# Arsenic(III) Remediation from Water using *Dillenia indica* Leaf Powder: Characterization, Kinetics and Isotherms

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Abstract—The present study uses the Dillenia indica leaf powder as adsorbents for adsorptive removal of As(III) from spiked distilled water by batch processes. The biosorbent of size 63-75 µm was characterised by specific surface area, anion exchange capacity and Fourier transform infrared spectroscopy. Rapid equilibrium is attained within 60 minutes following a second order kinetics. The application of Elovich suggested that the rate determining step may be chemisorption. The intra-particle diffusion and liquid film diffusion mechanism also plays a significant role in the adsorption of As(III) but is not the slowest step. Equilibrium sorption data showed good fit to all the three isotherms, Freundlich, Langmuir, and Temkin showing sorption to be monolayer on the heterogeneous surface of the biosorbent. The maximum sorption capacity was found to be 3.85 mg  $g^{-1}$ . All adsorption experiments were done at pH close to the pH of natural water with As(III) concentrations close to the concentrations at which they appear in contaminated ground water at 303 K. This potential of Dillenia indica leaf powder for groundwater and surface water remediation for As(III) removal can further be increased by acid-base activation.

## 1. INTRODUCTION

Arsenic is a poisonous heavy metal that is widely distributed in nature [1]. Arsenic exists in nature by both natural and anthropogenic activities. Natural activities like volcanic eruption, rocks erosion and forest fires and anthropogenic activities like industrial waste discharges, mining, combustion of fossil fuels, use of arsenic pesticides, herbicides, paper and pulp production, manufacturing of glass, semiconductors, wood preservatives and a number of applications [2]. Arsenic is found in more than 320 minerals, and arsenopyrite is the most common As mineral. The primary sources of arsenic in natural water include arsenic minerals, together with extensive use of insecticides, pesticides and herbicides containing arsenic [3]. In natural water arsenic exists in inorganic formsarsenite  $(AsO_3^{3-})$  and arsenate  $(AsO_4^{3-})$  referred to as As(III) and As(V) respectively. Generally, As(V) species predominate in oxygen rich aerobic conditions such as surface water while As(III) species predominate in moderately reducing anaerobic conditions such as groundwater [4]. Arsenite is 60 times more toxic than arsenate due to the absence of electrostatic

attraction, as compared to As(V) [5]. The pentavalent species i.e. arsenic acid (H<sub>3</sub>AsO<sub>4</sub>) are strongly bound to soil constituents, which can explain its lower mobility, while the trivalent species i.e. arsenous acid (H<sub>3</sub>AsO<sub>3</sub>) species are weakly bound to inorganic sorbents regardless of the pH [6]. Arsenic has been determined to be a class A human carcinogen by the US EPA and the WHO [7]. Long term ingestion of arsenic leads to cancer, skin lesions, respiratory, reproductive and neurological disorders etc. Inorganic arsenic can cross the membrane of placenta so it is teratogenic to animals. [8]. Arsenic poisoning has been reported in majority of the countries, with highest population at risk is in Bangladesh, followed by West Bengal in India [9]. Due to the severe health effects of arsenic ingestion, WHO and US EPA have allowed the maximum concentration of arsenic in drinking water is 10 µg/L [10].

There are several methods for arsenic decontamination from water among which adsorption is found superior for arsenic removal because it is cheap, easy to operate and does not form toxic substances [11,12]. To carry out adsorption, adsorbents are required and problem arises in terms of cost and efficiency of adsorbents. In this context, biosorbents or biomaterials are found to be excellent alternatives. Various biosorbents including agricultural waste and microorganisms have been reported for efficiently accumulating arsenic from water [13,14].

In the present work, *Dillenia indica* leaf powder (DILP) was used to remove As(III) from aqueous solution under different experimental conditions. The sorption capacity of DILP was compared with other biosorbents already used for aqueous removal of arsenite.

## 2. MATERIALS AND METHOD

## 2.1 Biosorbent preparation

Mature leaves of the *Dillenia indica* were collected from the Botanical garden of Gauhati University. The leaves were

thoroughly washed to remove dust and other impurities. The leaves were first sun dried and then dried in an oven at around 343K for few hours till they became crisp. These were then crushed in a grinder to obtain leaf powder, followed by several washings till the washings were free of plant pigments. The washed leaf powder was again dried in an oven for several hours up to 343K till water is removed completely. Lumps were ground in a mortar and the powder was sieved through a 75  $\mu$ m and then by 63  $\mu$ m sieve to obtain particles of uniform size, which were preserved in clean plastic containers and kept in dessicator for carrying out adsorptions.

## 2.2 Characterization of biosorbents

**2.2.1 FTIR study.** The adsorbents were characterized with FTIR using KBr pallet (IR Affinity-1 Fourier Transform Infrared Spectrophotometer, Shimadzu, range 4000 - 400 cm<sup>-1</sup>).

**2.2.2 Specific surface area.** The specific surface area (S in  $m^2$  g<sup>-1</sup>) of the biosorbents was determined by the methylene blue adsorption method by using following equation:

$$\mathbf{S} = (\mathbf{q}_{\mathrm{m}} \cdot \mathbf{N}_{\mathrm{A}} \cdot \mathbf{A}) / \mathbf{M}$$
(1)

where,  $q_m (mg g^{-1})$  is the maximum adsorption capacity which can be obtained from Langmuir model,  $N_A$  is the Avogadro's number (6.023x10<sup>23</sup>mol<sup>-1</sup>), A is the surface occupied by a molecule of Methylene Blue (taken as 130 Å<sup>2</sup>) and M is the molecular weight of Methylene Blue (319.86 g mol<sup>-1</sup>) [15,16].

**2.2.3 Anion exchange capacity.** Anion exchange capacity (AEC) of the biosorbents was determined by conductometric method. AEC was calculated using the formula,

$$AEC = NV/W$$
 (2)

where N is the normality of  $AgNO_3$  solution and V is its volume required by W g of anion exchanger [17].

## 2.3 Preparation of As (III) solution

A stock of 1000 ppm sodium arsenite solution was prepared by dissolving the required amount of NaAsO<sub>2</sub> in double distilled water. Solutions of desired concentrations were prepared from the stock solution in its natural pH. Sodium arsenite AR (Loba Chemie, Mumbai) was used without further purification and obtained as sealed from the chemical store.

## 2.4 Adsorption experiments

The adsorption experiments were carried out in 100 ml Erlenmeyer flasks by mixing a fixed amount of biosorbents with 50 ml of aqueous As (III) solution. The mixture was agitated in a thermostatic water bath (NSW, Mumbai, India) for a pre-determined time interval. The mixture was centrifuged for 15 min (Eltec centrifuge, TC4100 ~ 8000 rpm) and As (III) remaining unadsorbed in the supernatant liquid was determined with the help of an atomic absorption spectrophotometer (Varian SpectrAA 220). Before the actual

experiments, sets of blank experiments were done to find if there was any adsorption on the walls of the container.

## 2.5 Adsorption kinetics

The adsorption rate processes are tested with a few well known kinetic models to understand the possible mechanism.

**2.5.1 Pseudo-first order model.** The first-order Lagergren model (Lagergren, 1898) [18] given by the equation,

$$\log (q_e - q_t) = \log q_e - k_1 t/2.303$$
(3)

which shows how  $q_t$  the amount of As(III) adsorbed per unit mass of adsorbent at time t and  $q_e$  is the amount adsorbed per unit mass at equilibrium,  $k_1$  being the first order rate coefficient. The values of  $k_1$  and  $q_e$  are obtained by a plot of log  $(q_e - q_t)$  vs. t.

**2.5.2 Pseudo-second order model.** The second order kinetics is given by the rate equation,

$$t/q_t = 1/(k_2 q_e^2) + (1/q_e) t$$
 (4)

where  $k_2$  is the rate constant (g mg<sup>-1</sup> min<sup>-1</sup>).  $k_2$  and  $q_e$  values are obtained by slope and intercept of a plot of  $t/q_t$  vs. t [19,20].

**2.5.3 Elovich equation**. The linear form of Elovich equation is,

$$q_{t} = (1/\beta) \ln (\alpha\beta) + (1/\beta) \ln t$$
(5)

where,  $\alpha$  (mg g<sup>-1</sup> min<sup>-1</sup>) represents the rate of chemisorption at zero coverage and  $\beta$  (g mg<sup>-1</sup>) is the desorption constant related to the extent of the surface coverage and activation energy for chemisorption. A plot of q<sub>t</sub> against ln t is linear when the Elovich equation is obeyed [21].

2.5.4 Intra-particle diffusion. The intra-particle diffusion model is given by the equation :

$$q_t = k_i t^{0.5}$$
 (6)

where,  $k_i \text{ (mg g}^{-1} \text{ min}^{-1/2}\text{)}$  is the intra-particle diffusion rate coefficient. One significant feature of the plot is that it has zero intercept [22].

**2.5.5 Liquid film Diffusion Model.** Boyd et al. (1949) [23] suggested the following equation:

$$\ln\left(1-F\right) = -kt\tag{7}$$

where F is the fractional attainment of equilibrium and F=  $q_t/q_e$ , k is the adsorption rate constant. A linear plot of  $-\ln(1 - F)$  vs. t with zero intercept is a clear indication that the adsorption process is controlled by liquid film diffusion.

#### 2.6 Adsorption isotherms

Adsorption can be quantified by isotherm equations. In the present study, Langmuir, Freundlich and Temkin isotherms were applied to the experimental data.

**2.6.1 Freundlich isotherm.** The Freundlich (Freundlich, 1906) [24] isotherm equation is given as follows:

$$\log q_e = 1/n \log C_e + \log K_f \tag{8}$$

where  $C_e$  is the equilibrium concentration of As(III) (mg L<sup>-1</sup>);  $q_e$  is the amount of the As(III) adsorbed per unit mass (mg g<sup>-1</sup>),  $K_f$  (mg<sup>1-1/n</sup> L<sup>1/n</sup> g<sup>-1</sup>) and n are Freundlich coefficients related to adsorption capacity and adsorption intensity, respectively. It is generally accepted that the reciprocal Freundlich intensity (1/n) is < 1.0 (i.e., 1 < n < 10) for favorable adsorption.

**2.6.2 Langmuir isotherm.** The Langmuir (Langmuir, 1916) [25] isotherm equation is given as follows:

$$C_e/q_e = 1/(bq_m) + (1/q_m) C_e$$
 (9)

 $q_m(mg g^{-1})$  and b (L mg<sup>-1</sup>) are Langmuir coefficients related to adsorption efficiency and energy of adsorption respectively.

**2.6.3 Temkin isotherm.** The Temkin (Temkin and Pyzhev, 1940) [26] isotherm is,

$$q_e = 2.303 B \log k_T + 2.303 B \log C_e$$
 (10)

where, B = (RT/b), with  $k_T (L mg^{-1})$  corresponding to maximum binding energy and b (J mol<sup>-1</sup>) and B the Temkin coefficients. The dimensionless constant, B, is related to the heat of adsorption.

### 3. RESULTS AND DISCUSSIONS

#### 3.1 Characterization of adsorbent

**3.1.1 Specific surface area and Anion exchange capacity.** The Specific surface area and Anion exchange capacity of the OLP is found to be  $491 \text{ m}^2 \text{ g}^{-1}$  and  $9.85 \text{ meq } 100 \text{ g}^{-1}$ .

# 3.2.2 FTIR study.



Fig. 1: FTIR spectra of OLP before adsorption

A sharp peak at 667 cm<sup>-1</sup>, 1647 cm<sup>-1</sup> and 2924 cm<sup>-1</sup> indicates the presence of C-Cl, aliphatic alkene C=C and -CH<sub>2</sub>- (asym. stretch) groups respectively. A broad band at 3417 cm<sup>-1</sup> between 3350-3450 cm<sup>-1</sup> indicates the presence of -OH group. The absorbance at 1064 cm<sup>-1</sup> (1000-1200 cm<sup>-1</sup>) can be assigned to -C-C-, -C-N-, -C-O- groups. Medium peaks at 1373 cm<sup>-1</sup>, 1732 cm<sup>-1</sup> and 2850 cm<sup>-1</sup> indicate the presence of -CH<sub>3</sub> (sym. Deform.), C=O and -CH<sub>2</sub>- (sym. Stretch) respectively (Fig. 1) [27,28].

#### 3.2 Adsorption kinetics

The kinetics for As(III) adsorption (As(III) concentration of  $5.0 \text{ mg L}^{-1}$ ) on 1 gL<sup>-1</sup> of adsorbent at pH 7.46 and temperature 303 K were obtained by varying agitation time from 5-360 minutes. Adsorption of As(III) on OLP reached equilibrium within 60 minutes.

**3.2.1 Pseudo-first-order model.** Although the pseudo-first-order plot is linear (r = +0.97), the model was not appropriate to explain the kinetics of the process, due to the large deviations (~73%) observed between the  $q_e$  values calculated from the plots and those obtained experimentally (Table 1). Kamsonlian et al. (2012) [29] have reported similar first order rate constant value of 1.50 (x  $10^{-2}$  min<sup>-1</sup>) for adsorption of arsenite on banana peel at 308 K.

**3.2.2 Pseudo-second-order model.** Linear plot (r = 0.99) was obtained for  $t/q_t$  vs. t and the close agreement between the experimental  $q_e$  values and those obtained from the plots (deviation = -0.23) strongly support a pseudo second order mechanism for As (III) take-up by DILP i.e. each adsorbed As(III) molecule is attached to two active sites of the adsorbent surface through some chemisorptive bonds (Fig. 3).



Fig. 2: First-order plots for adsorption of As(III) on adsorbents at 303 K (adsorbent amount 1.0 g/L).

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Fig. 3. Pseudo second order plots for adsorption of As(III) on adsorbents at 303 K (DILP amount 1.0 g/L).

**3.2.3 Elovich equation.** Elovich plot yield straight lines with correlation coefficient values of 0.90 (Fig. 4). This suggests that the sorption systems studied belong to the second order kinetic model based on the assumption that the rate determining step may involve valence forces through sharing or exchange of electrons between adsorbent and adsorbate. The Elovich equation does not predict any definite mechanism, but it is found to be useful in describing adsorption on highly heterogeneous adsorbents when the interactions are predominantly chemical in nature [30].



Fig. 4: Plots of Elovich equation for adsorption of As(III) on adsorbents at 303 K (adsorbent amount 1.0 g/L).

Table 1: Kinetic parameters for sorption of As(III) on DLP, ADLP, OLP and RLP (Adsorbent 1.0 g L<sup>-1</sup>, As (III) concentration 5.0 mg L<sup>-1</sup>; temperature 303 K)

Kinetics	Parameters	OLP
First order	k1 x 10-2 min-1	1.66
	r	0.97

	(exp) mg/g	3.96
	qc (cxp) mg/g	5.70
	qe (plot) mg/g	1.07
	% deviation	73.04
Second order	k2 x 10-2 g mg-1min-1	6.80
	r	0.99
	qe (exp) mg/g	3.96
	qe (plot) mg/g	3.97
	% deviation	-0.23
Elovich	$\alpha$ (g mg-1min2)	229.16
	β (g mg-1)	2.86
	r	0.90
Intra-particle	ki x 10-2 mg g-1 min-0.5	16.30
diffusion	r	0.94
	Intercept (mg g-1)	2.51
Liquid film	kfd x 10-2 (min-1)	1.60
diffusion	r	0.97
	Intercept	1.31

**3.2.4 Intra-particle diffusion model.** In the plot, the intercept gives the idea about the thickness of boundary layer. Larger the intercept, greater is the boundary-layer and greater is the contribution of surface sorption in the rate determining step. From Fig. 5, we can say that the thickness of the boundary layer is high enough to invalidate this model.



Fig. 5: Plots of q<sub>t</sub> vs. t<sup>0.5</sup> for As(III) adsorption at 303 K (adsorbent amount 1.0 g/L).

**3.2.5 Liquid film diffusion model.** The linearity of the plot indicates some contribution to the overall mechanism, but the model requires the plot to pass through the origin. The condition of zero intercept is not followed, so this model is not valid for explaining the sorption interactions.

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Fig. 6: Plots of liquid film diffusion model for As(III) adsorption at 303 K (adsorbent amount 1.0 g/L).

#### 3.3 Adsorption isotherms

The isotherms were obtained by varying As(III) concentration from 0.5-5.0 mg  $L^{-1}$  with 1 g $L^{-1}$  of adsorbent loading at 303 K, agitated for 60 minutes.

At 303K, the Freundlich isotherm plots were linear with (1/n) < 1 indicating favorable adsorption (Fig. 7 and Table 2). Langmuir plots of C<sub>e</sub>/q<sub>e</sub> vs.C<sub>e</sub> (Fig. 8) were also linear (r = +0.87). DILP having maximum adsorption capacity of 3.85 mg g<sup>-1</sup>. The 'b' value is high enough for favorable formation of As(III)-DILP sorption complex.

The Temkin plots ( $q_e$  vs. log  $C_e$ ) for As (III) adsorption on DILP are linear ( $r \sim +0.89$ , Fig. 9) indicating that both adsorbate-adsorbate and adsorbate-adsorbent interactions are likely to play role in uptake of As (III) by the biosorbents. The values calculated from the plots are presented in Table 2. Sorption of As(III) by DILP predicted  $k_T$  value of 25.95 L mg<sup>-1</sup> suggesting a stronger bond formation between them. Large B value for the DILP showed that this biosorbent has higher heats of adsorption.



Fig. 7: Freundlich plots for adsorption of As(III) on DILP at 303 K



Fig. 8: Langmuir plots for adsorption of As(III) on adsorbents at 303 K



Fig. 9: Temkin plots for adsorption of As(III) on DILP at 303 K

 $\begin{array}{l} \label{eq:constraint} Table 2: Isotherm parameters for adsorption of As(III) from aqueous solution on DILP at 303 K. [As (III) 0.5 - 5.0 mg L^{-1} for DILP (1.0 g L^{-1})] K_f in mg^{1-1/n} L^{1/n} g^{-1}, q_m and b are in mg g^{-1} and L mg^{-1}, k_T in L mg^{-1}. \end{array}$ 

Freundlich isotherm		Langmuir isotherm		Temkin isotherm	
Kf	2.16	qm	3.85	kT	25.95
1/n	0.50	b	1.51	В	0.69
r	0.96	r	0.87	r	0.89

#### 4. CONCLUSIONS

The adsorption of As(III) on DILP closely followed second order kinetics. Equilibrium sorption data showed good fit to all the three isotherms, Freundlich, Langmuir, and Temkin.

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Thus, the sorption occurs on the heterogeneous surface of the biosorbent by monolayer formation. Easy-to-use systems make it ideal for homemade approaches for arsenic removal which do not require much technical knowhow. The selected plants are widespread species with the potential for application for the development of cheap arsenic filters with minimal capital investment.

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