIR spectra of Raw-Bent, TOAB-Bent, and HDBA-Bent composites.*

X-ray diffraction (XRD). Figure 2 shows the XRD diffractograms of Raw-Bent, HDBA-Bentonite, and TOAB-Bentonite. It can be noted that the peak of bentonite at 2θ = 6° shifted to a smaller angle. The basal spaces of HDBA- Bentonite (2θ = 4.9°) and TOAB-Bentonite (2θ = 4.8°) were calculated at 14.72.17 and 17.70 Å, respectively. These results indicate that the bentonite layers were expanded due to the insertion of the surfactants into the bentonite's interlayer space, thus increasing the basal space d001. For the two surfactants HDAB and TOAB, we note that TOAB has a slightly higher interlayer distance (d001) than HDAB, as TOAB has a larger alkyl chain than HDAB (CH2 plus) in the order d (TOAB)> dHDAB >drawbentonite.

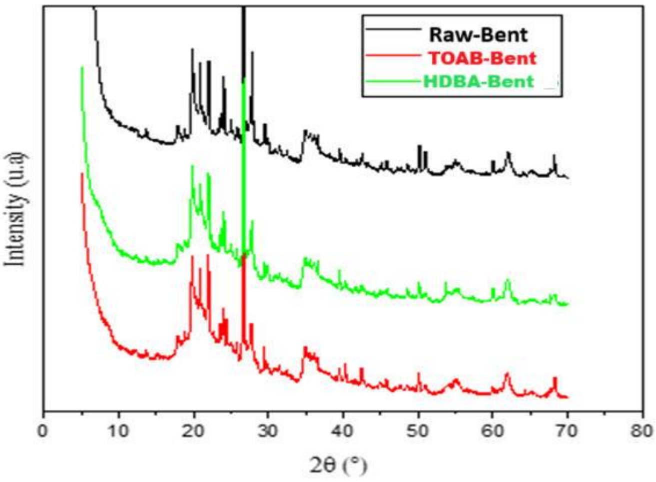

Figure 2. *XRD diffractogram of Raw-Bent and organobentonites*

Specific surface measurement. According to the results of the BET surface area measurements (Table 2), Raw-Bent, HDBA-Bent, and TOAB-Bent decrease in the following order: Raw-Bent (25.67) > TOAB-Bent (11.14) > HDBA-Bent (4.28), indicating that organic molecules have covered its surface and penetrated the Raw-Bent layers, resulting in a reduction in specific surface area. This result also confirms the decrease in pore volume (0.040049, 0.0085, 0.01559 cm3g-1 for Raw-Bent, HDBA-Bent, and TOAB-Bent, respectively), as well as the increase in pore diameters (233.67 nm, 1398.98 nm, 538.5531 nm for Raw-Bent, HDBA-Bent and TOAB-Bent, respectively).

Table 2. *BET specific surface area (SBET), pore volume (Vp), and mean pore diameter for Raw-Bent, HDBA-Bent and TOAB-Bent.*

Materials	SBET (m^2/g)	VP (cm^3/g)	Average diameter Pore size (nm)
Raw-Bent	25.6765	0.040049	10.8871
HDBA-Bent	4.2888	0.008552	16.0057
TOA-bent	11.1410	0.015598	11.4655

Zero charge point (pHpzc). The pH(pzc) values of Raw-Bent, HDBA-Bent, and TOAB-Bent were determined to be 7.9, 8.0, and 8.1, respectively. An increase in pH(pzc) is observed following the incorporation of surfactants. This modification enhances the pH range favorable for the adsorption of anionic species, indicating that surfactant-modified bentonite exhibits a higher adsorption capacity for anionic molecules compared to Raw-Bent.

Adsorption study

Parameter optimization

Contact time effect. The effect of contact time on glyphosate adsorption by Raw-Bent, HDBA-Bent, and TOAB-Bent is shown in Fig. 3. The contact time ranged from 5 to 60 minutes at room temperature, with an initial glyphosate concentration of 100 mgL-1 and an adsorbent mass of 0.05 g. The adsorption of glyphosate by the three adsorbents occurs in two stages, with the first stage being rapid in the first 10 minutes for Raw-Bent, 25 minutes for TOAB-Bent, and 30 minutes for HDBA-Bent, followed by a second slow stage until equilibrium and site saturation are achieved. In the case of Raw-Bent, the percentage adsorption efficiency was increased to 10 mg/g in 10 minutes, which was lower than TOAB-Bent and then HDBA-Bent, where the percentage adsorption efficiency reached 49 mg/g and 42 mg/g at 25 and 30 minutes, respectively. The time difference between the three materials is most likely due to differences in free site

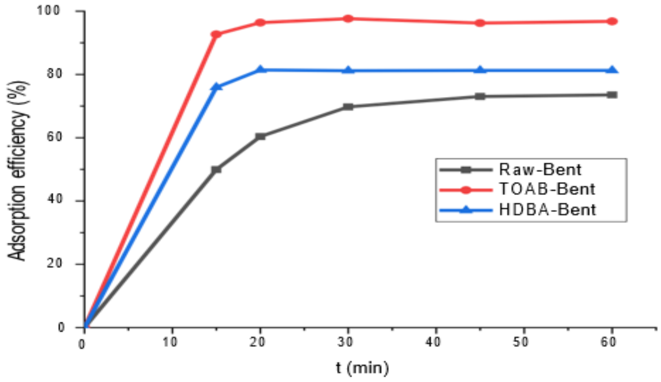

Figure 3. *Contact time effect of glyphosate adsorption on Raw-Bent, HDBA-Bent and TOAB-Bent.*

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Effect of adsorbent mass. Figure 4 shows the effect of the mass of Raw-Bent, HDBA-Bent and TOAB-Bent on glyphosate adsorption efficiency. It was found that the adsorption efficiency of glyphosate increases with increasing mass of the three adsorbents: Raw-Bent, HDBA-bent and TOAB-Bent. This result is due to the increase in available adsorption sites up to 0.05g, as our materials reach adsorption equilibrium and saturation of the active sites with adsorption percentages of 65%, 80% and 90% for Raw-Bent, HDTMA-bent and TOAB-Bent, respectively.

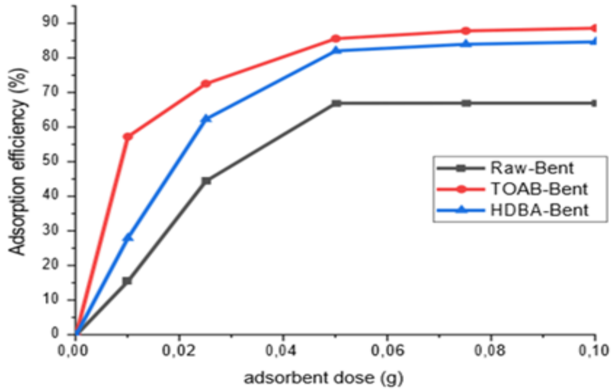

Figure 4. *Effect of mass on glyphosate adsorption*

Effect of pH solution. To investigate the effect of pH on glyphosate adsorption, 50 ml of glyphosate solution (100 mg L-1) was stirred at 298 K for 25 and 30 minutes, respectively, for Raw-Bent, HDBA-Bent, and TOAB-Bent, with a mass of 0.05g for each material. The effect of pH on glyphosate adsorption efficiency was investigated across the pH range of 2 to 12. Figure 5 shows that for all three adsorbents, the pH=2-4 range yielded the highest percentage of adsorption efficiency. The pHpzc test results show that the pHpzc determined is equal to 8 for Raw-Bent, 8.1 for HDBA-Bent, and 8.5 for TOAB-Bent (Fig. 3), indicating that the surface charge is positive below the pHpzc of the three materials, but negative above the pHpzc. This finding is explained by the electrostatic attraction between the free glyphosate doublets and the positively charged surfaces of all three materials (solution pH < pH(pzc)). Therefore, lowering the pH of the solution increases the number of positively charged sites (pH below pHpzc), which are ideal for anionic pollutant removal.

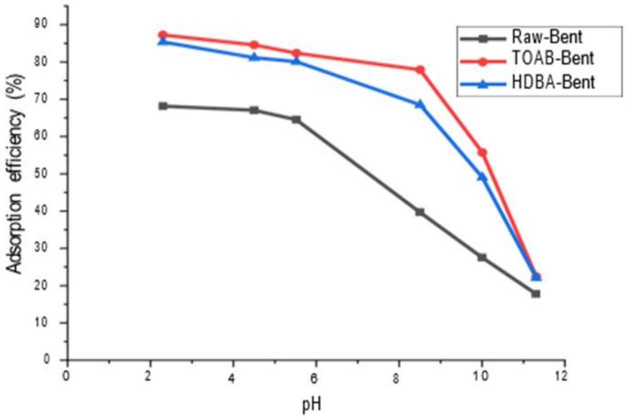

Figure 5. *Effect of solution pH on glyphosate adsorption.*

Kinetic study

The kinetics of glyphosate adsorption in various materials were investigated using models: pseudo-first-order pseudo-second-order. The results are shown in Table 3. For all three materials, the pseudo-second-order model outperformed the pseudo-first-order model in describing adsorption kinetics. It should be noted that the qe values were consistent with the experimental qe values. This finding implies that chemical interactions, possibly electrostatic attraction, influenced the adsorption process.

Table 3
Adsorption kinetic parameters according to the models studied.

Kinetic models	Adsorbent	Values		
		Qe (mg g^{-1})	K1 (min^{-1})	R2
Pseudo-first-order	Raw-Bent	6.0799	0.309	0.697
	HDBA-Bent	46.021	0.0936	0.7539
	TOAB-Bent	3.68	0.0599	0.0414
		Qe (mg g^{-1})	K2 (g/mg min^{-1})	R2
Pseudo-second- order	Raw-Bent	12.06	0.266	0.99
	HDBA-Bent	49.26	0.0031	0.973
	TOAB-Bent	54.34	0.00248	0.974

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Isotherm study

Adsorption isotherms were simulated using the Langmuir, Freundlich, and Temkin models (Table 4). Considering R2 as the factor that validates the model isotherms and analyzing the parameter results obtained for each model. Figure 6 shows that the linear Langmuir model best fits glyphosate adsorption on Raw-Bent, HDBA-Bent, and TOAB-Bent with R2 compared to the Freundlich and Temkin models for the three materials, indicating monolayer adsorption. The Langmuir isotherm also provides information on the adsorption capacities of three materials, simulating a maximum adsorption capacity of 34, 44.8, and 46 mg/g for Raw-Bent, HDBA-Bent, and TOAB-Bent respectively, implying that TOAB-Bent adsorbs glyphosate better than Raw-Bent and HDBA-Bent. The Langmuir separation factors (RL) for Raw-Bent, HDBA-Bent, and TOAB-Bent are 0.17, 0.12, and 0.15, respectively. The non-linearity index (n) of the Freundlich equation is less than n < 1, indicating that glyphosate adsorption on the three materials studied is favorable under the conditions investigated. In the Temkin isotherm, Raw-Bent, HDBA-Bent, and TOAB-Bent have low BT values (4.93, 10.79, and 10.78 Kj.mol- 1), indicating a weak ionic interaction (B(T)<35 kJ.mol-1) between glyphosate and the adsorbent surface. HDBA-Bent and TOAB-Bent have higher B(T) values than Raw-Bent, indicating a stronger HDAB- bent-glyphosate and TOAB-Bent-glyphosate interaction than bent-glyphosate (Yang et al., 2018 ; Gimsing et al., 2006 ; Besghaier et al., 2022).

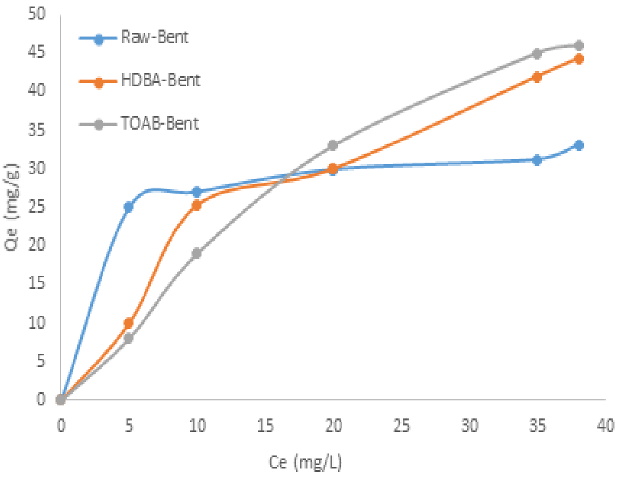

Figure 6. *Adsorption isotherm of the materials studied*

Table 4
Adsorption isotherm constants

Adsorbent	Isoterm Models		
	Langmuir isotherm model		
	Qm (mg /g)	K_L (dm^3/mg)	R^2
Raw-Bent	34	0.36	0.91
HDBA-Bent	44.8	0.013	0.99
TOAB-Bent	46	0.011	0.9961
	Freundlich isotherm model		
	K_F (mg/g) (L/mg)1/n	n	R^2
Raw-Bent	18.01	0.17	0.98
HDBA-Bent	1.84	0.58	0.98
TOAB-Bent	1.44	0.623	0.995
	Temkin		
	B_T	K_T (l/mol)	R^2
Raw-Bent	4.93	22.85	0.974
HDBA-Bent	10.79	0.11	0.98
TOAB-Bent	10.78	0.0949	0.994

Thermodynamic study

To better understand the thermodynamics of glyphosate adsorption on our materials, we performed retention experiments for this herbicide, varying the temperature of the contaminated solutions from 27°C to 62°C. The results show that ΔG° values are negative at all temperatures, indicating that glyphosate adsorption occurs spontaneously on the three materials. In contrast, the decrease in the absolute value of ΔG° with increasing temperature for raw clay suggests that glyphosate adsorbs better at low temperatures for TOAB and HDAB. The negative value of ΔH° confirms that the adsorption reaction is Exothermic for all three materials, probably due to physical

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adsorption, as the ΔH°<35 kj/mol for all three materials. It also confirms that the system is favored as temperature decreases. The low ΔH° values indicate that glyphosate adsorption is physical. The positive value of ΔS° in raw clay indicates glyphosate's affinity for our material and increased randomness at the solid-solution interface during adsorption (Saha et al.,2011 ; Jiang et al., 2016 ; Ueda et al., 2016).

Conclusions

This study investigated the combination of natural bentonite with the surfactants TOAB and HDAB to create composites referred to as TOAB-Bent and HDBA-Bent. XRD analysis indicates that the surfactants were absorbed into the interstitial spaces of bentonite, thereby expanding the interlamellar zones and, as a result, increasing the quantity of glyphosate adsorbed. The GPH adsorption process on all synthesized materials was characterized by pseudo-second-order kinetics. Thermodynamic data indicate that the adsorption process is spontaneously endothermic and exhibits greater efficiency at elevated temperatures. The study's findings indicate that organic-montmorillonite is superior for treating glyphosate-contaminated water and is more environmentally sustainable for pesticide adsorption compared to GPH.

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