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Removal of orthophosphate ions from aqueous solution using chitin as natural adsorbent

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ABSTRACT

In the present work, HPO_4^{2-} and $H_2PO_4^-$ ions adsorption onto the chitin has been studied in the static system using synthetic solutions of (Na_2HPO_4, $12H_2O$) and NaH_2PO_4, with initial concentrations of 100 and 1,000 mg/l. The adsorption process is affected by various parameters such as contact time, initial concentration, pH solution, and temperature. The results show that the chitin used avoids high capacity to retain orthophosphate ions up to 0.91 and 0.75 g/g for HPO_4^{2-} and $H_2PO_4^-$, respectively. The equilibrium is reached after 1 h for HPO_4^{2-} and 3 h for $H_2PO_4^-$ ions. The adsorption isotherms show a good correlation with the Langmuir and Freundlich model.

Keywords: Adsorption; Orthophosphates; Chitin; Wastewater

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1. Introduction

The excessive presence of orthophosphate compounds in water resources has an adverse risk to human health and leads to eutrophication phenomena [1–5]. Several purification techniques based on the physicochemical and biological processes have been designed to remove or reduce the amount of toxic pollutants in wastewater. Most of these purification techniques require significant technological and financial investment [2,5,6].

The search for new materials that would be good adsorbents, easy to prepare, and whose prices are low is one of the most promising ways. The research based on natural adsorbents of animal, mineral or vegetable origin was carried out by various teams [2,6–14]. In our team, the effectiveness of several adsorbents has been demonstrated with significant potential to remove and reduce heavy metals and inorganic contaminants from synthetic aqueous solutions or from real wastewater [15–17].

Chitin, biomaterial of animal origin may be part of these materials, due to its intrinsic properties, its abundance, and low cost [18]. This is the second most abundant natural polymer after cellulose. It is mainly extracted from the shells of crustaceans such as crabs, shrimp, and lobsters [19,20]. Chitin and its derivatives, in particular chitosan, have shown a significant adsorption potential for the removal of various pollutants especially heavy metals, dyes, and phenol [21,22]. Indeed, it includes within its structure biopolymer more functional groups (carboxyl, alcool, amino group…) that can create interactions with different chemical entities such as orthophosphate ions. Our

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research included in this framework, it aims to assess the potential of the chitin adsorbent to remove orthophosphate ions. The purpose of this study was to evaluate the adsorption capacity of chitin towards orthophosphate ions (HPO_4^{2-} and $H_2PO_4^-$) from synthetic solutions. The study of the effect of various parameters such as mass of the adsorbent, stirring time, pH, temperature, and initial concentration on the adsorption of HPO_4^{2-} and $H_2PO_4^-$ by chitin was also performed.

2. Materials and methods

2.1. Adsorbent

Chitin is the polymer N-acetyl-β-D-glucosamine or 2-acetamido-2-deoxy-β-D-glucose linked by β (1–4) linkages. It differs from other polysaccharides by the presence of nitrogen in addition to carbon, hydrogen, and oxygen in the macromolecular chain [23].

In this study, we used the commercial chitin brand "loba chemie cas N° 1398-61-4" and molar mass 400.000 g/mol. Chitin is sieved (particle size < 500 microns).

2.2. Preparation of orthophosphate solutions

Solutions of orthophosphate ions (HPO_4^{2-} and $H_2PO_4^-$) were prepared from ($Na_2HPO_4 \cdot 12H_2O$) and (NaH_2PO_4) salt. The concentrations lie in the range between 50 mg/l and 8 g/l. The chosen of the very high range of orthophosphates concentration is justified by knowledge of the maximum amount retained by chitin.

Effect of pH on adsorption of nitrate ions by chitin: R = 25 g/l; T = 25°C; pH 6.11; T_c = 6 h.

2.3. Determination of pH_{zpc}

For the determination of pH_{zpc} of the adsorbent, 50 mL of 0.01 M NaCl solutions were taken in different conical flasks of 100 mL and 0.5 g of adsorbent was introduced in each of them. Now, pH values of these solutions were adjusted in 2–12 range by 0.1 M HCl/NaOH solutions. These flasks were kept for 48 h and the final pH of the solutions was measured. Graphs were plotted between pH_{final} vs. $pH_{initial}$. The point of intersection of the curve of pH_{final} vs. $pH_{initial}$ was recorded as pH_{zpc} of the chitin.

2.4. Adsorption in a static system

The study of the adsorption in the static system based on the introduction of a mass m of chitin in a volume V of orthophosphates ions solution at initial concentration C_i. The mixture is stirred at constant temperature of 25°C using a thermostat. Samples are taken at different contact time and then centrifuged at 500 cycle/min and analyzed. Each experiment was carried out in duplicate and the average results are presented in this study.

The ratio $R = m/V$ used in our study should be the smallest mass which leads to maximum adsorption rates. The determination of this ratio is presented in the results section.

2.5. Analysis and calculation of the adsorbed quantities method

Orthophosphates ions are analyzed by UV–visible spectrophotometer at 537 nm, according to spectrophotometric method (AFNOR T-90–013) norm [4].

The retained concentrations C_r (mg/l) by the chitin at the equilibrium time (T_e) and quantity of adsorption Q_{ads} (mg/g) are given by the following equation:

$$Q_{ads}(\mathrm{mg/g}) = (C_i - C_e) \times \frac{V}{m} \tag{1}$$

where m: mass of crushed Chitin (g), V: total volume (l), C_i: initial concentration (mg/l), C_e: equilibrium concentration (mg/l), Q_{ads}: adsorbed quantity per 1 g of chitin (mg/g).

3. Results and discussion

3.1. Characterization of the adsorbent

The scanning electron microscope (SEM) of chitin (Fig. 1) reveals a homogeneous surface, microscopic multilayer superposables with net interstices between sheets (micro layers) and a high porosity.

The structure of chitin possesses several functional groups to create affinities (hydrogen bonds, electrostatic interaction ...) with several chemical entities with pollution effects as evidenced by its IR spectrum (Table 1) which is consistent with literature data [24,25].

3.2. Zero point charge of adsorbent

The influence of pH on the orthophosphate adsorption can be illustrated on the basis of isoelectric point of the adsorbent surface. Solution pH is a significant parameter which affects the dye adsorption process. It also alters the surface charge of the adsorbent, the ionization extent of different pollutants, as well as

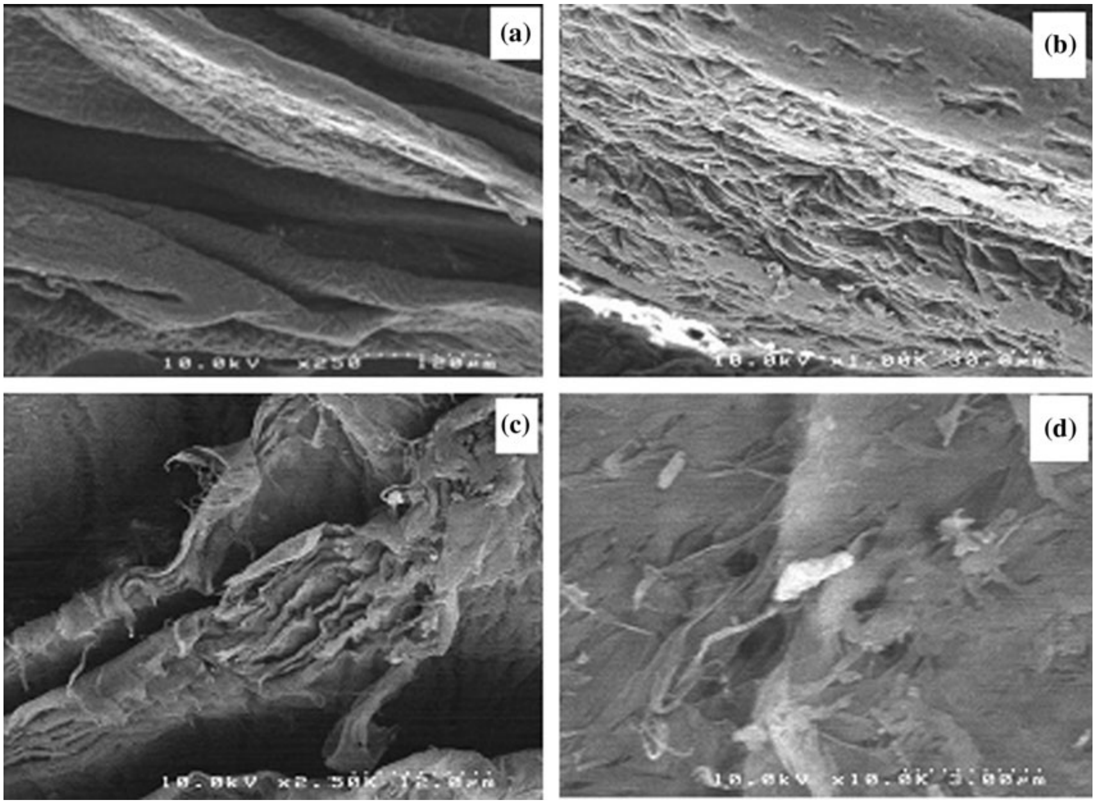


Fig. 1. SEM chitin, particle size <500 microns (Scanning Electron HITACHI S-4500) (a) (magnifications: 250×), (b) (magnifications: 1,000×), (c) (magnifications: 2,500×), and (d) (magnifications: 10,000×).

Table 1
Interpretation of the FT-IR spectrum of chitin (KBr)

v OH	3,400
v NH	3,270; 3,114
v CH	2,890
v C=O	1,640
δ NH + v CN	1,550; 1,320
δ CH	1,440; 1,380
v C–O	1,070; 1,030
v–stretching vibrations	
δ–bending angular	

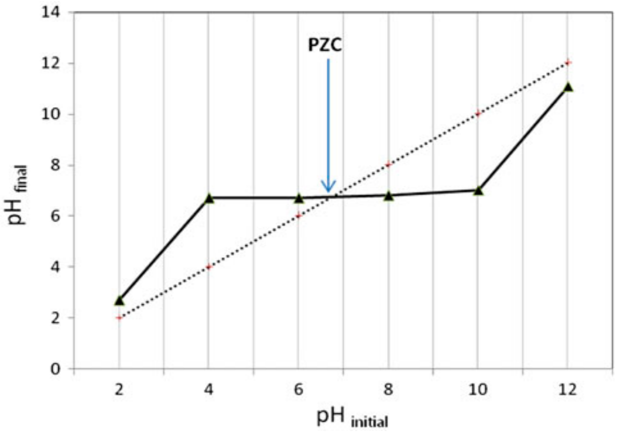


Fig. 2. Point of zero charge for chitin adsorbent.

the structure of the orthophosphate ions. The presence of various ligands such as carboxyl, phosphate, and amino group on lignin- and cellulose-based materials in the ionic state contributes to the reaction with oxyanions. At pH_{zpc}, the acidic and basic functional groups no longer contribute to the pH of the solutions. The zero point charge, pH_{zpc} of chitin, was found to be 6.7 (Fig. 2). At a pH of the solution below pH_{zpc} the surface of the sorbent is positively charged and can attract anions from the solution. When the solution pH is greater than pH_{zpc} the surface of chitin is negatively charged and can attract cations from the solution. In this study, a lower pH is favorable for orthophosphate ions as at low pH, the number of positively charged sites is increased which favor the adsorption of negatively charged orthophosphate ions because of the electrostatic force of attraction.

3.3. Determination of the ratio mass/volume

This ratio corresponds to the lowest mass leading to a maximum rate of removal of $H_2PO_4^-$ and HPO_4^{2-} ions. All our study was performed with a ratio R = mass/volume of the solution as $R = 25$ g/l, which is 1 g/40 ml. This ratio was determined from the evolution of the concentration retained in orthophosphates ions $H_2PO_4^-$ and HPO_4^{2-} depending on the mass/volume ratio R (Figs. 3 and 4). This change was followed for a $C_i = 100$ mg/l and $C_i = 1$ g/l.

It is found that the concentration selected stabilizes at a maximum value corresponding to a ratio (m/V) equal to 25 g/l. This result shows that 1 g of chitin with 40 ml of solution is sufficient to achieve the maximum adsorption for orthophosphates ions. This ratio is similar to that obtained previously with the vegetable adsorbents studied in our laboratory [16,17].

3.4. Effect of contact time

The contact time of adsorbate and adsorbent has a great importance in batch adsorption experiments, because it depends on the nature of the system used. Consequently, it is important to establish the time dependence of such systems under various process conditions. The effect of contact time on the adsorption of $H_2PO_4^-$ and HPO_4^{2-} ions was performed for two initial concentrations: $C_i = 100$ mg/l, and $C_i = 1$ g/l (Figs. 5 and 6).

It is noted that the amount adsorbed of $H_2PO_4^-$ and HPO_4^{2-} ions retaining by chitin surface increases with contact time. The adsorption equilibrium is reached after a contact time of about 1 h for HPO_4^{2-}. While for $H_2PO_4^-$ ions, it is practically reached after 4 h at low concentration ($C_i = 100$ mg/l) and 3 h at high concentration (1 g/l).

3.5. Determination of the maximum amount adsorbed at saturation

The effect of the initial concentration on the adsorption of $H_2PO_4^-$ and HPO_4^{2-} ions was investigated at different concentrations ranging from 50 mg/l to 8 g/l (Fig. 7).

Analysis of this curve shows that the adsorption capacity of orthophosphates ions increases with the initial concentration, even for high initial concentrations (8 g/l) without reaching the level corresponding to saturation.

The values of adsorbed amounts Q_{ads} per unit mass of the chitin, with high concentrations of orthophosphates ions are summarized in Table 2.

To determine the maximum amount adsorbed ($Q_{ads,max}$), we have calculated the theoretical maximum amount adsorbed by plotting the curve corresponding to the variation of $1/C_r$ in terms of $1/C_i$

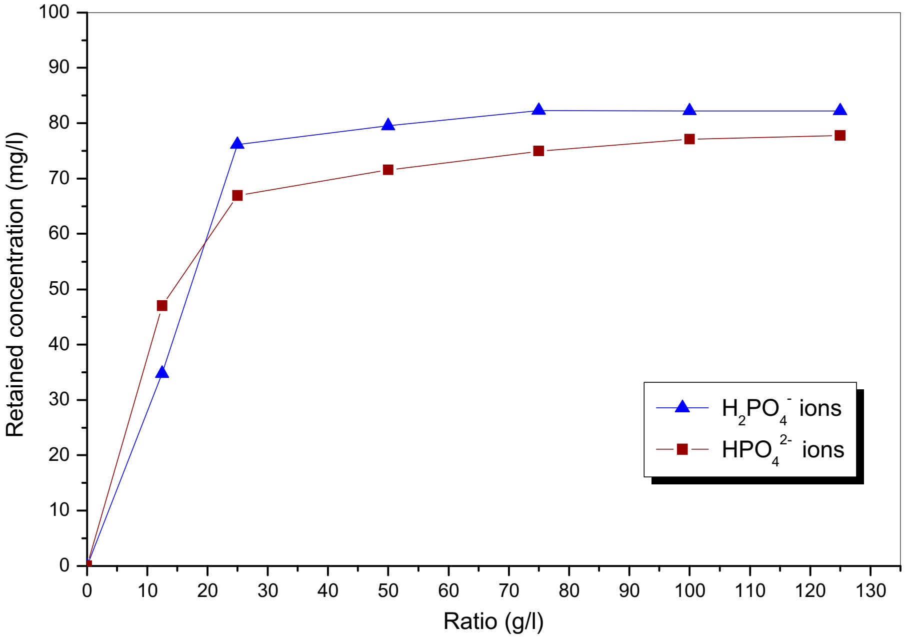


Fig. 3. Variation of the retained $H_2PO_4^-$ and HPO_4^{2-} ions concentration in terms of the ratio R at $C_i = 100$ mg; $T = 25$℃; pH 6.1; $T_c = 24$ h.

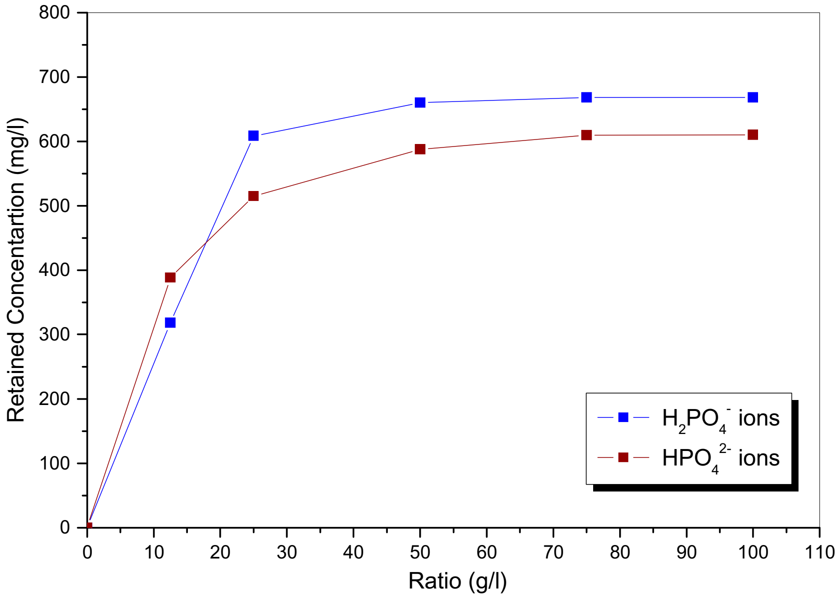


Fig. 4. Variation of the retained $H_2PO_4^-$ and HPO_4^{2-} ions concentration in terms of the ratio R at $C_i = 1$ g/l; $T = 25$°C, pH 6.5; $T_c = 24$ h.

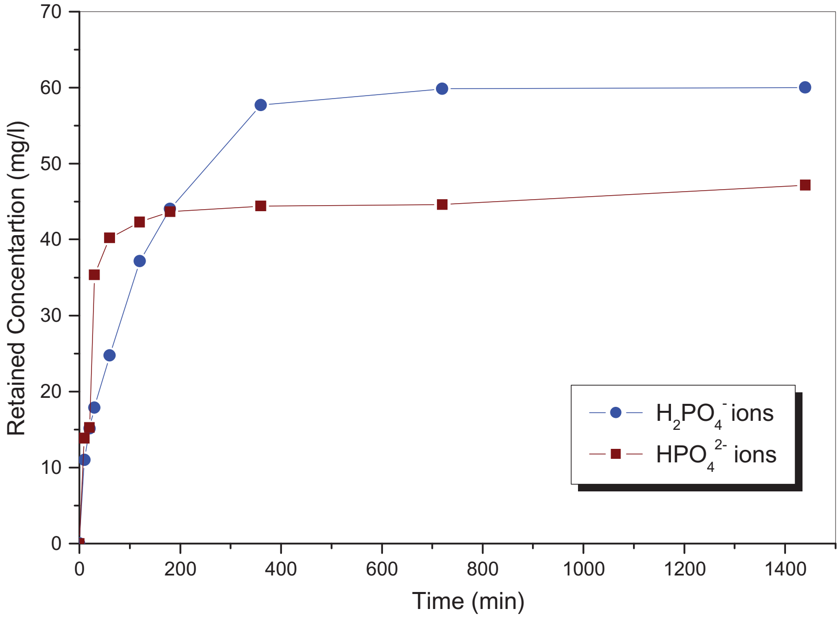


Fig. 5. Variation of the retained $H_2PO_4^-$ and HPO_4^{2-} ions concentration (C_r) in terms of contact time at $C_i = 100$ mg/l; $R = 25$ g/l, $T = 25$°C, pH 6.5.

extrapolated to an infinite concentration (Fig. 8). We have taken the points approaching the saturation level and which corresponding to high concentrations.

By extrapolation to an infinite concentration, the maximum retained concentration of $H_2PO_4^-$ and HPO_4^{2-} at total saturation of the chitin was about 18.93 and 22.78 g/l, respectively. When the amounts adsorbed at saturation by 1 g of chitin can reach up to about 0.75 and 0.91 g/g for $H_2PO_4^-$ and HPO_4^{2-}, respectively. These amounts are much higher than those obtained with several other natural adsorbents [2,25]. These results suggest that chitin has a significant adsorption potential towards orthophosphates ions.

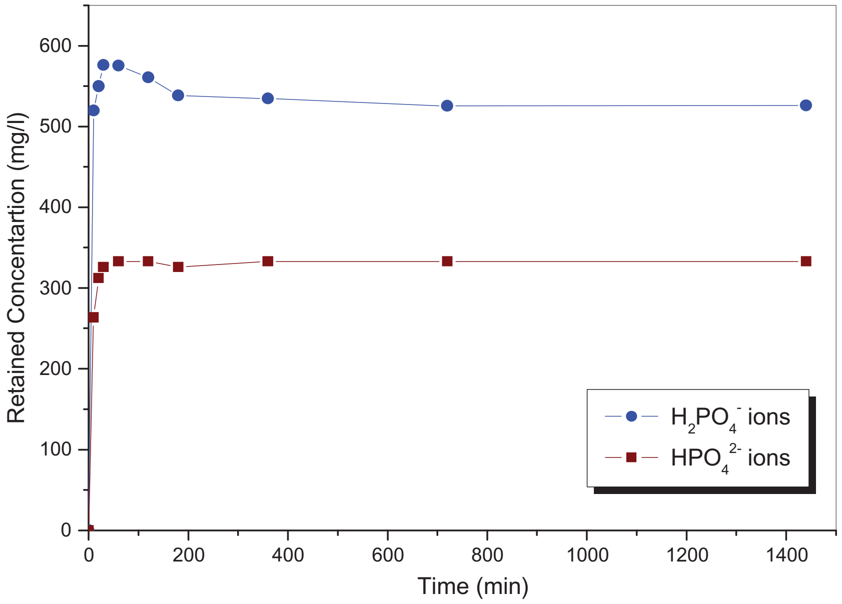


Fig. 6. Effect of contact time on adsorption of $H_2PO_4^-$ and HPO_4^{2-} ions onto chitin adsorbent: $C_i = 1$ g/l; $R = 25$ g/l; $T = 25$°C; pH 6.12.

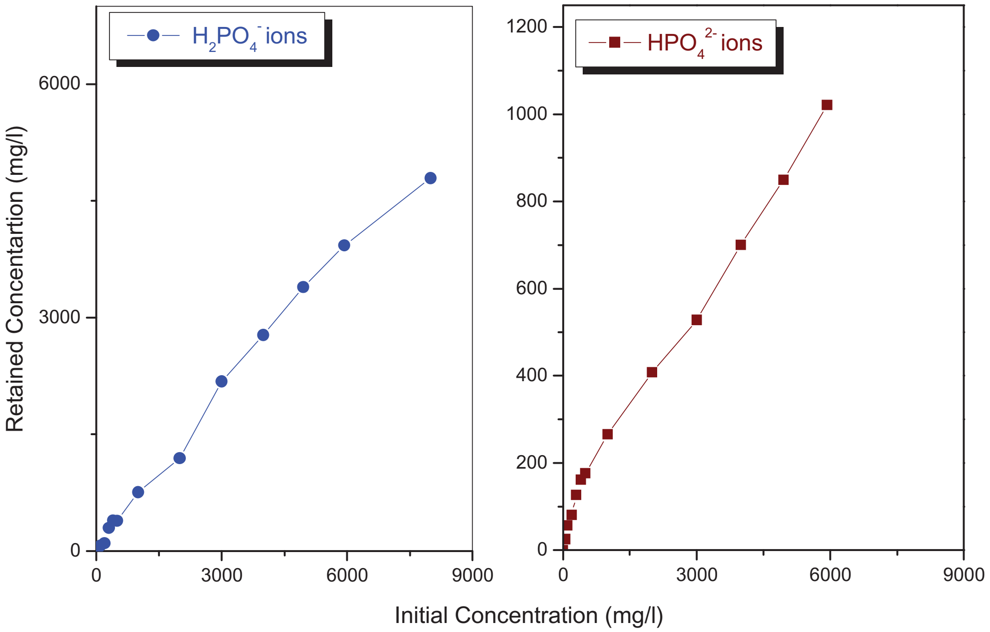


Fig. 7. Effect of initial orthophosphate concentration on adsorption of $H_2PO_4^-$ and HPO_4^{2-} ions by chitin adsorbent: $R = 25$ g/l; $T = 25$°C; pH 6.1; $T_c = 6$ h.

3.6. Adsorption isotherms

The adsorption isotherms of orthophosphate ions on chitin were determined for Freundlich and Langmuir models.

Langmuir Isotherm [26]:

$$\frac{1}{C_r} = \frac{1}{C_{r\,max}} + \frac{1}{K\,C_{r\,max}}\left(\frac{1}{C_e}\right) \tag{2}$$

where C_e: concentration of orthophosphate ions at equilibrium (mg/l), C_r: retained concentration

Table 2
The amount retained at equilibrium of chitin as function of the initial concentration of $H_2PO_4^-$ and HPO_4^{2-}

C_i (g/l)		0.5	1	2	3	4	5	6	8
$H_2PO_4^-$	C_r (g/l)	0.39	0.755	1.19	2.18	2.77	3.39	3.93	4.79
	Q_{ads} (mg/g)	15.54	30.22	47.65	87.17	110.9	135.59	157.07	191.54
HPO_4^{2-}	C_r (g/l)	0.18	0.266	0.41	0.53	0.70	0.85	1.022	–
	Q_{ads} (mg/g)	7.05	10.62	16.33	21.12	28.02	33.99	40.86	–

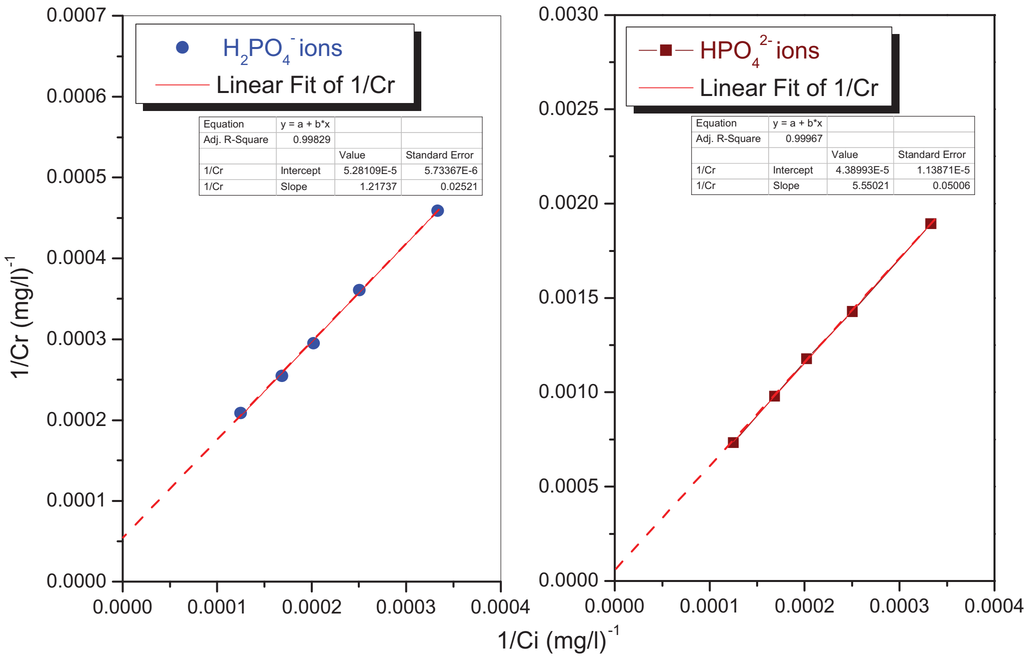


Fig. 8. Variation of $1/C_r$ in terms of $1/C_i$.

orthophosphate ions (mg/l), $C_{r,max}$: maximum adsorption concentration of orthophosphate ions (mg/l), K_L: Langmuir constant (l/mg).

Freundlich Isotherm [27]:

$$\mathrm{Ln}\, Q_{ads} = \mathrm{Ln}\, K_f + \frac{1}{n_f}\mathrm{Ln}\, C_e \tag{3}$$

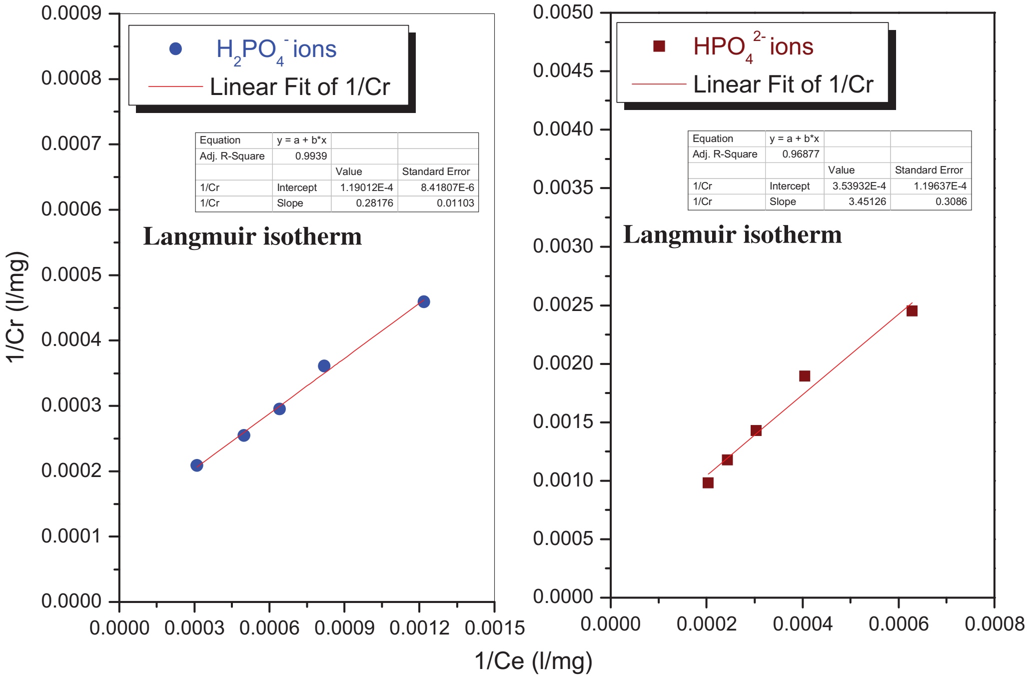
where Q_{ads}: adsorption capacity (mg/g), C_e: concentration of orthophosphate ions at equilibrium (mg/l), K_f and n: Freundlich constants.

Figs. 9 and 10 show plots of the Langmuir and Freundlich adsorption isotherms. Table 3 summarizes the parameters for both the adsorption isotherms. It was found that this adsorption is well described by the Langmuir isotherm ($R^2 = 0.99$ for $H_2PO_4^-$ and $R^2 = 0.96$ for HPO_4^{2-}), which causing adsorption at independent sites of the same nature, and therefore the lack of interaction between the adsorbed ions. A good correlation obtained with the Freundlich isotherm suggests the existence of several actives sites of adsorption which obey the independence criteria Langmuir and reinforces the good correlation obtained with the Langmuir isotherm.

The value of the constant $1/n_f$ is less than 1 which suggests that the adsorption sites are not homogeneous. In similar studies on clays, Bouras [28] attribute this phenomenon to the existence of several types of interaction and believe that the adsorption is of chemical type.

3.7. Effect of initial pH

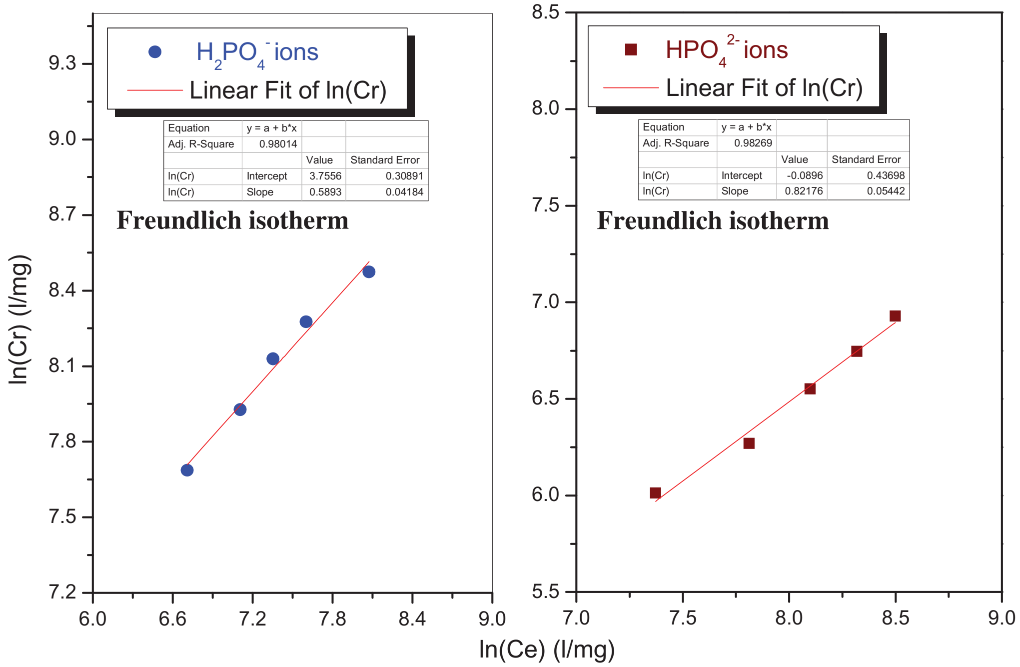
One of the most critical parameters in the adsorption process of phosphate ions from aqueous solutions is the pH of the medium. The initial pH of adsorption



Equation	y = a + b*x		
Adj. R-Square	0.9939		
		Value	Standard Error
1/Cr	Intercept	1.19012E-4	8.41807E-6
1/Cr	Slope	0.28176	0.01103

Equation	y = a + b*x		
Adj. R-Square	0.96877		
		Value	Standard Error
1/Cr	Intercept	3.53932E-4	1.19637E-4
1/Cr	Slope	3.45126	0.3086

Fig. 9. Langmuir adsorption isotherm for $H_2PO_4^-$ and HPO_4^{2-} on the chitin adsorbent.



Equation	y = a + b*x		
Adj. R-Square	0.98014		
		Value	Standard Error
ln(Cr)	Intercept	3.7556	0.30891
ln(Cr)	Slope	0.5893	0.04184

Equation	y = a + b*x		
Adj. R-Square	0.98269		
		Value	Standard Error
ln(Cr)	Intercept	-0.0896	0.43698
ln(Cr)	Slope	0.82176	0.05442

Fig. 10. Freundlich adsorption Isotherm for $H_2PO_4^-$ and HPO_4^{2-}on the chitin adsorbent.

medium is related to the adsorption mechanisms onto the adsorbent surface from water and reflects the nature of the physicochemical interaction of the species in solution and the adsorptive sites of adsorbents [17]. The effect of initial pH on the adsorption of $H_2PO_4^-$ and HPO_4^{2-} ions by chitin was performed at an initial

Table 3
Langmuir and Freundlich parameters of adsorption of $H_2PO_4^-$ and HPO_4^{2-} ions on the chitin

	Langmuir			Freundlich		
	Q_m (g/g)	K_L	R^2	$1/n_f$	K_f	R^2
$H_2PO_4^-$	0.336	4.22×10^{-4}	0.99	0.589	42.75	0.98
HPO_4^{2-}	0.11	1.025×10^{-4}	0.96	0.821	0.914	0.98

Table 4
Values of initial pH and pH at equilibrium

Initial pH		2	4	6	8	10
Equilibrium pH	$H_2PO_4^-$	–	5.73	5.51	5.61	5.84
	HPO_4^{2-}	–	5.22	5.48	5.54	5.92

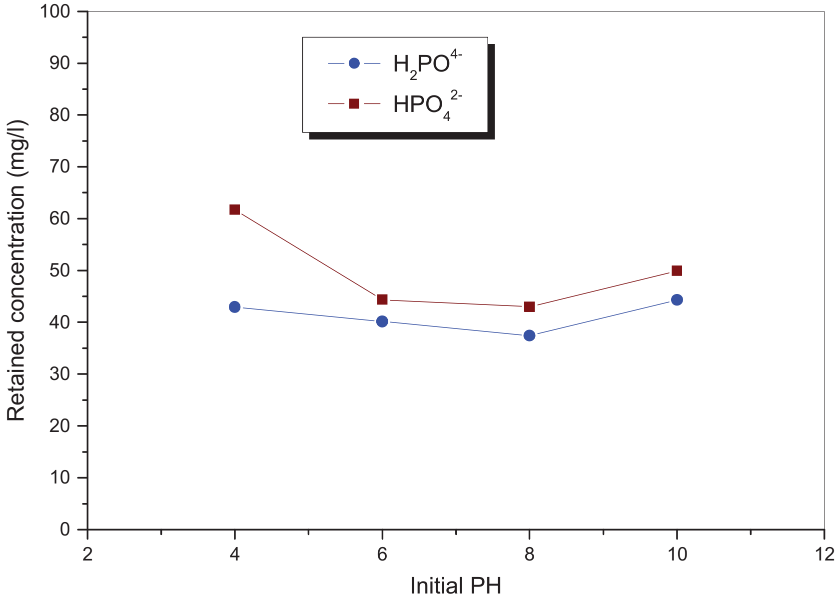


Fig. 11. Effect of initial pH on adsorption of $H_2PO_4^-$ and HPO_4^{2-} ions onto chitin adsorbent: $R = 25$ g/l, $T = 25$°C, $T_c = 24$ h; $C_i = 100$ mg/l.

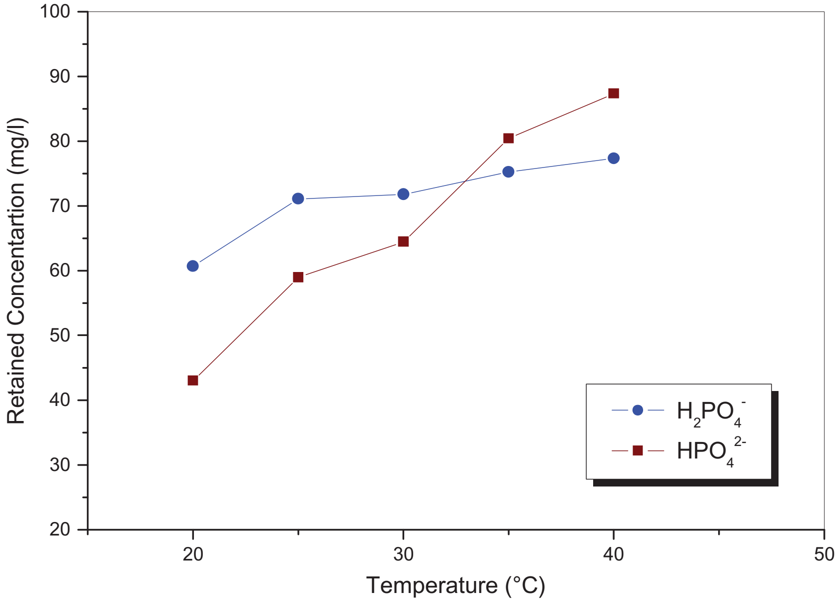


Fig. 12. Effect of temperature on adsorption of $H_2PO_4^-$ and HPO_4^{2-} ions onto chitin adsorbent: $R = 25$ g/l; $T = 25$°C; pH 6.51; $T_c = 24$ h; $C_i = 100$ mg/l.

concentration of 100 mg/l at different pH values. The pH of the solutions was adjusted in the range 2–12 using 1 M HCl or 1 M NaOH. The adsorption results are shown in Fig. 11. Analysis of this curve shows that the retained quantity in $H_2PO_4^-$ ions is slightly increased. While for HPO_4^{2-}, it slightly increased for the initial pH of the solution which is acidic and remains unchanged as the pH of the solution is basic.

The measurement of pH solutions (Table 4) shows that the pH of the solution to the equilibrium does not change in the same order as the initial pH. The chitin would have a regulator effect of pH and therefore the variation of the retained concentration is not important even though the pH varies significantly.

3.8. Effect of temperature

A change in temperature will change the equilibrium capacity of the adsorbent for particular adsorbate. Usually, the adsorbate uptake decreases with increasing temperature due to the exothermic nature of the simple adsorption reaction, while the phosphate adsorption on dried plants was reported as an endothermic process [17]. In Fig. 12, we have presented the evolution of the retained concentration of $H_2PO_4^-$ and HPO_4^{2-} ions in terms of temperature at initial concentration of 100 mg/l. This figure shows that the retained orthophosphate ions concentration (C_r) increases with increasing the temperature; the adsorption of orthophosphate ions is endothermic. Indeed, research has showed that the adsorption of phosphate ions increases with increasing temperature [29].

4. Conclusion

(1) The chitin used has great adsorbent potential to remove orthophosphate ions. At full saturation, the maximum retained concentrations of HPO_4^{2-} and $H_2PO_4^-$ was, respectively, about 0.75 and 0.91 g/g.
(2) The adsorbed amount depends on the contact time and the pH solution. Apparently chitin acts as a pH regulator, and therefore the variation of the concentration is not significant restraint although the initial pH varies significantly.
(3) The adsorption of orthophosphate anions on chitin is endothermic, an increase in temperature causes an increase in adsorption.
(4) A good correlation of Langmuir and Freundlich adsorption isotherms leads to independent sites of the same nature, and therefore the lack of interaction between the adsorbed ions.

Abbreviations

Q_{ads}	—	adsorption capacity (mg/g)
C_i	—	initial concentration (mg/l)
C_e	—	equilibrium concentration (mg/l)
m	—	mass of chitin (g)
V	—	volume of the ionic solution (l)
R	—	mass/volume ratio (g/l)
T_c	—	contact time (min)
T_e	—	equilibrium time
T	—	temperature (°C)
C_r	—	retained concentration (mg/l)
R^2	—	correlation coefficient

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