## Original Article

# Biosorption of heavy metals from aqueous solutions onto chitin

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## ABSTRACT

**Aims:** This study investigates the potential of chitin shrimp shells for the removal of arsenic (V) and zinc (II) ions from aqueous solutions.

**Materials and Methods:** In this study, the chitin was extracted from shrimp shells for arsenate and zinc ions removal from aqueous solutions. The effects of pH, adsorbent dosage, contact time and initial metal ion concentration on As (V) and Zn (II) removal were investigated in a batch system.

**Results:** The equilibrium data were described using the Langmuir and Freundlich isotherm models. The Langmuir equation was used to find the maximum adsorption capacity for arsenate (11.574 mg/g) and Zn (270.27 mg/g). Biosorption was found to depend significantly on the pH of the solution and is optimal at pH values of 4 and 7. To determine the rate-controlling mechanism for metallic ion adsorption, pseudo-first-order and pseudo-second-order equation kinetic models were tested with experimental adsorption kinetic data. Modeling results revealed that As (V) and Zn (II) kinetics data were successfully described using pseudo-first and second-order models, respectively. FTIR analyses showed that hydroxyl and carboxyl groups could be very effective for capturing these metals.

**Conclusion:** Results showed that the chitin is a good adsorbent for the removal of arsenate and zinc ions from aqueous solutions.

**Key words:** Biosorption, chitin, heavy metal, isotherm, kinetic

## INTRODUCTION

In recent years, a sudden increase of industrial activities has intensified more environmental problems as seen for example in the deterioration of several ecosystems due to the accumulation of perilous pollutants, such as heavy metals. Heavy metals are still being used in various industries such as battery manufacture, tanneries, paint manufacture, pigment manufacture, printing and photographic industries, etc., due to their technological importance.[1,2] The enormous increase in the use of the heavy metals over the past few decades has inevitably resulted in an increased flow of metallic substances in the aquatic environment.[3] Heavy metal unlike organic pollutants, the majority of which are susceptible to biological degradation and do not degrade into any harmless end products.[4] Therefore, it becomes

necessary to remove them from wastewaters to a feasible extent by an appropriate treatment.[5] There are various methods for the removal of toxic metals from aqueous solution such as chemical precipitation, evaporation, ion-exchange, adsorption, cementation electrolysis and reverse osmosis.[6] But these methods have some disadvantages. Conventional precipitation method does not always provide a satisfactory removal rate to meet the pollution control limits and synthetic ion-exchange resins are often subjected to be expensive and therefore, the cost-effective alternative technologies or sorbents for treatment of metals from aqueous solutions are needed.[7] Recently, adsorption has become one of the alternative treatments. Basically, adsorption is a mass transfer process by which a substance is transferred from the liquid phase to the surface of a solid, and becomes bound by physical and/or chemical interactions.[8] In this method, different materials such as chitinous materials (e.g., shrimp, krill, squid, and crab shell), microbial biomass (e.g., bacteria, fungi, and yeast), and algal biomass can be used.[9] Among the many other low-cost adsorbents identified, chitin [Figure 1] and its derivatives are nontoxic, biodegradable, biocompatible, antibacterial and environmental-friendly polymers with high molecular weight.[10]

The objectives of this study are to investigate the efficiency of chitin extracted from shrimp shell in the removal of zinc and arsenate ions from aqueous solutions. The effects of initial solution pH, initial metal ion concentration, adsorbent dosage and time contact on adsorption characteristics of chitin extracted of shrimp shell were examined. The experimental data was compared with Langmuir and Freundlich isotherms.

# MATERIAL AND METHODS

## The extraction of chitin from shrimp shells

Shrimp shell were washed several times with water and dried at 50°C overnight in a dry heat incubator. After this, they were mechanically grinded in a mixer and passed through a 16 mesh filter. Demineralization step was carried out by a treatment with 10% HCl at room temperature for 24 h, which also resulted in removal of the $CaCO_3$. The demineralization step was followed by rinsing with distilled water until neutrality was reached. Deproteinization was performed using alkaline treatment with 10% NaOH solution at room temperature for 24 hours. The chitin
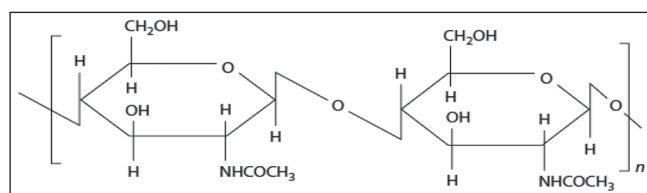


**Figure 1:** Chemical structure of chitin

product was filtered through Whatman filter paper with the aid of a vacuum pump and washed with distilled water until the pH became neutral. Then the shrimp shell residue was subjected to the above-mentioned program twice. Pigment traces responsible for the brown color of this product were removed using 250 ml of 95% and absolute ethanol. Finally, white chitin was washed with distilled water and dried at 50°C in a dry heat sterilizator. The structure of chitin and its purity were evaluated using FT-IR spectroscopy and chemical methods.

## Sorption experiments

Sorption experiments were carried out in batches as follow. A stock solution of Zn (II) and As (V) was prepared by dissolving $Na_3AsO_4$ and $ZnCl_2$ in double distilled water. Experiments were carried out in 500 ml Erlenmeyer flasks containing 250 ml of different heavy metal concentrations ($Zn^{2+}$ = 50-500 mg/l, As (V): 0.5-10 mg/l). The initial pH of the solution was adjusted to 3 and 7 for zinc and arsenate ions by adding small amounts of dilute NaOH or HCl solution. To each flask was added the same amount of sorbents, and then all the flasks were sealed with silicon cap to minimize evaporation and shaken at 300 rpm in rotary shaking incubator. Samples of 25 ml were taken from each flask at the proper time period and analyzed by an atomic absorption spectrophotometer.

The amount of adsorbed Zn (II) on adsorbent ($q_e$, mg/g), and percent removals (%R) were calculated using the general equation:

$$q = \frac{(C_0 - C_e)V}{W} \tag{1}$$

$$\%R = \frac{C_0 - C_e}{C_0} \tag{2}$$

where $q$ is the adsorption amount (mg/g), $C_0$ and $C_e$ the initial and final concentrations (mg/l), V the volume of solution (l) and W is the mass of biosorbent used (g).

## Analytical methods

The concentration of Zn (II) and As (V) were determined by a flame atomic absorption spectrometer (AAS 5FL model, Germany). The pH of solution was measured at the beginning ($pH_{in}$) and at the end ($pH_{fin}$) of each experiment by a pH meter (Metrohm 827 pH lab, Switzerland). The functional groups on the surface site of chitin were characterized using a Fourier transform infrared spectroscometer (FT-IR System 2000 Model, PerkinElmer, USA) in the range of 400-4000 $cm^{-1}$. The specific surface area of chitin was calculated according to the BET equation.

Zeta potentials of adsorbents were measured across the pH range 2-12. Samples were prepared identically to those of the batch experiments. Thirty measurements were made for each sample on a zeta potential analyzer and the average was

taken as the zeta potential of chitin. The isoelectric point ($pH_{iep}$), the pH at which the positive surface charge equals the negative surface charge, can be determined from these measurements.

## Adsorption isotherms and kinetics

The adsorption isotherms for the As (V) and Zn (II) removed were studied using various initial concentrations at an adsorbent dosage optimal. Then the data obtained were fitted to the Langmuir and Freundlich adsorption isotherm applied to equilibrium adsorption. Equations of these isotherms are:

$$logqe = \log k_f + \frac{1}{n} \log C_e:$$  Freundlich (3)

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{bq_m}:$$  Langmuir (4)

where $q_e$ and $C_e$ are the equilibrium concentration of the adsorbent on the solid (mg/g) and in the liquid phases (mg/l), respectively; $q_m$ is the maximum adsorption capacity (mg/g) according to the Langmuir model; b, $K_f$, and n are constants.

For adsorption kinetic, the mixture of the test solution and chitin was stirred in a shaker at 300 r/min and 25°C continuously for 4 hours. Separate samples for metal ions were drawn after 10, 20, 30, 40, 50, 60, 90, 120, 180 and 240 min intervals.

# RESULT

## Characterization of chitin

The physical properties of chitin extracted from shrimp shell were measured and the results are arranged in Table 1. Figures 2 and 3 show the results of the FTIR analyses of chitin before and after biosorption. The results of the FTIR spectrum showed that there were different functional groups detected on the surface of the chitin. Broad peaks detected in spectra were identified at 3500 to 3200 cm⁻¹ (O-H and N-H), 3000 to 2850 cm⁻¹ (–CH of the alkanes group), 2517.95 cm⁻¹ (OH), 1647 cm⁻¹ (C = O of amide groups), 1633.61 cm⁻¹

(NH of the Amides groups), and 1419 cm⁻¹ (–CH₃). The peak value at 1068 cm⁻¹ is attributed to the C–O links of the carboxylic acids and esters groups.

## Effect of pH

The pH of a solution affects metal ion solubility and biosorbent total charge. Figure 4 shows the biosorption efficiency of each metal at different pH values from 3 to 7. The highest removal of arsenate was achieved at pH of 4, while at pH of 7 the highest removal of zinc was obtained. The maximum uptake ($q$) obtained at the optimum pH value and 0.1 g doses of metals were 1.7 and 15.04 mg/g for $Zn^{2+}$ and $As^{5+}$, respectively.

## Effect of adsorbent dosage

The influence of adsorbent dosage on the removal of heavy metal ions can be studied by varying the adsorbent concentration ranging from 0.1 to 10 g by keeping the volume of the effluent solution constant. The effect of adsorbent dosage on the removal of Zn (II) and As (V) by chitin at optimum temperature (T) 25°C and time ($t$) 180 min is shown in Figure 5. At first, 1 mg/g of As (V) was adsorbed at 0.1 g, which decreased with the increase of adsorbent dosage and reaching the minimum of 0.18 mg/g at 1 grams. Likewise, 4.81 mg/g of Zn (II) was adsorbed at initial dosage of 0.5 g, which decreased with the increase of adsorbent dosage reaching the minimum of 0.56 at 10 grams.

## Effect of contact time and initial metal concentration

The equilibration time is one of the parameters for economical wastewater treatment plant applications. Figure 6 shows the

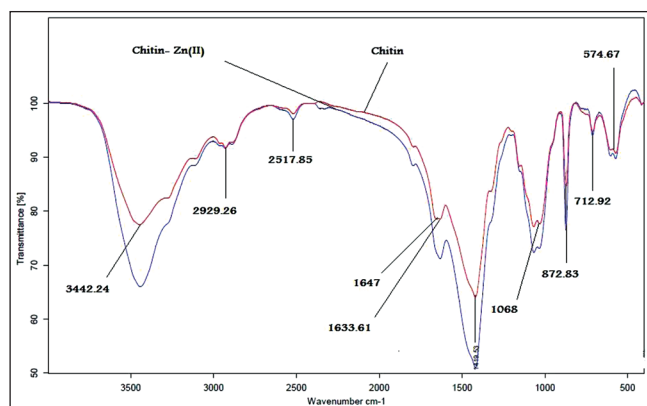| Table 1: Physico-chemical characteristics of chitin | |
|---|---|
| **Characterization** | **Chitin** |
| Yield (%) | 25.21 |
| $P_{zc}$ | 6.7 |
| Moisture (%) | 2.813 |
| Ash (%) | 0.8 |
| Water-binding capacities (%) | 452 |
| Fat-binding capacities (%) | 387 |
| Surface Area(m²/g) | 3.95 |



**Figure 2:** FTIR spectra of the chitin shrimp shells – Zn (II)
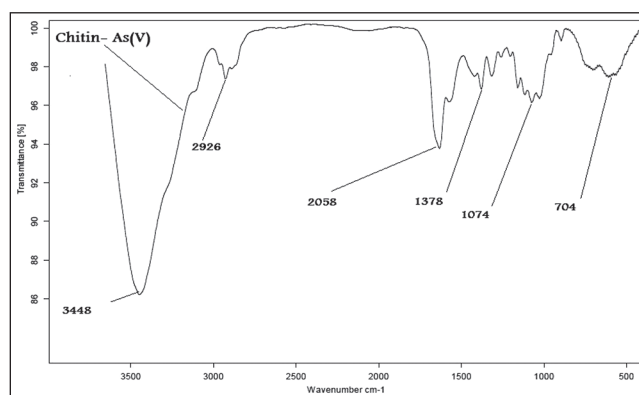


**Figure 3:** FTIR spectra of the chitin shrimp shells – As (V)

effect of contact time on removal of arsenate and zinc. According to this figure the rate of sorption of arsenate and zinc on chitin was rapid, with 40-50% of arsenate and zinc adsorption occurring within the first 30 min and attaining equilibrium were at 120 and 180 min for arsenate and zinc, respectively.

Figure 7 results demonstrate that at a fixed adsorbent dose, the amount adsorbed increased with increasing concentration of solution, but the percentage of adsorption decreased.

### Biosorption equilibrium and kinetics

The calculated results of biosorption of zinc and arsenate on the chitin as a function of the initial concentration of zinc and arsenate are shown in Table 2. It is found that the biosorption of zinc and arsenate on the chitin were correlated well with the Freundlich equation as compared with Langmuir equation under the concentration range studied. Table 3 indicates that the pseudo-second-order

and pseudo-first-order kinetic equations provided the best model for describing the adsorption of the zinc and arsenate on chitin, respectively.

### Comparison with other adsorbents

The maximum adsorption capacities obtained in this study were compared to reports of other adsorbents and are presented in Table 4. According to this table chitins have higher efficiency comparative to other adsorbent. But due to variable experimental condition a direct comparison between different adsorbents cannot be made.

## DISCUSSION

Based on the results of Table 1, chitin was obtained from shrimp shells with a yield of 24.5%. According to Muzzarelli,[17] shrimp shells consist mainly of chitin, protein/caroteins, and calcium carbonate with an average composition of 25, 35, and 40% by weight, respectively. This implies that the production of chitin was high (approximately 98%) on the laboratory scale. On the other hand, the results show similarities with the moisture and ash content of chitin obtained from different sources (Artemia, crab).[18] The FTIR spectrum of chitin also confirmed the presence of chitin in the shell of shrimp. Also, the FTIR spectra of chitin–metallic ion show changes in the intensity of the adsorption bands at 3442 and 1419 cm$^{-1}$, attributable to $CH_3$, –OH and $–NH_2$ groups. These changes



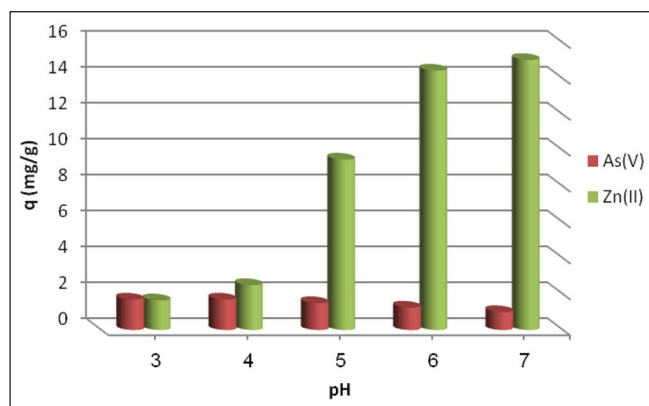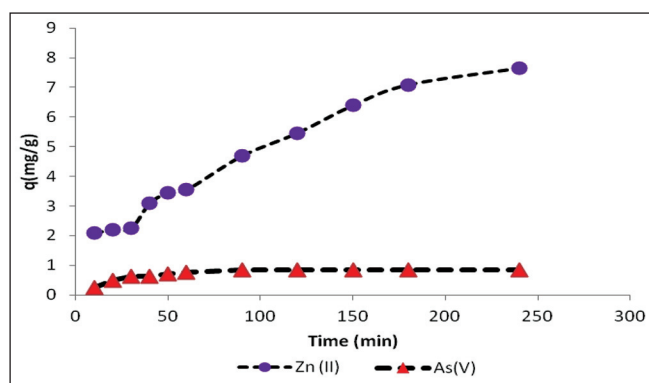**Figure 4:** Influence of solution pH on As (V) and Zn (II) uptake by chitin



**Figure 5:** Influence of adsorbent dose on As (V) and Zn (II) uptake by chitin



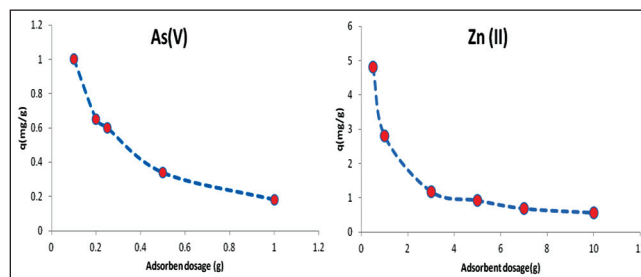**Figure 6:** Effect of contact time on the removal of As (V) and Zn (II) by chitin
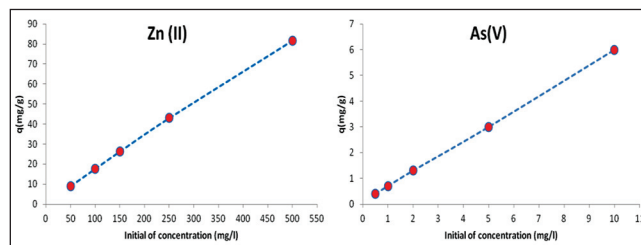


**Figure 7:** Influence of Initial metal concentration on As (V) and Zn (II) uptake by chitin

### Table 2: Parameters of Langmuir and Freundlich isotherms for adsorption of Zn (II) and As (V) on chitin

| Metal | Freundlich parameters | | | Longmuir constants | | | |
|---|---|---|---|---|---|---|---|
| | $K_f$ | $n$ | $R^2$ | $q_{max}$ | $R_L$ | b | $R^2$ |
| Zn (II) | 0.9664 | 1.15446 | 0.9992 | 270.27 | 0.52-0.915 | 0.00247 | 0.9783 |
| As (V) | 1.8932 | 1.3601 | 0.9861 | 11.574 | 0.3093-0.8995 | 0.2233 | 0.6337 |

indicate complex formation, which decreases the energies of the bonds due to the metallic ions adsorbed. Similar observations were reported by Kousalya *et al.*,[19] while studying the removal of copper using chitosan derivatives. Moreover, Kalyani *et al.*[20] studied the competitive adsorption of Cu (II), Co (II) and Ni (II) from aqueous solutions using chitosan/perlite composites. Based on Fourier transform infrared (FTIR) spectra, Kalyani *et al.* found that amine and hydroxyl groups were the main active sites for heavy metal ions.

In this research, in order to find out the equilibrium pH for zinc and arsenate adsorption, batch experiments were conducted over a pH range of 3 to 7 using a chitin adsorbent. As shown in Figure 4, the adsorption capacities were very high for arsenate at pH< 5. At lower pH, more protons were present and protonated the amino groups in chitin, resulting in increasing the electrostatic attractions between the arsenate and the positively charged active sites. However, at pH >5, the adsorption capacity decreased tremendously due to the decrease in the protonation of the active sites. This conclusion is also supported by the work of Choong.[21] But in the case of zinc ions, the adsorption amount increased with an increase of pH of the solution in the range 4-7. This could be explained that at low pH, amine groups in the chitin easily form protonation that induced an electrostatic repulsion of metal ions. Moreover, the $H^+$ ions in the solution at lower pH will compete with metal ions for the active sites ($-NH_2$), thereby reducing the adsorption capacity. With increasing pH, more ligands, such as amino and carboxyl

groups, on sorbent are exposed and thus negative charges result and attraction between these negative charges and the metals increases the biosorption capacity on the cell surface. Rocha *et al.*[22] investigated Zn (II) removal from industrial effluents using rice straw. They reported that the maximum Zn (II) removal occurred at pH 5.0. In another study, Freitas *et al.*[23] studied Zn (II) adsorption from aqueous solutions using brown marine macro algae. The author found that the maximum metal removal of 34.1 mg/g occurred at pH 5.0.

The data obtained from the biosorption of zinc and arsenate ions on the chitin powder showed that the amount of zinc and arsenate adsorbed varied with varying adsorbent concentration. The amount removal efficiency of zinc and arsenate increases with an increase in adsorbent concentration. The increase in the adsorption of the amount of solute is obvious due to increasing chitin surface area. Kundu *et al.*[24] reported that about 15 g/l of Portland cement was needed for the removal of 95% of arsenate with an initial concentration of 200 µg/l. Moreover, in this study the experimental data shown in Figure 7 indicate that the adsorption capacity increases with increase in initial metal ion concentration for metal on the chitin but the percentage adsorption of metal ion decreases with increase in metal ion concentration. This increase is a result of increase in the driving forces, that is, concentration gradient. Though an increase in metal uptake was observed, the decrease in percentage adsorption may be attributed to lack of sufficient surface area to accommodate much more metal available in the solution. Sdiri *et al.*[25] obtained the same results and showed the amount of zinc adsorbed increased with increasing initial metal concentration until the saturation of available adsorption site. Moreover, Salameh *et al.*[26] showed that the adsorption of arsenate on charred dolomite increased when the initial metal concentration was higher than 50 µg/l. Also, this finding is consistent with results reported in previous studies, for example, King *et al.*[27]

To establish a suitable contact time between the chitin and metallic ion solution, adsorption capacities of metal ion were

**Table 3: Pseudo-first-order and pseudo-second-order kinetic constants for adsorption of Zn (II) and As(V) on chitin**

| Metal | $q_e$(exp) mg/g | Pseudo-first-order kinetics | | | Pseudo-second-order kinetics | | |
|---|---|---|---|---|---|---|---|
| | | $K_1(10^{-4})$ | $q_e$ | $R^2$ | $K_2(10^{-4})$ | $q_e$ | $R^2$ |
| Zn (II) | 7.64 | 149 | 10.01 | 0.9577 | 5.4 | 12.562 | 0.9815 |
| As (V) | 0.850 | 329.3 | 0.737 | 0.9581 | 338.2 | 1.1282 | 0.9519 |

**Table 4: Comparison of maximum capacity of Chitin for zinc and arsenate with other adsorbents**

| Metal | Adsorbent | Uptake (mg/g) | Isotherm model | pH |
|---|---|---|---|---|
| Zn(II) | Bentonite[11] | 68.4931 | Freundlich | 6.76 |
| | Red mud[12] | 12.59 | Freundlich | 7 |
| | Amberlite IRC-718[13] | 156.89 | — | 6 |
| | Lewatit TP-207[13] | 89.56 | — | 5 |
| | Powered waste sludge[13] | 168 | — | 5 |
| | Neem bark[13] | 137.67 | Langmuir | 4 |
| | Neem leaves[13] | 147.08 | Langmuir | 5 |
| | Zinc oxide nanoparticle[14] | 357 | Langmuir | — |
| | Gn-MNPs[15] | 24.3 | Langmuir and Freundlich | 7 |
| | Chitin[This study] | 270.270 | Freundlich | 7 |
| As(V) | Rice polish[16] | 0.147 | — | 4.5 |
| | Tea fungal biomass[16] | 0.31 | — | — |
| | Bone char[16] | 1.43 | — | — |
| | Bauxsol[16] | 1.081 | — | — |
| | Kaolinite[16] | 0.23 | — | — |
| | Red mud[16] | 0.514 | — | — |
| | Chitin[This study] | 11.574 | Freundlich | 4 |

measured as a function of time [Figure 6]. The plot exhibits that the value of the percentage of metal ion removal is higher at the beginning. That is probably due to the presence of amine and carboxylic functional groups and larger surface area of chitin that are more available at the beginning for the adsorption of metals. As the surface adsorption sites become exhausted, the uptake rate is controlled by the rate at which the adsorbate is transported from the exterior to the interior sites of the adsorbent particles. The rate of arsenate and zinc removal was attained after approximately 2 and 3 h stirring, respectively. Equilibrium contact time from Benavente *et al*.[17] study for zinc removal by chitosan obtained was 180 minutes. And in study of Bhattacharya *et al*.,[28] contact time for zinc adsorbed by Rice husk ash was 180 minutes. The equilibrium results from Choong [21] study showed that the equilibrium contact time of arsenate adsorption (at a $C_0$ of 9.3 mg/l, contact time of 180 min) by crab shell was 120 minutes.

The results obtained from investigation of the equilibrium isotherm showed that the $R^2$ of the Freundlich isotherm was greater than that of the Langmuir isotherm for the adsorption of both investigated metal ions. The maximum predictable adsorption capacity of zinc and arsenate were 270.27 and 11.574 mg/g on chitin, respectively. These results are in agreement with study of Mishra *et al*.[29] Other studies, however, have found that the equilibrium data for these metallic ions are best described by the Langmuir isotherm when chitosan is used, meaning that different factors, such as physical form, can affect the adsorption onto the adsorbent.[30] To describe the adsorption behavior and rate, the data obtained from adsorption kinetic experiments were evaluated using pseudo-first and pseudo-second-order reaction rate models. Summary of experimental results of the two models fitted to the selected adsorption models that are shown in Table 3. As shown in Table 3, higher values of $R^2$ were obtained for pseudo-second-order than pseudo-first-order adsorption rate models, indicating that the adsorption rates of zinc onto the chitin can be more appropriately described using the pseudo-second-order rate rather than pseudo-first-order rate. Many studies reported the first-order equation of Lagergren does not fit well to the initial stages of the adsorption processes. The first-order kinetic process has been used for reversible reaction with an equilibrium being established between liquid and solid phases. Whereas, the second-order kinetic model assumes that the rate-limiting step may be chemical adsorption. In many cases, the second-order equation correlates well to the adsorption studies.[31,32] In the case of metalloids arsenate, the results reveal that there is a good correlation between experimental data and pseudo-first-order model. Similar results were observed by Gerente *et al*.[33]

## CONCLUSIONS

The present study shows that the chitin extracted of shrimp shells was an effective biosorbent for the biosorption of zinc and arsenate ions from an aqueous solution. The biosorption performances are strongly affected by parameters such as pH, initial concentration, chitin dosage and contact time. Adsorption study suggested that adsorption of Zn (II) and As (V) ions onto chitin followed Freundlich isotherm, with the corresponding maximum adsorption capacities 270.27 and 11.574 mg/g, respectively. Kinetic studies show that experimental data can be described by the pseudo-second-order and pseudo-first-order kinetic for the adsorption of zinc and arsenate on chitin, respectively. FTIR spectra of these adsorbent indicated that $-NH_2$, $-OH$ and $-COOH$ groups were all involved in the adsorption process. Additionally, based on these finding, it is concluded that this chitin adsorbent is relatively more effective for the removal of arsenate and zinc in batch condition.

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