



Isotherm and Kinetics of As(III) Uptake from Aqueous Solution by *Cinnamomum zeylanicum*

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Abstract

Cinnamon-bark (*Cinnamomum zeylanicum*) powder (CP) was investigated to evaluate the As(III) biosorption capability from aqueous solution. Biosorption behavior of As(III) was studied by batch column experiments. The adsorption phenomenon were analyzed with various experimental variables such as solution pH, sorbent amount, agitation speed, initial As(III) concentration and temperature. Maximum sorption was occurred at pH 5.0 while the equilibrium was established in 8 h. Langmuir and Freundlich isotherm models were employed for fitting the experimental data. The maximum sorption capacity of CP was observed to be 36.311 $\mu\text{g g}^{-1}$. The biosorption kinetics was speculated to follow pseudo-first-order kinetic model ($R^2 = 0.998$) with the sorption rate of $15.340 \times 10^{-3} \text{ min}^{-1}$ for the initial As(III) concentration of 500 $\mu\text{g L}^{-1}$. The biosorbent material was characterized by scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR). The activation energy (E_a) and heat of biosorption (ΔH) were calculated to be 17.780 and 30.724 kJ mol^{-1} respectively. Thermodynamic parameters were evaluated which revealed the spontaneous nature of biosorption on CP accompanied with the physical activated process. The break through characteristics of *C. zeylanicum* packed column was also investigated by small scale column tests (SSCT).

Keywords: *Cinnamomum zeylanicum*, arsenic, biosorption, FTIR, Freundlich isotherm, SEM.

Introduction

Heavy metal pollution of surface and ground water is considered to be one of the most common environmental threats, since the toxic ions dissolved can eventually reach the top of the food chain and thus become a risk factor for human health¹. Arsenic (As) is the twentieth most abundant element in earth's crust which was considered an ultra-trace nutrient and its deficiency can result in inhibited growth, but it becomes a potential toxin to plants, animals, and human beings when intake just exceeds the necessary level². Recently arsenic contamination in ground water has been emerged as a major problem of catastrophic proportions³. The scale of the arsenic problem is most serious in the alluvial and deltaic aquifer of Bangladesh and West Bengal (India), where millions of people drink water with high levels of arsenic⁴. The current regulation of drinking water standard has become more stringent and requires the arsenic content to be reduced to a few parts per billion (10 $\mu\text{g L}^{-1}$, WHO, US-EPA). However, many countries have been kept the earlier WHO guideline of 50 $\mu\text{g L}^{-1}$ as their standard, including Argentina, Bangladesh and China. Arsenic exists in both organic and inorganic forms in ecosystems, and it occurs in the environment, in four oxidation states (+V, arsenate; +III, arsenite; 0, arsenic; and -III, arsine)⁵. Arsenic is undoubtedly detrimental to human beings and animals, leading to several neurological, dermatological, gastrointestinal, cardiorenal diseases, and carcinogenic problems⁶.

Numerous treatment methods (e.g., solvent extraction, chemical precipitation as synthetic coagulants, ferric-hydrate precipitation, iron co-precipitation, ion exchange and reverse osmosis) so far been reported to be found capable of remediation of arsenic content from drinking water. However, plant-based products, particularly those containing cellulose showed potential metal biosorption capability⁷. Various type of biosorbents like chicken fat, coconut fiber, charcoal, Atlantic cold fish scales, chitosan-coated biosorbent, modified sugarcane bagasse, pine leaves, rice polish, rice husk, maize (*Zea mays*) leaves etc. were reported for lessening arsenic from drinking water⁸⁻¹².

In our previous papers we reported the arsenic removal by jute stick powder (JSP), jute leaf powder (JLP), sugarcane powder (SP), fish ayes powder (FAP), dheki vegetable powder (DVP), fern plant powder (FPP) and water hyacinth root powder (WHRP), neem leaf powder, tea leaf powder, mushroom, lemon peel and lemon segments^{13,14}. In this study, we investigated arsenic biosorption efficiency of Ceylon cinnamon (*Cinnamomum zeylanicum*), locally known as "daruchini" a commonly used spice in most Indian cuisines. We believe that this technique could offer an alternative way of As(III) lessening from contaminated drinking water in rural areas, where peoples are not well skilled to handle the other sophisticated arsenic removal technologies.

Material and Methods

Material Development: Fresh *C. zeylanicum* barks were collected from the Faculty of Forestry of Shahjalal university campus, Sylhet, Bangladesh (24.8917°N: 91.8833°E). Later, barks were washed with distilled water followed by subsequent drying and oven dried (Miken Instruments, India) at 80°C for 6 h to remove the moistures located inside the pores of leaves. The dried barks were boiled in hot distilled water for 2 h to remove the coloring components for ensuring clear water. The samples were then dried and finely crushed manually followed by washing with 0.001 M HCl to remove any residual precipitate salt inside the pores¹⁵. Later the materials were repeatedly washed with doubly distilled water (DDW) to remove all traces of acids and dried at 40°C for 72 h to volatilize any organic impurities. Finally, the bark powder was sieved in the particle size ranging from 200–250 μm. This biosorbent material was stored in the desiccator for further analysis. The surface morphology was observed by scanning electron microscope (SEM; FEI Company Quanta400, Japan) and the surface functional groups were analyzed by Fourier transform infrared spectroscopy (FTIR; Shimadzu prestige-21 FTIR Spectrophotometer, Japan).

Stock Solutions and Standards: All chemicals and reagents were of analytical grade and purchased from BDH (England), Merck (Germany) and Kanto Chemical Co. (Japan), and were used without any further purification. Standard As(III) stock solution was prepared by dissolving 1.320 g arsenic trioxide (As₂O₃) in 10 mL of DDW containing 4 g NaOH followed by a gentle heating at 50°C. Desired amount of DDW was added to make the total volume up to 1000 mL. This solution is likely to be stable under ambient condition. Different concentrations ranged from 100–500 μg L⁻¹ were prepared by proper dilution with DDW.

Analysis Method: The arsenic concentration was determined by standard silver diethyldithiocarbamate (SDDC) method followed by an UV-Visible spectroscopy¹⁶. The detection limit of arsenic for SDDC method ranging from 5–250 μg L⁻¹ (ASTM D 2972: 2008) which also meet USEPA and WHO limit of drinking water. A homemade U-like arsine generator consists of a three neck round bottom flask especially prepared by quick fit apparatus was used for this experiment. The arsine (AsH₃) generated in the flask was passed through glass wool impregnated with lead acetate (CH₃COO)₂Pb, to absorb any hydrogen sulfide (H₂S), which may be evolved from the sample, and then the arsine gas bubbled into the SDDC solution which slowly turned into reddish color and the respective absorbance was measured by UV-Visible spectrophotometer (UV-1650 PC, Shimadzu, Japan) at 535 nm against reagent blank.

Biosorption Experiments: The batch experiments were performed in a conical flask placed on an orbital shaker containing 100 mL of As(III) solution having the biosorbent dosage ranging from 4.0–40 g L⁻¹ at room temperature (25±1 °C). The solution pH was pre-adjusted at 5.0 using 0.5 M HCl

or 0.1 M NaOH (pHs -25, REX, CHINA) before shaking at the speed of 150 rpm for 8 h. After the equilibrium become established, the biosorbent was separated by filtration through 0.8 μm cellulose acetate membrane (Whatman), then the equilibrium concentration of As(III) ions in the filtrate was determined. The amount of As(III) adsorbed was calculated from the difference between the initial and equilibrium concentration. The concentrations were measured thrice in each experimental run. Small scale column tests (SSCT) were conducted with a down flow column system containing a sandwiched bed supported by normal sand which was washed with 1.0 M HNO₃ for 24 h before installment.

Adsorption Isotherms and Kinetics: Adsorption isotherms were studied in five 250 mL Erlenmeyer flask containing 100 mL of As(III) solutions of different initial concentrations (100, 200, 300, 400 and 500 μg L⁻¹) and the pH was adjusted at 5.0. All of the vessels were agitated at the speed of 150 rpm for 8 h. Afterwards, the solutions were filtered and analyzed to determine the amount of arsenic adsorbed on biosorbent according to equation 1. Langmuir (equation 2), Freundlich (equation 3) isotherms were plotted and the respective parameters were evaluated. Separation factors (R_L) for different initial concentration were also calculated employing the equation 4.

$$q_e = \frac{V(C_o - C_e)}{1000W} \quad (1)$$

$$\frac{1}{q_e} = \frac{1}{QbC_e} + \frac{1}{Q} \quad (2)$$

$$\log q_e = \log k_f + \frac{1}{n} \log C_e \quad (3)$$

$$R_L = \frac{1}{1 + bC_o} \quad (4)$$

In the same way, kinetic studies were performed by using biosorbent dose of 20.0 g L⁻¹ at pH 5.0. The agitation speed was maintained at 150 rpm and different temperature was also controlled. The samples at different time intervals (30–180 min) were pipette out and analyzed. Then, pseudo-first-order (equation 5) and pseudo-second-order (equation 6) kinetic models were fitted with the experimental outcome. The corresponding parameters were also calculated.

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (5)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (6)$$

Thermodynamic parameters like activation energy, Gibbs free energy, entropy and heat of adsorption were evaluated by equation 7, 8 and 9 respectively as follows.

$$\ln k = \ln k_o - \frac{E_a}{R_g T} \quad (7)$$

$$\Delta G = -R_g T \ln K_e \quad (8)$$

$$\ln K_e = \frac{\Delta S}{R_g} - \frac{\Delta H}{R_g T} \quad (9)$$

Results and Discussion

Characterization of Biosorbent by SEM and FTIR: Figure 1 shows the scanning electron microscopic (SEM) images of *C. zeylanicum* bark powder (CP) at different magnifications which indicates the particle sizes ranging from 10–20 μm . The surface microstructure suggested the fibrous and porous morphology. To detect the main organic functional groups of CP surface and the possible interaction with As(III) ions, a Fourier transform infrared (FTIR) analysis was conducted in solid phase KBr disk method. The comparative FTIR spectra of unloaded and As(III) loaded CP is shown in figure 2. A broad and strong band ranging from 3000 to 3600 cm^{-1} observed due to the overlapping of –OH and –NH₂ stretching vibrations. This observation is also accorded with existence of the peaks at 1025 and 1153 cm^{-1} for C–O and C–N stretching vibrations¹⁷. The peak observed at near 2926 cm^{-1} could be related to the existence of C–H groups. Bands around 1612 cm^{-1} are indicative of carboxylic group (C=O) whereas a shoulder peak at around 1733 cm^{-1} could be assigned for aldehyde group (–CHO). The FTIR spectra revealed that the carbons possess different surface structure, e.g. aliphatic, aromatic and cyclic as one can observe bands at 1445 cm^{-1} and at around 1260 to 1025 cm^{-1} . It is worthwhile to mention that most of the functional groups originally present on CP were intact with a marginal shifts of peak position, even after loaded with As(III).

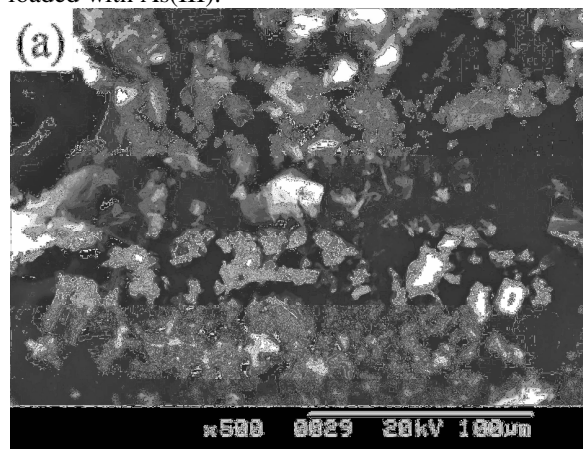


Figure-1a

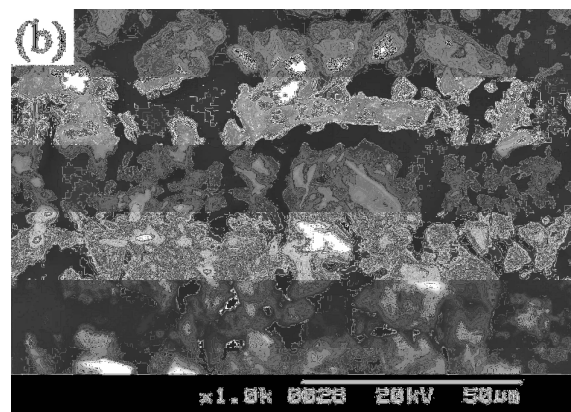


Figure-1b

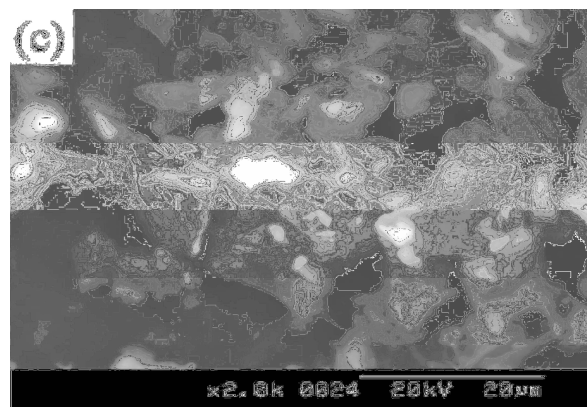


Figure-1c

SEM images of *C. zeylanicum* bark powder at different magnifications (a) 500 \times , (b) 1000 \times and (c) 2000 \times

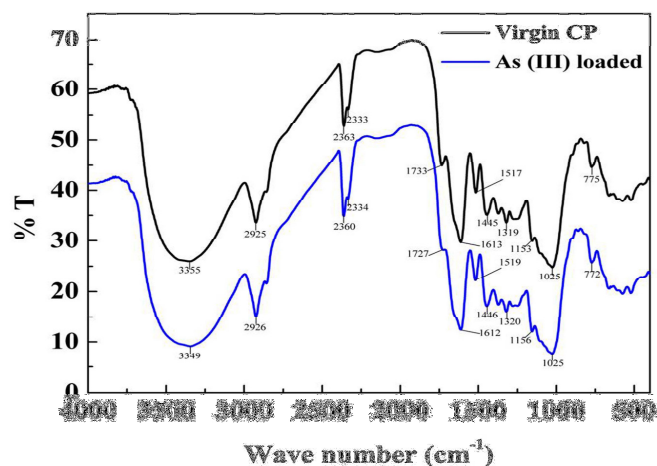


Figure-2

FTIR spectra of *C. zeylanicum* bark powder unloaded and loaded with As(III) ions

Effect of Adsorbent Dose: The effect of biosorbent dosage on As(III) sorption was investigated using the sorbent dosage from 4 to 40 g L^{-1} and presented in figure 3a. The initial concentration and pH of the solution were 500 $\mu\text{g L}^{-1}$ and 7.0 respectively. An initial quick increase in adsorption efficiency followed by a final stability with the further increase of biosorbent dose was observed. The adsorption efficiency has climbed up from 20.01% at a dose of 4 g L^{-1} to 46.50 % at 16 g L^{-1} . The sharp rising in removal efficiency might be related to the greater availability of the exchangeable sites or surface area at higher biosorbent dosage¹⁸. In the early stage of adsorption, the whole surface was uncovered and the adsorption capacity was sharply inclined from the initial value of 10.50 to 29.00 $\mu\text{g g}^{-1}$ when the sorbent dosage increased from 4 to 20 g L^{-1} . However, the sorbent dosage above 20 g L^{-1} , the sorption capacity was started to decrease because of the interference of binding sites and insufficiency of metal ions in solution with respect to available binding sites between biosorbent and biosorption medium¹⁹. The sorbent dosage of 20 g L^{-1} was considered as an optimized level of adsorbent and followed throughout the study.

Effect of pH: Figure 3b depicts the effect of the solution pH on As(III) sorption onto the CP. The sorption efficiency of As(III) under the pH range of 2.0–11.4 was studied for the 500 $\mu\text{g L}^{-1}$ As(III) solution at the biosorbent dose of 20 g L^{-1} . It is speculated that at lower pH, the functional groups ($-\text{COOH}$, $-\text{NH}_2$, $-\text{OH}$) might get positively charged ($-\text{COOH}_2^+$, $-\text{NH}_3^+$ and $-\text{OH}_2^+$). These positively charged functional groups can interact with the oxyanion of arsenic leading to effective adsorption²⁰. The maximum sorption efficiency of around 49.92% was found at pH 5.0, which was selected as an optimum pH condition for further experiments. On the other hand, at relatively higher pH, the surface of adsorbent would get negatively charged by larger size of OH^- ions resulting the increased diffusion hindrance for As(III) ions, leading to a decreased adsorption¹¹.

Effect of Agitation Speed: Figure 3c shows the effect of agitation speed on As(III) ion sorption on CP. At the agitation speed of around 150 rpm, maximum sorption took place (28 $\mu\text{g g}^{-1}$) which was considered as optimum agitation speed. It was believed that at lower agitation speed, the bark powder instead of spreading in the solution conglomerated that buried many active sites under the top layer of sorbent. It results the sorption only at the top layers and the under buried layers could not take part in the sorption process, since they had no contact with As(III) ions. On the other hand, at high agitation speed (>150 rpm) caused random collision between existing particles in the sorption vessel (sorbent-sorbent, sorbent-sorbate and sorbate-sorbate) and might not provide adequate time to the As(III) ions to make a possible binding with the surface of *C. zeylanicum* bark powder.

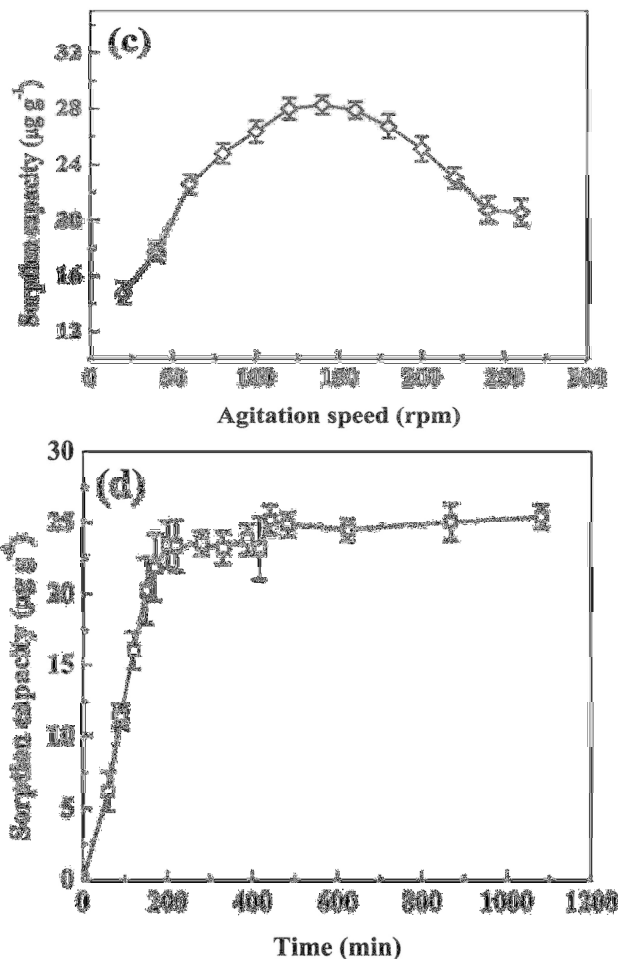
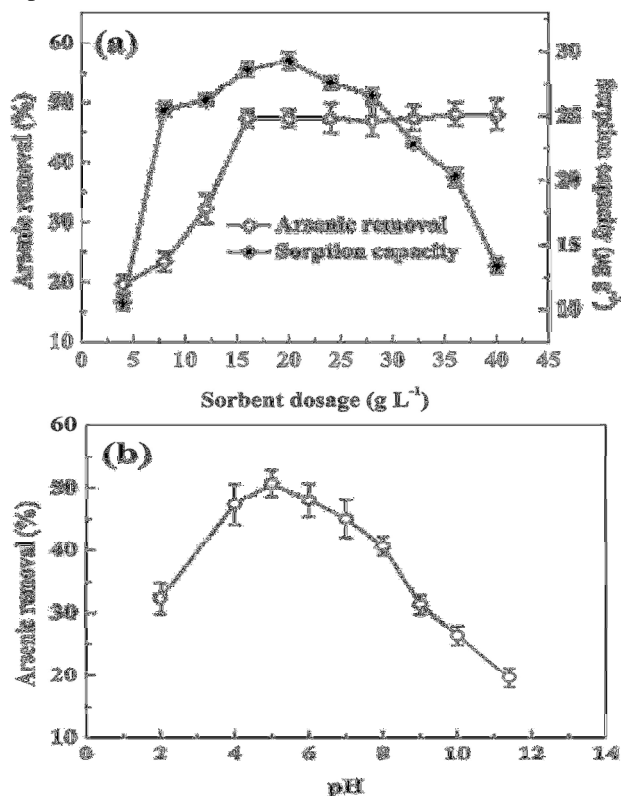


Figure-3
(a) Effect of biosorbent dose (b) solution pH and (c) agitation speed and (d) variation of As(III) sorption on *C. zeylanicum* bark powder with time (initial As(III) concentration: 500 $\mu\text{g L}^{-1}$; agitation speed: 150 rpm; temperature: $25 \pm 1^\circ\text{C}$)

Equilibrium Study: Sorption equilibrium studies were performed for 1000 mL of 500 $\mu\text{g L}^{-1}$ As(III) solution with the biosorbent dosage of 20 g L^{-1} at the agitation speed of 150 rpm. At different time interval, 20 mL of solution was pipette for analysis and the sorption capacity was plotted against time (figure 3d). It is clear from the figure 3d; the equilibrium was appeared to reach in 8 h. The sorption process was found to be rapid at the initial period of contact time then slowed down followed by an equilibrium establishment.

Adsorption Isotherms: Langmuir and Freundlich isotherm models were used to elucidate the adsorption characteristics of As(III) on CP. Firstly, Langmuir model was employed to establish the relationship between the amount of As(III) ions adsorbed (q_e) on biosorbent and its equilibrium concentration (C_e) in the aqueous solution based on the equation 2. Figure 4a represents the linearized fitting of biosorption data and the

evaluated parameters are listed in table 1. The correlation coefficient (R^2) of the value 0.994 implies the homogeneous distribution of As(III) ions on *C. zeylanicum* bark powder. The maximum adsorption capacity (Q) and Langmuir constant (b) were calculated to be $36.311 \mu\text{g g}^{-1}$ and $0.013 \text{ L } \mu\text{g}^{-1}$ respectively at room temperature. The separation factor, R_L , values found to be in the range of 0.137–0.442 which resembled the favorable process and the Langmuir isotherm was in its proper portrayal. The equilibrium data was also analyzed with Freundlich model using the linear logarithmic form of equation 3. Figure 4b shows the linear fitting of Freundlich model of

experimental data and the parameters were calculated and enlisted in table 1. The relative adsorption capacity, k_f ($1.418 \mu\text{g g}^{-1}$) and the empirical constant n (1.768) were calculated from the slope and intercept. The value of n ranging from 1–2 implies the moderate adsorption phenomenon. Both of these isotherm models were well fitted according to the correlation coefficients values listed in table 1. This observation proved homogeneous and heterogeneous distribution of trivalent arsenic on the surface of *C. zeylanicum* bark powder⁹.

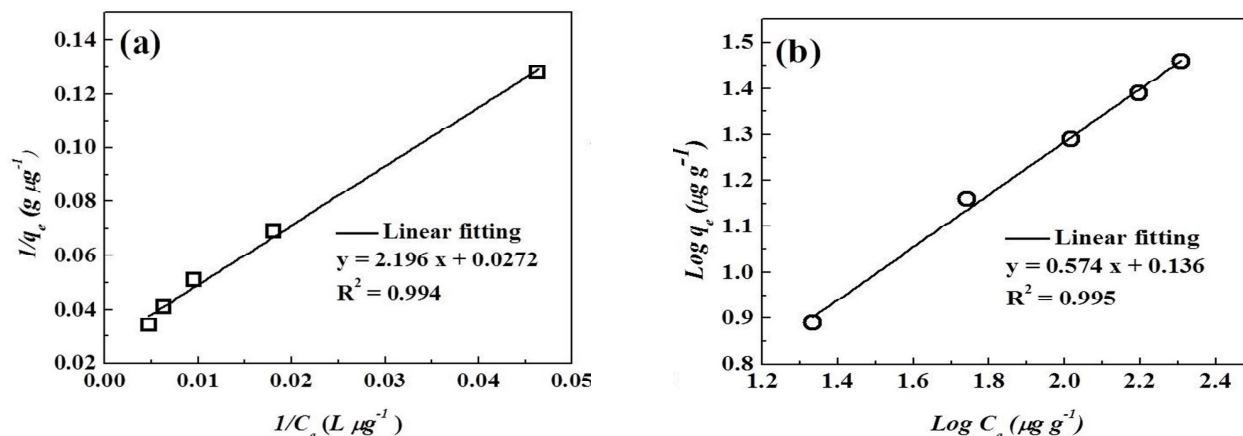


Figure-4

(a) Langmuir and (b) Freundlich isotherm of As(III) biosorption on *C. zeylanicum* bark powder (initial pH: 5.0; biosorbent content: 20 g L^{-1} ; agitation speed: 150 rpm; temperature: $25 \pm 1^\circ\text{C}$)

Table-1

Langmuir, Freundlich coefficients and separation factors for biosorption of As(III) at different initial concentration by *C. zeylanicum* bark powder

Initial concentration ($\mu\text{g L}^{-1}$)	R_L	Langmuir constants		Freundlich constants	
		Q ($\mu\text{g g}^{-1}$)	b ($\text{L } \mu\text{g}^{-1}$)	k_f (L g^{-1})	n
100	0.442	36.311	0.013	1.418	1.768
200	0.283				
300	0.209				
400	0.165				
500	0.137				

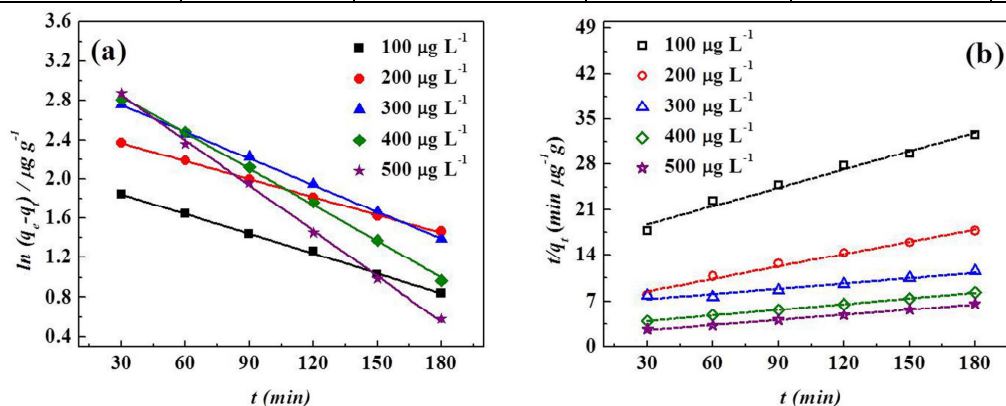


Figure-5

(a) Pseudo-first-order (b) pseudo-second-order kinetic model fitting for As(III) biosorption at different initial concentrations (initial pH: 5.0; biosorbent content: 20 g L^{-1} ; agitation speed: 150 rpm)

Table-2
Kinetic parameters obtained from pseudo-first-order and pseudo-second-order model for As(III) biosorption onto *C. zeylanicum* bark powder

Initial conc. C_0 , ($\mu\text{g/L}$)	Pseudo-first-order			Experimental, q_e ($\mu\text{g g}^{-1}$)	Pseudo-second-order		
	$k_1 \times 10^3$ (min^{-1})	Theoretical, q_e ($\mu\text{g g}^{-1}$)	R^2		$k_2 \times 10^4$ ($\text{g } \mu\text{g}^{-1} \text{min}^{-1}$)	Theoretical, q_e ($\mu\text{g g}^{-1}$)	R^2
100	6.742	7.771	0.999	7.842	3.810	12.191	0.996
200	6.112	12.792	0.999	14.474	6.020	15.862	0.988
300	9.081	20.622	0.999	19.579	1.100	37.449	0.951
400	12.280	24.745	0.998	24.264	2.720	34.586	0.999
500	15.340	27.488	0.998	29.106	4.020	37.840	0.997

Kinetic Study: Pseudo-first-order and pseudo-second-order kinetic models were employed to investigate the mechanism of sorption processes such as mass transfer and chemical reaction. The plots of both equations (equation 5 and 6) exhibited straight lines with very promising correlations, presented in figure 5. The slopes and intercepts were used to calculate the adsorption rate constant and theoretical adsorption capacity which were presented in table 2. However, data was more linked when examined with the first order kinetic model as depicted by high correlation values and closeness of the theoretical adsorption capacities with the experimental one. The rate constants and theoretical adsorption capacities were found to be dependent on the initial concentration of As(III) ions. The first order sorption rate constant was calculated to be $15.340 \times 10^{-3} \text{ min}^{-1}$ for the initial arsenic concentration of $500 \mu\text{g L}^{-1}$. Thus it could be conferred that As(III) adsorption on *C. zeylanicum* bark powder was kinetically controlled by pseudo-first-order model.

Thermodynamic Study: The activation energy (E_a) was obtained from the slope of the plot between $\ln k_1$ versus $1/T$ (figure 6a). The relatively lower activation energy of $17.780 \text{ kJ mol}^{-1}$ is attributed to the involvement of activated physical process for adsorption. The thermodynamic parameters of ΔH , ΔS were also calculated from the linear regression according to the equation 9. The Van't Hoff plot is shown in figure 6b and the obtained parameters are listed in table 3. The ΔH was observed to be $30.724 \text{ kJ mol}^{-1}$, which implies the endothermic nature of As(III) adsorption on *C. zeylanicum* bark powder. The Gibbs free energy change (ΔG) was also calculated according to the equation 8 which indicated the spontaneous nature of the sorption process²¹. In addition, the positive value of ΔS suggested an increase in randomness at the solid/liquid interface during biosorption of As(III) ions on CP.

Table-3

Thermodynamic parameters of the As(III) biosorption on *C. zeylanicum* bark powder at different temperatures

Arrhenius parameters		Thermodynamic parameters			
E_a (kJ mol^{-1})	ΔH (kJ mol^{-1})	ΔS	ΔG		
			296.5 K	301 K	306 K
17.780	30.724	105.420	-521.509	-1031.310	-1523.490

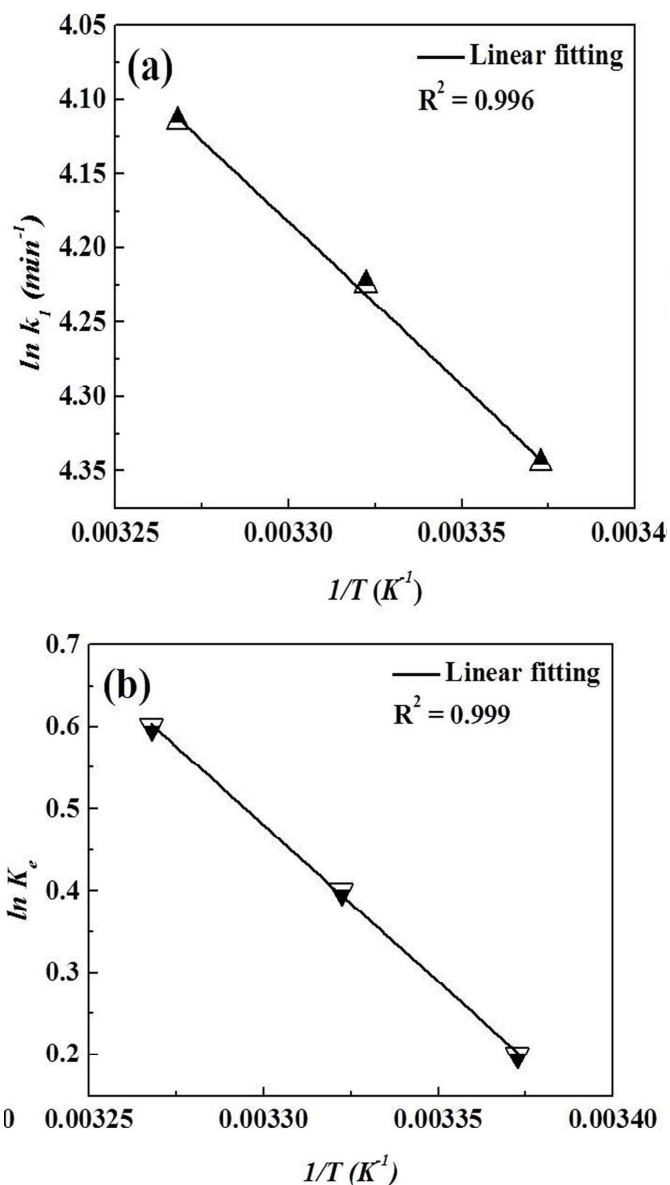


Figure-6
(a) Arrhenius and (b) Van't Hoff plot of As(III) biosorption at different temperatures (initial concentration: $500 \mu\text{g L}^{-1}$; initial pH: 5.0; biosorbent content: 20 g L^{-1})

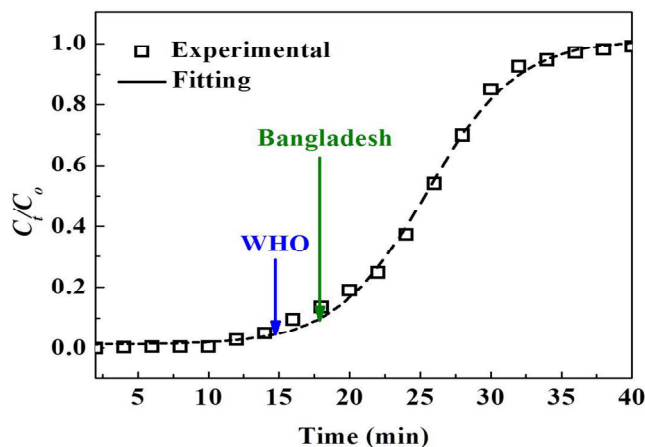


Figure-7

Experimental breakthrough curve for As(III) removal on *C. zeylanicum* bark powder bed by small scale column test (SSCT)

Small Scale Column Tests (SSCT): To investigate the removal efficiency of arsenite by CP, small scale column tests (SSCT) were conducted which consist of a glass column with 3.6 cm inner diameter loaded with 20 g of biosorbent and sand as a supporting layer^{8,22}. To eliminate the supporting materials contribution for As(III) removal, blank test was performed. Arsenic solution possessing the concentration of $500 \mu\text{g L}^{-1}$ was passed through the column in a downward direction at the flow rate of 20 mL min^{-1} . The pH of the influent water was 5.0. The effluents were analyzed for As(III) concentration at different time intervals. The breakthrough curve for As(III) from the column loaded with *C. zeylanicum* bark powder is shown in figure 7. The plot indicates that, arsenic concentration below the WHO standard ($10 \mu\text{g L}^{-1}$) was found in the effluent up to 15 min of continuous flowing. Whereas, the curve was broken down after 18 min of the continuous flow according to Bangladesh standard ($50 \mu\text{g L}^{-1}$). Thus, the SSCT shows the mini scale applicability of *C. zeylanicum* bark powder packed column to mitigate As(III) from aqueous solution.

Table-4

Comparison of adsorption capacity (Q , $\mu\text{g g}^{-1}$) of various biosorbent to remove As(III)

Sl No.	Biosorbent	Uptake capacity ($\mu\text{g g}^{-1}$)	pH	Reference
1	Human hair	10.00	7.0	²³
2	Atlantic Cod fish scale	26.67	4.0	²¹
3	Coconut charcoal	38.00	5.0	²⁴
5	Rice waste	41	6.8	²⁵
6	Saccharomyces cerevisiae	54.132	5.0	²⁶
7	Pyrolysed sewage sludge	71.00	3.0-3.5	²⁷
8	Rice polish	138.88	7.0	²¹
9	<i>C. zeylanicum</i> bark powder	36.311	5.0	Present study

Comparison with Other Biosorbents: Comparative adsorption capacities of *C. zeylanicum* bark powder with other homologous biosorbents to uptake As(III) are illustrated in table 4. It was observed that the As(III) remediation efficiency by CP is moderate in nature under the experimental condition compared to other literature values. It is also worthwhile to mention that the absolute value of arsenite uptake from aqueous solution is somewhat lower than that of arsenate. Therefore it could be concluded that this study gives an implication of using low cost biosorbent to mitigate arsenic from aqueous solution.

Conclusion

C. zeylanicum bark powder was investigated for As(III) mitigation from aqueous solution containing arsenic concentration from 100 to $500 \mu\text{g L}^{-1}$. The adsorption behaviors were thoroughly studied by Langmuir and Freundlich isotherms. The biosorption was observed to be the combination of homogeneous and heterogeneous processes. The adsorption kinetics was better described by pseudo-first-order kinetic model compare to pseudo-second-order model. The endothermic and spontaneous nature of adsorption was confirmed by thermodynamic study. Small scale column tests (SSCT) also revealed the feasibility of using this biosorbent under real situation. This paper demonstrates the use easily accessible biosorbent which is economical, effective and could be an alternative to more costly adsorbents without any adverse effect on human body.

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