Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2023.29295

285 (2023) 121–128
February

Sorption of Bisphenol A from aqueous solutions by acid activated bentonite clay

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Received 21 September 2022; Accepted 2 January 2023

ABSTRACT

Batch measurements were conducted to assess the adsorption performance of raw and acid-activated bentonite with various operating conditions, including pH, initial Bisphenol A (BPA) concentration, contact time, and temperature. Fourier-transform infrared spectroscopy, X-ray diffraction, and scanning electron microscopy were used to study the structure and morphology of bentonite. On a laboratory scale, isotherm experiments were carried out, and the results were compared to the Langmuir and Freundlich isotherm models. The Langmuir model fits the equilibrium data well. The optimum conditions for the removal of BPA within the experiment range of variables studies were 1 g/L of initial BPA concentration, pH value of 3, 0.25 g/100 mL of adsorbent dose and 180 min of contact time. The maximum removal efficiency was 51.8% under these conditions. The experimental results indicate that the adsorption was degraded by increasing the pH. The decrease of adsorbent characteristics with increasing temperature from 25°C to 65°C was observed in this work, indicating exothermic and chemical adsorption in nature.

Keywords: Adsorption; Bentonite; Bisphenol A; Kinetics; Equilibrium isotherms

1. Introduction

Bisphenol A (BPA), 2,2-bis(4-hydroxyphenyl) propane is a chemical compound that forms part of many everyday objects, such as electrical equipment, auto parts, glazing, roofing, medical equipment, sunglasses, baby bottles, beverage cans, cans, including boxes of milk powder for babies [1]. A monomer widely utilized in the industrial manufacturing of polycarbonate plastics and epoxy resins via polymerization. It is also utilized in plasticizers as an antioxidant and as a polymerization inhibitor in polyvinylchloride [2,3].

Low amounts of BPA are enough to have a harmful impact on humans [4,5]. Other studies show a link between exposure to environmental contaminants, including BPA, and metabolic disorders. BPA has recently been shown to be able to escape from these materials and contaminate the environment since water pollution has become one of the main environmental problems due to the release of toxic and dangerous chemicals from various industrial activities [6,7]. In recent decades, various techniques have been used to eliminate BPA. However, there is an urgent need to develop a rapid and effective treatment to eliminate organic micropollutants and in particular phenolic compounds. Adsorption is one of the most straightforward and extensively utilized strategies for removing organic micropollutants from water [8–10]. BPA was efficiently removed from aqueous solution using the adsorption potential of chosen activated

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carbons [11,12]. Much research has been carried out by other researchers on the chemical treatment of different types of clays in order to improve the adsorption capacity. Several parameters influenced adsorption behavior, including operating temperature, adsorption time, pH, and bentonite pretreatment [13–17].

Bentonites are clays rich in smectite regardless of their mode of origin [18], essentially made up of montmorillonite, $(SiO_2)_m(Al_2O_3)n(H_2O)_p$, and are valued for features such as crystal structure and size, hydration and swelling, cation exchange capacity, and susceptibility to react with organic molecules [19]. The swelling and permeability properties of bentonite and their performance in water treatment depend essentially on the content of montmorillonite present in the product and also on the relative concentration of bentonite in sodium and calcium [20], also used as a source of silica and alumina and traditionally used as a raw material by potters [4].

Cationic surfactant molecules and ion exchange can modify the constantly negatively charged clay surface. These bentonites are efficient adsorbents for the removal of phenolic compounds and the appropriate results for removing contaminants have been mentioned [21–23]. The north-eastern region of Morocco is distinguished by the presence of large bentonite deposits.

The objectives of this paper are to: (i) investigate the (bentonite) ability to remove the BPA from aqueous solution; (ii) investigate the impact of temperature in a range from 25°C to 65°C on BPA sorption on bentonite, in order to obtain representative transfer parameters; (iii) determine the impact of solution pH and initial BPA concentration on sorption and contact time; (iv) perform isothermal and kinetic experiments to illustrate the adsorption mechanism.

2. Experimental

2.1. Presentation of bentonite studied

The bentonite employed in this study came from the Trebia deposit in the Nador region (North-East of Morocco). It was washed several times with distilled water, filtered and dried in an oven at 60°C for 24 h, before the experiments. Bisphenol A, phosphoric acid was purchased from Sigma-Aldrich (France).

2.2. Preparation of adsorbent

We prepared a solution of phosphoric acid (34%) with 85% purity, by adding 40 mL of the acid to 60 mL of distilled water (total volume 100 mL). The bentonite was activated by phosphoric acid by adding 20 g of bentonite to 20 mL of water, after which the acid was added to the bentonite dropwise under stirring for 24 h. The adsorbent was then filtered on filter paper. The pH of the filtered adsorbent was then decreased to 6–7 by washing it multiple times with distilled water. The adsorbent was dried in an oven at 60°C for 24 h. The acid-activated bentonite was stored for further use [24].

2.3. Scanning electron microscopy analysis

The scanning electron microscopy (SEM) micrographs of bentonite composites that were taken at 3,000 magnification are shown in Fig. 1. On the one hand, the image shows an almost uniform nanocomposite, on the other hand, it shows that the surface of the bentonite has an heterogeneous structure, which is typical of extremely porous materials, indicating that BPA could be adsorbed on various regions of the biosorbent.

2.4. X-ray diffraction analysis

In our research, we used the "Bruker D8 Advance Twin" diffractometer, which is equipped with a copper anticathode attacked with electrons accelerated to a voltage of 40 kV and emits radiation with a wavelength of $\lambda(K\alpha 1) = 1.5406 \times 10^{-10}$ m. Diffraction patterns are recorded in 2θ in an angular range of 10° to 80°. The samples to be analyzed are first ground in an agate mortar.

Fig. 2 shows the diffractogram of raw and activated bentonite. Raw bentonite is mostly made of montmorillonite and quartz, as illustrated in the diagram. Other from montmorillonite, all components are present as impurities. The intensity of the peak at 22° is increased in the diffractogram of phosphoric acid activated bentonite, which corresponds to quartz. However, the intensity of the montmorillonite peaks is also decreased at 20° as can be seen in Fig. 2. Aluminum leaching occurs during acid activation, resulting in a drop in intensity. External studies by X-ray diffraction, infrared spectroscopy, and thermal analysis show that the raw Nador bentonite is a type of montmorillonite [25].

2.5. Bisphenol A

We used a stock solution of 99% pure BPA ($C_{15}H_{16}O_2$) from Aldrich, this was then diluted to acquire the necessary concentration of source solutions. For Bisphenol A-d4 (BPA-d4) (99%) provided by CDN isotopes (Pointe-Claire, Canada); The structure of BPA and physicochemical characteristics are summarized in Table 1. BPA is a solid at room temperature and white color, its important properties are moderate solubility in water and low volatility [26]. Therefore, BPA would be readily adsorbed on the particles and possibly on surfaces such as membranes. In the pH range of most natural fluids and wastewaters, BPA is uncharged because of its extremely high pKa value [27]. In the treatment of polluted water such as leachate, the parameters influencing the adsorption of an organic pollutant on a solid (such as biomass or biosolids) are: solubility and octanol/water partition coefficient (K_{ow}).

2.6. Wastewater analysis and concentration measurement

We used UV-VIS spectrophotometer to evaluate 10 mL of effluent in a quart cuvette. A BPA calibration curve was first created in order to calculate the BPA concentration after each experiment. A UV-Vis spectrometer was used to measure the absorbance of six different concentrations of pure BPA at a wavelength of 276 nm. Following that, the maximal absorbance at 276 nm was plotted against each BPA concentration. The pollutant concentration was calculated using this plot.

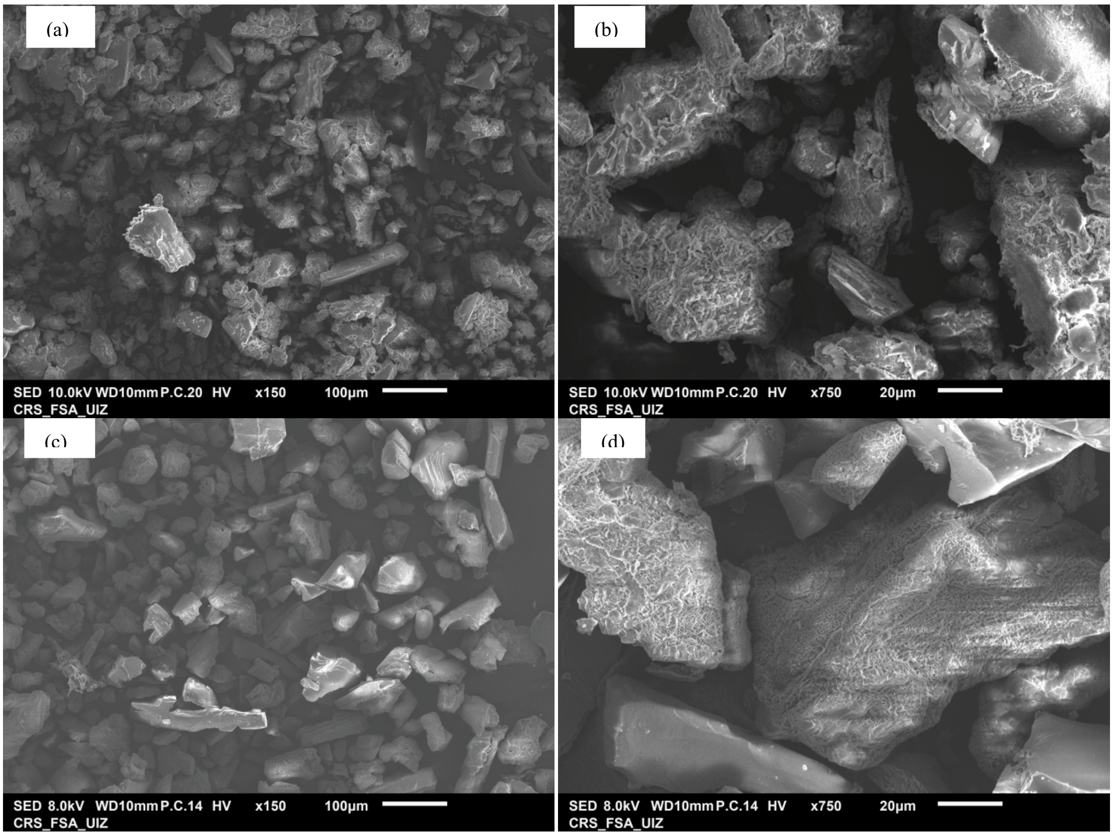


Fig. 1. SEM micrographs of raw bentonite composite (a, b) modified bentonite (c, d).

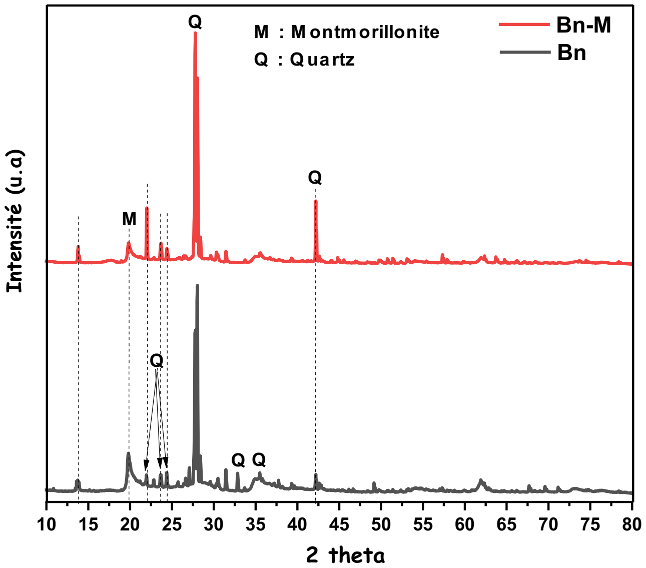
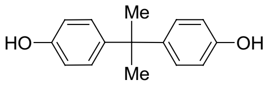


Fig. 2. X-ray diffraction patterns of the raw and modified bentonite.

2.7. Batch adsorption test on bentonites

The adsorption efficacy of raw and acid activated bentonite was tested in batch experiments, the BPA solution was prepared by dissolving a quantity of solid BPA with deionized water (1 g/L). Adsorption experiments were performed by adding a fixed amount of 0.25 g of adsorbent to a series of Erlenmeyer flasks (100 mL) filled with 50 mL of BPA solutions. The Erlenmeyer flasks were then stirred until equilibrium was reached. Samples (10 mL) were taken at specified time intervals and filtered to remove any remaining adsorbent in the solution. The UV spectrophotometer (Rayleigh, UV-1800) was then used to determine the concentrations of the liquid samples at the maximum wavelength of 276 nm for Bisphenol A.

Table 1
Physico-chemical characteristics of Bisphenol A (BPA)

Formula	$C_{15}H_{16}O_2$
Molecular weight (g/mol)	228.29
Solubility g/L (at 20°C)	0.3
pKa	9.59–11.30
$\log K_{ow}$	3.40
Dipole moment (Debye)	0.709 [42], 1.411 [43]
Molecular size (nm)	Molecular width X: 0.383; Molecular width Y: 0.587; Molecular width Z: 1.068 [43]
Structure	

Different parameters were highlighted in order to study their effect on the adsorption capacity, which are: contact time (10–180 min), initial concentration of the BPA solution (100–1,000 mg/L), pH (3–11) and temperature (25°C–65°C).

We carried out batch adsorption studies to investigate the adsorption capability of modified bentonite for BPA. UV-Visible spectrophotometry was used to determine the concentration of BPA. Eqs. (1) and (2) were used to compute the removal efficiency and adsorption capacity of BPA on modified bentonite:

$$\%R = \frac{\left(C_0 - C_e\right)}{C_0} \times 100 \tag{1}$$

$$q_e = \frac{\left(C_0 - C_e\right)V}{W} \tag{2}$$

where R (%) represents removal efficiency of BPA on raw and modified bentonite, q_e is the equilibrium adsorption capacity of raw and modified bentonite for the BPA solution (mg/g), C_i and C_e are the initial and equilibrium concentration in BPA solution (mg/L), V (L) is the volume of the BPA solution and m is the mass of raw and modified bentonite (g).

3. Results and discussion

3.1. Effect of time of adsorption

A preliminary experiment was conducted with natural bentonite to determine the equilibrium time. Samples were taken after 10, 20, 30, 40, 60, 80, 100, 120 and 180 min of contact between the solutions and bentonite. The removal efficiency shown at various contact durations in Fig. 3 demonstrates a two-stage kinetic process: fast adsorption for the first 40 min, followed by a considerably longer period of significantly slower uptake. After 120 min, which was chosen as the experimental contact time for this investigation, the greatest elimination efficiency was observed. The fast adsorption observed is most likely owing to the abundance of active sites on bentonite's surface, and with the progressive occupation of these sites, adsorption has become less efficient [28].

It should be noted that the more the initial BPA concentration the more would be the adsorption capacity of adsorbents. It can be seen that the adsorption capacity (q_e) increased from 12 to 60 mg/g when the initial concentration of BPA is increased from 250 to 1,000 mg/L.

The driving force of the concentration gradient increases as the concentration increases, that is, the molecules diffusion of BPA in solution in the adsorbent surface increases [29]. On the other hand, Fig. 4 shows that the initial concentration has no effect on the contact time, which corresponds to the adsorption equilibrium at the adsorbent/adsorbate interface when the support is saturated with the solute. Because of the huge amount of BPA bound to the sorbent, the sorption process can be deemed quick [30,31].

For the raw bentonite (Bn) and modified bentonite (Bn-M) adsorbents, the contact time is 120 min, which corresponds to a reduction rate of 30% for Bn (adsorption capacity of 60 mg/g) and 40.5% (adsorption capacity of 81 mg/g) for Bn-M in case of initial concentration of 1,000 mg/L (Fig. 4)

3.2. Effect of pH

The effect of pH on BPA removal by raw bentonite and modified bentonite have studied by variation of solution pH (3–11) at constant BPA concentration (1 g/L) and adsorbent dosage (5 g/L) for 120 min. The solution pH was adjusted to 3–11 using HCl or NaOH. The relationship between the solution pH and adsorption capacity (mg/g) of BPA is shown in Fig. 5.

It can be seen that the amount adsorbed decreases with increasing pH value (Fig. 5). This can be attributed to the dependence of the ionization of BPA on the pH value, which can be explained by the fact that BPA is a weak acid with pKa ≈ 9.5 and dissociates at pH > pKa [15]. The removal percentage of BPA on the raw bentonite at pH 3, 5, 7, and 11 was 42.3%, 37.1%, 34.4% and 36.1%, respectively. While, the removal percentage of BPA on the modified bentonite at pH 3, 5, 7, and 11 was 51.8%, 50.3%, 45.1% and 42.5%, respectively.

This increase in BPA adsorption can be attributed to the coating of the bentonite surface with positive charges and

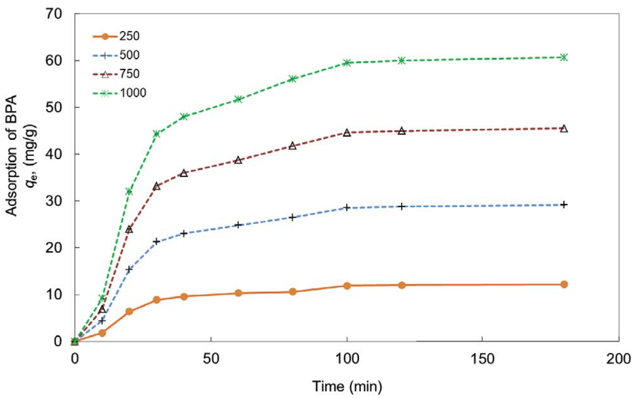


Fig. 3. Dynamics of BPA uptake by bentonite for various initial BPA concentrations.

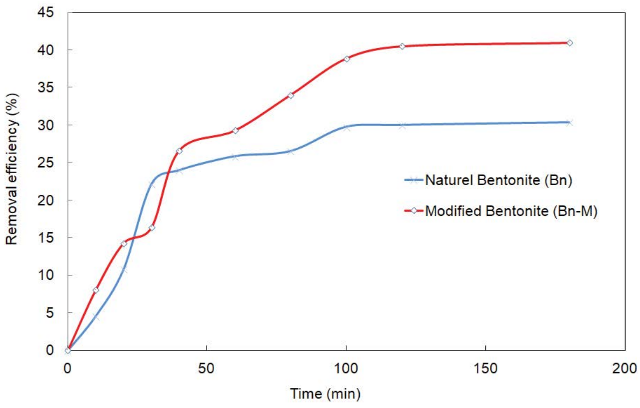


Fig. 4. Effect of contact time and the nature of bentonite on the removal efficiency of BPA.

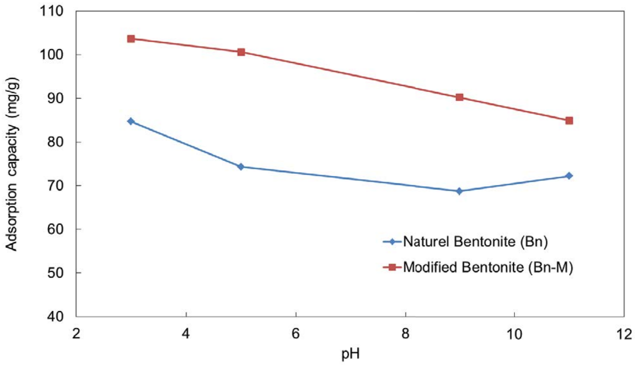


Fig. 5. Effect of pH on BPA adsorption by raw bentonite and modified bentonite.

the enhancement of the electrostatic interaction between the modified bentonite and BPA [32].

3.3. Adsorption isotherm

Adsorption isotherms play an important role for understanding the adsorption mechanism and it is very important to evaluate the feasibility of the adsorbate–adsorbent system. The Langmuir and Freundlich adsorption isotherm models were used to characterize BPA adsorption on bentonite and modified bentonite in this work. The result obtained is non-linear and the data was calculated for affinity with Freundlich isotherm model. Adsorption isotherms of BPA onto bentonite and modified bentonite are presented in Fig. 6.

The sorption coefficient was calculated using the Freundlich model specified in Eq. (3):

$$q_e = K_F C_e^{1/n} \tag{3}$$

where C_e and q_e are the equilibrium solute concentration in mg/mL and the concentration in the solid in mg/g, respectively, $1/n$ is related to the energy distribution of the sorption sites and K_F represents the sorption capacity.

The Freundlich isotherm model has a linear form, which is given by Eq. (4):

$$\log q_e = K_F + \frac{1}{n}\log C_e \tag{4}$$

The intercept and slope, respectively, of the linear plot of experimental data of logq_e vs. logC_e, as shown in Fig. 7, can be used to calculate K_F and $1/n$.

The linear form of Langmuir equation can be written as follows [Eq. (5)]:

$$\frac{C_e}{q_e} = \frac{1}{\left(q_m K_L\right)} + \left(\frac{C_e}{q_m}\right) \tag{5}$$

where C_e (mg/L) is the concentration of BPA at equilibrium, q_e (mg/g) is the amount of BPA adsorbed by the bentonite at equilibrium, K_L (L/mg) is the Langmuir constant, and q_m (mg/g) is the maximum adsorption capacity corresponding to monolayer coverage [33].

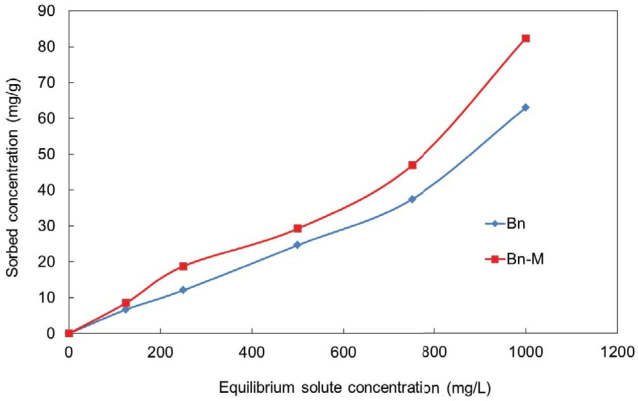


Fig. 6. Adsorption isotherm of BPA using bentonite (Bn) and modified bentonite (Bn-M) at temperature of 25°C.

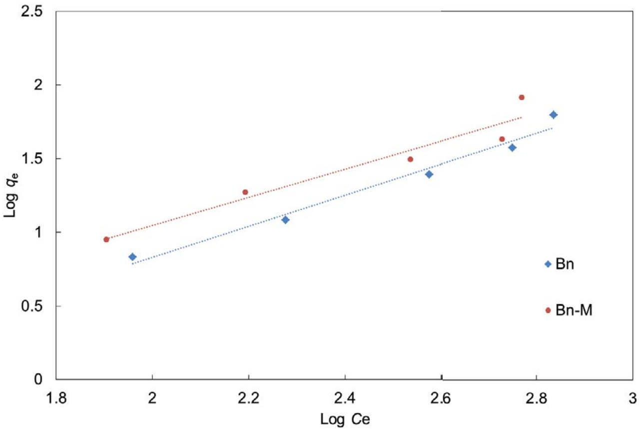


Fig. 7. Freundlich isotherms for BPA adsorption on bentonite (Bn) and modified bentonite (Bn-M) at temperature of 25°C.

Equilibrium parameter R_L, is defined according to the following equation:

$$R_L = \frac{1}{\left(1 + K_L C_0\right)} \tag{6}$$

where K_L (L/mg) is the Langmuir constant and C_0 (mg/L) is the initial BPA concentration.

The intercept and slope of the linear plot of experimental data of $1/q_e$ vs. $1/C_e$, as shown in Fig. 8, is used to calculate these constants.

The values of the Langmuir and Freundlich constants were determined using the plots in Figs. 7 and 8, and the results are presented in Table 2.

If we compare the correlation coefficients, we can say that the experimental data fitted the Langmuir isotherm model better. For monolayer adsorption on a surface with a finite number of identical sites, the Langmuir isotherm is correct. The value of R_L was determined to be 0.7710–0.8274, confirming that the Langmuir isotherm was favorable for BPA adsorption on bentonite at the conditions used in this study. The maximum amount adsorbed q_{max} was 355.87 and 359.71 mg/g for clay and modified clay, respectively. These values are largely superior compared to other experimental data as shown in Table 3. The use of modified and unmodified Moroccan bentonite is very effective in eliminating pollutants and improving water quality [34].

When the Freundlich model is applied, we find a K_F value of 0.0539 $mg^{1-1/n}·mL^{1/n}·g$ for bentonite and 0.1393 $mg^{1-1/n}·mL^{1/n}·g$ for modified bentonite. For example, the adsorption coefficients K_F for BPA on natural sodium bentonite from Wyoming are from 5.68 to 6.27 $mg^{1-1/n}·mL^{1/n}·g$ [1,21,35,36].

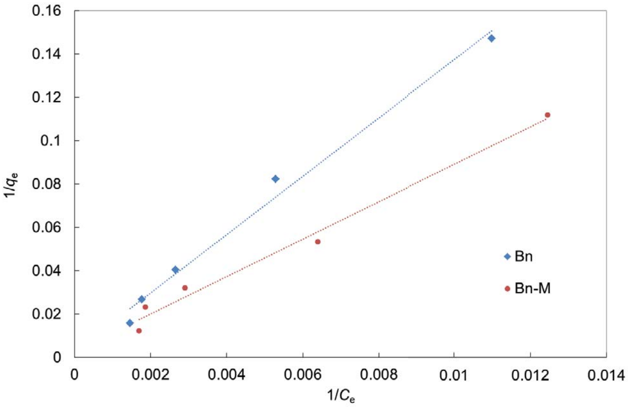


Fig. 8. Langmuir isotherms for BPA adsorption on bentonite (Bn) and modified bentonite (Bn-M) at temperature of 25°C.

Table 2
Langmuir and Freundlich constants for the adsorption of BPA onto bentonite (Bn) and modified bentonite (Bn-M) at temperature of 25°C

		Bn	Bn-M
Freundlich isotherm model	n	0.95	1.05
	K_F	0.05	0.14
	R^2	0.97	0.93
Langmuir isotherm model	q_m	355.87	359.71
	K_L	0.0002	0.0003
	R_L	0.83	0.77
	R^2	0.99	0.99

Table 3 shows comparison data for the removal capacity of BPA by several adsorbents, such as montmorillonite modified with DDDMA, thermo-responsive powdered activated carbon, Iron nanoparticle-doped magnetic ordered mesoporous carbon, organo-montmorillonite and barley husk [15]. Although the maximum adsorption capacity (q_m) of BPA by the activated bentonite obtained by the Langmuir equation was relatively great than that by barley husk and montmorillonite, it is of the same order at that of heat-sensitive thermo-responsive powdered activated carbon and iron nanoparticle-doped magnetic ordered mesoporous carbon.

3.4. Effect of temperature

The objective of this study is to investigate the impact of temperature in a range from 25°C to 65°C on the sorption of BPA on bentonite, at solid/ liquid ratio, 0.25 g/50 mL, and adsorption time of 120 min, in order to obtain representative transfer parameters. The experimental results obtained for the determination of the adsorption parameters of BPA calculated for the bentonite at 25°C, 35°C, 45°C, 55°C and 65°C are shown in Table 4.

It is noted that in a linear approximation of the experimental results, the values of the adsorption capacity

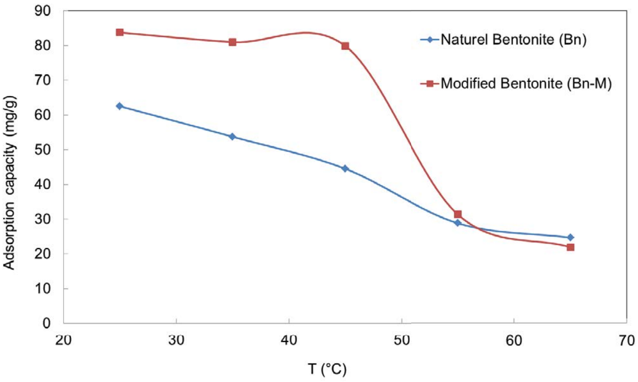


Fig. 9. Effect of temperature on BPA adsorption onto bentonite and modified bentonite.

Table 3
List of models for the isotherms and kinetics of Bisphenol A adsorption on various materials

Adsorbents	Maximum adsorption capacity q_m (mg/g)	Solid/liquid ratio	Isotherm models	References
Montmorillonite modified with DDDMA	256.41	0.25 g/40 mL	L	[21]
Raw bentonite	3.413	1 g/150 mL	F	[37]
Thermo-responsive powdered activated carbon	354.71	5 mg/100 mL	L,F	[38]
Iron nanoparticle-doped magnetic ordered mesoporous carbon	347.2	5 mg/10 mL	F	[39]
Organo-montmorillonite	222.2	0.05 g/50 mL	L	[40]
Barley husk	15.51	5 g/L	L	[15]

Table 4
Removal efficiency and adsorption capacity of BPA on bentonite and modified bentonite

T (°C)		25	35	45	55	65
Capacity adsorption q_e (mg/g)	Bn	62.52	53.74	44.50	28.80	24.64
	Bn-M	83.77	81.00	79.84	31.34	21.87
Removal efficiency R%	Bn	31.26	26.87	22.25	14.40	12.32
	Bn-M	41.88	40.50	39.92	15.67	10.94

q_e (mg/g) of BPA onto modified bentonite are 83.7690 and 21.8704 mL/g at 25°C and 65°C, respectively. We observed a decrease in q_e when the temperature increases which is consistent with the results presented in the literature for VOCs [30,41]. Fig. 9 shows clearly the impact of the temperature on the adsorption of BPA on natural bentonite. It has indeed been observed, a decrease in adsorbent properties with the increase in temperature from 25°C to 65°C, in other words, the temperature increases the diffusive transfers. The decrease of adsorption capacity with the increase in temperature indicated that the adsorption of BPA onto bentonite is exothermic in nature.

4. Conclusion

During this work, we studied the adsorption of BPA on raw and modified bentonite in the Bach system in an aqueous medium. This adsorption depends on the physicochemical parameters involved such as the pH, the temperature and the mass of adsorbent. The sorbed amounts of BPA increase when increasing contact time, and reaching a maximum value after 120 min. The first result obtained is that the sorption isotherms obtained are non-linear, and the Langmuir isotherm was demonstrated to provide the best correlation for the adsorption of BPA.

The solution pH played a significant role in influencing the capacity of an adsorbent towards BPA molecules. An increase in the pH of solutions leads to an decrease in the sorption capacities of BPA on the sorbent under study. As a result of this study, a decrease in adsorbent properties was observed with the increase in temperature from 25°C to 65°C. The decrease of adsorption capacity with the increase in temperature indicated that the adsorption of BPA onto bentonite is exothermic in nature. Finally, with results like this, natural bentonite, a cheap and readily available clay, can be an alternative to more expensive adsorbents used for the removal of pollutants in the leachate treatment processes.

Author's contribution

The current research done by the authors.

Disclosure statement

Authors have no competing interest.

Funding

There was no any funding received at any part for the completion of this thesis.

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