

# Removal of As (III) from Aqueous Solution by Biosorption onto Maize (*Zea mays*) Leaves Surface: Parameters Optimization, Sorption Isotherm, Kinetic and Thermodynamics Studies

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## Abstract

Biosorption has been used for the removal of As (III) ion from aqueous solution using powdered maize leaves biomass in this study. Batch experiments were conducted by varying various process parameters such as pH, contact time and temperature. At optimum treatment conditions (pH 8, contact time of 4 hrs and temperature of 40<sup>o</sup>C) 84.9% As (III) ion was removed from aqueous solution. Langmuir and Freundlich isotherms were used for equilibrium studies. Freundlich isotherm was better fitted with experimental data. For kinetic studies, pseudo second order kinetic model ( $R^2 = 0.997$ ) appeared to be more suitable model to describe As (III) ion biosorption. The positive value of  $\Delta S^\circ$  and  $\Delta H^\circ$  depicts the feasibility of the biosorption and the spontaneous nature was confirmed by negative value of  $\Delta G^\circ$ . Fourier Transformation infrared spectrometry (FTIR) and Scanning Electron Microscopy (SEM) were used for characterisations of maize leaves biomass.

**Key words:** As (III) removal, biosorption, maize leaves, isotherm models, kinetics and thermodynamics.

## Introduction

The growth of industries has generated various types of pollutants. Effluent of industries like mining and chemicals processing units, surface finishing industry, fertilizer and pesticide industry and application, metallurgy, iron and steel, electroplating, electrolysis, electric appliance manufacturing, metal surface treating etc., contain tremendous amount of heavy metals<sup>1,2</sup>. Water contaminated by arsenic is a worldwide concern because of their carcinogenicity and delirious effects on human health and to the aquatic system<sup>3,4</sup>. Due to the contamination of water by industrial discharge and acid mine drainage millions of people are affected by arsenic. Arsenic is reported to cause acute and chronic metabolic disorders in humans. Due to its carcinogenic and other delirious effects on human health and aquatic environment, it is imperative to remove arsenic from contaminated water. World Health Organisation (WHO) and United States Environmental Protection Agency (USEPA) have established the maximum contamination limit (MCL) for arsenic in drinking water as 50 $\mu$ g/L to 10 $\mu$ g/L<sup>6</sup>. However, MCL is still high in Bangladesh, China and India as 50 $\mu$ g/L<sup>7</sup>.

Biosorption is a new and promising field of research of biomass wastes for removal of metal ions from aqueous solutions due to cost-effective and environmentally favorable. In this investigation maize leaves biomass was used and study its efficiency in As (III) ion removal. The parameters affecting the biosorption process such as pH, contact time and temperature were studied. Moreover, sorption equilibrium, kinetic models and thermodynamics were also evaluated in this work.

## Material and Methods

**Biosorbent Preparation and Characterization:** The fresh maize leaves used in this investigation were thoroughly washed with distilled water, dried at room temperature and then dried in an oven at 60<sup>o</sup>C for 5 hours. Dried maize leaves were grounded and sieved (range 1.18 mm to 425 $\mu$ m) and used as biosorbent in present study. Morphology and characterization of solid maize leaves were determined by using Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR).

**Chemicals and Stock Preparation:** All chemicals used in the present experiment were of analytical reagent (AR) grade. For preparation of stock solutions and dilutions, double deionized water (Millipore 17.9 M $\Omega$ /cm) was used. Stock solution of 1000 ml/l concentration of As (III) was prepared from sodium meta arsenite (As<sub>2</sub>O<sub>3</sub>).

**Batch Experimental Studies:** A known amount of biosorbent was added to 100 ml conical flask containing arsenic solution. Thus the mixture was agitated at 180 rpm of a predicted constant temperature for 4 hours to attain equilibrium. The desired pH was adjusted with 1N NaOH and 1N HCl using a pH meter (pH 510 Eutech Instruments, Cyberscan). The amount of arsenic sorbed per unit mass of the biosorbent (mg/g) was evaluated by using following equation

$$q_t = (C_i - C_e) \frac{V}{W} \dots\dots\dots (1)$$

where,  $C_i$  and  $C_e$  the initial arsenic concentrations (mg/l) and at equilibrium, respectively, V the volume of the arsenic solutions (ml), and W the weight of biosorbent (mg).

To study the effect of initial pH on the uptake of As (III) ion, pH was varied from 2 to 10 dosage was taken as 1 g, initial concentration ( $C_i$ ) 50 mg/l, contact time 4 hrs and temperature of 40°C respectively. Dose study was performed by taking pH 8 (optimum pH). Effect of agitation period on the uptake was carried out by varying the contact time from 0.5 hrs to 15 hrs. Effect of initial concentration of arsenic was determined by varying the initial concentration from 50 mg/l to 150 mg/l, pH 8, dose 1 g, contact time 4 hrs (optimum contact time) were maintained. Temperature was varied from 20°C to 50°C for the study. After the treatment, the solution was filtered and the concentration of arsenic in the filtrate was ascertained by spectrophotometric method<sup>8</sup>.

**Method of Analysis:** Silver diethyl diethiocarbamate (SDDC) method was used for determination of arsenic (IS 3025: 1988 and ASTM D 2972: 2008). The detection limit of arsenic for SDDC method ranges from 5 µg/l to 250 µg/l (ASTM D 2972: 2008) which also meet USEPA and WHO limit for drinking water of 10 µg/l. In this method AsH<sub>3</sub> (arsine gas) is generated in a SDDC reagent and subsequently reaction of AsH<sub>3</sub> with SDDC formed a coloured complex (reddish colour). The absorbance of the formed complex was measured by spectrophotometer (Tashniwal TVS 25A made) at a wavelength ( $\lambda$ ) of 540 nm<sup>9</sup>.

The percentage removal was calculated as follows

$$\text{Removal (\%)} = \frac{(C_i - C_f)}{C_i} \times 100 \dots\dots\dots (2)$$

where  $C_i$  and  $C_f$  are the initial and final concentrations of As (III) present in the biosorption studies.

## Results and Discussion

**Characterisation of Biosorbent:** The morphology of the biosorbent was examined through SEM at 200x magnification. SEM analysis reveals a highly porous structure (figure 1). FTIR spectrum analysis of the biosorbent revealed that the presence of functional groups like aliphatic ethers, aliphatic primary amines and primary aliphatic alcohols as shown in figure 2. Strong vibration peaks in the region of 3853.21 – 3430.97 cm<sup>-1</sup> were demarcated as the vibrations of OH or –NH functional groups<sup>10</sup>. The 2922.65 cm<sup>-1</sup> band show the presence of C–H asymmetric stretching vibration. The peaks in the regions 1734.05 – 1047.60 cm<sup>-1</sup> were due to the presence of –CN and C–C stretching. The peak at 605.97 cm<sup>-1</sup> was due to the presence of C–O stretching<sup>11</sup>.

**Effect of pH on As (III) ion Biosorption:** The pH value of aqueous solutions was a critical parameter affecting the biosorption process<sup>12</sup>. In this study, the effect of pH can be described by considering the surface charge on the biosorbent. The effect of pH on sorption capacity was studied in the range of 2 to 10 at 50 mg/l initial arsenic concentration.

The sorption of As (III) ion is highly pH dependent (figure 3). The percentage of As (III) ion uptake steeply increased

to 77.8% at pH 8, due to the electrostatic attractions. However, no further positive effect on arsenic removal was observed at higher pH values. The possible reason for this could be the limitations in metal ion mobility, which can be attributed to change in characteristics and charge<sup>13</sup>. The maximum removal of As (III) ion was obtained at pH 8.

### Effect of Contact Time and Temperature on As (III) Biosorption:

The effect of agitation time on the biosorption of As (III) ion onto maize leaves was studied from 0.5 to 15 hours (figure 4). Uptake of As (III) ion increases rapidly with the lapse of time and reaches to equilibrium in 4 hrs. Around 67.8% removal of As (III) ion was achieved within one hour of contact time, suggests an excellent affinity of the biosorbent for the uptake of metals from liquid phase<sup>14</sup>. It was found from experimental results that 84.98% removal of As (III) ion onto biosorbent was achieved at 4 hrs contact time.

The effects of temperature on biosorption capacity of As (III) ion onto the surface of maize leaves was studied from 20°C to 50°C (figure 5). In present investigation, increase in temperature induces positive impact on removal of As (III) ion from aqueous solution. At 40°C, 84.9% removal of As (III) ion was achieved. Further increased in temperature has no significant percentage removal of As (III) ion from the liquid phase due to the loss of surface active OH sites<sup>15</sup>.

### Equilibrium Modelling:

Sorption isotherms describe the partition of metal ions in solid and liquid phase at equilibrium. Sorption equilibrium study provides fundamental physicochemical results to evaluate the applicability of sorption processes as a unit operation<sup>16</sup>. In the present investigation, Langmuir and Freundlich isotherm models have been tested to describe the relationship between the amount adsorbed onto the biosorbent and its equilibrium concentration in liquid phase<sup>17</sup>.

Langmuir isotherm model assumes that the surface of adsorbent is homogeneous and all active sites are energetically symmetrical resulting mono layer adsorption. Langmuir model can be represented as

$$q_e = \frac{K_L \times C_e}{(1 + b \times C_e)} \dots\dots\dots (3)$$

where  $K_L$  and  $b$  are the maximum uptake capacity (mg/g) and Langmuir constant related to the capacity (l/mg), respectively.  $C_e$  is the equilibrium arsenic concentration (mg/l). The linearization of equation 3 results

$$\frac{C_e}{q_e} = \frac{1}{b \times K_L} + \frac{C_e}{K_L} \dots\dots\dots (4)$$

Freundlich isotherm model expresses the heterogeneity of the adsorbent material and multi layer coverage of biomass surface<sup>18</sup>. Mathematical expression of Freundlich model can be expressed as

$$q_e = K_f \times C_e^{(1/n)} \dots\dots\dots (5)$$

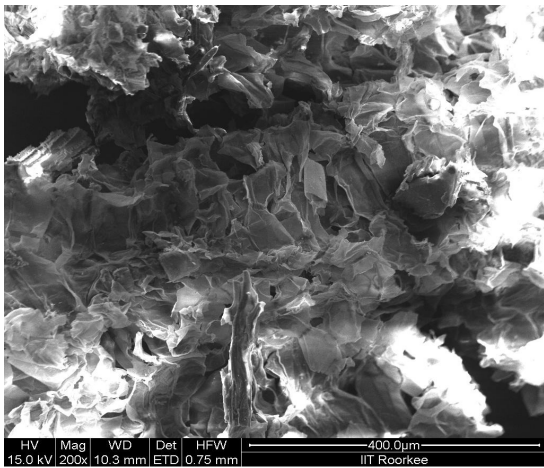


Figure-1

SEM photograph of maize leaves (200 X magnification)

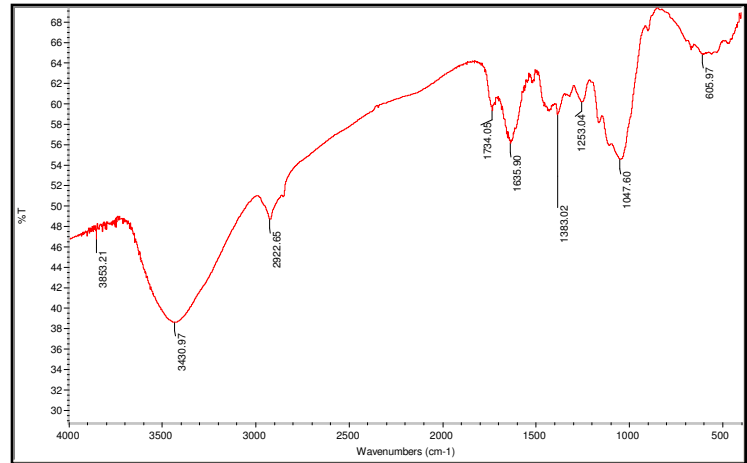


Figure-2

FTIR analysis of maize leaves

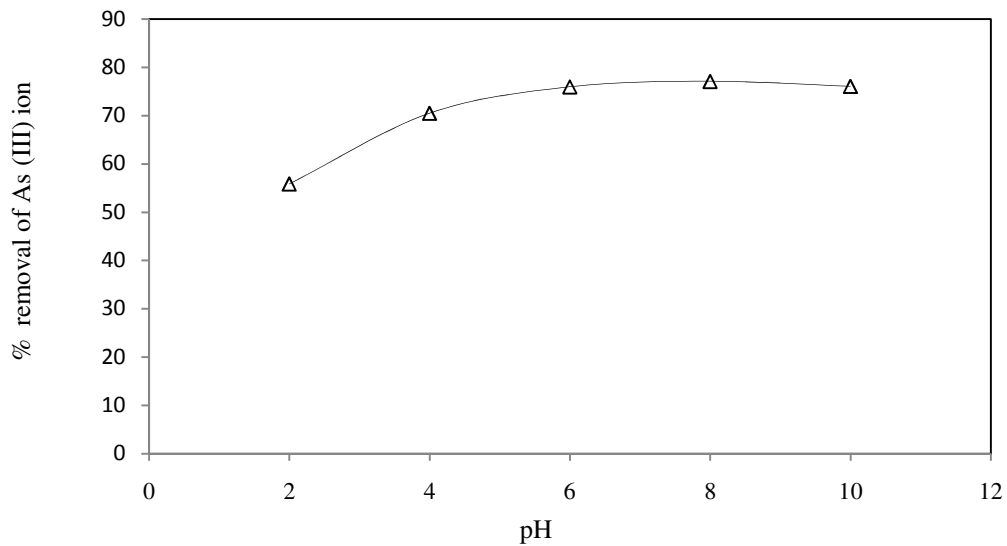


Figure-3

Effect of initial pH (pH 2-10) on sorption capacity of maize leaf for removal of As (III) ion (wt. = 0.1 g,  $C_0 = 50$  mg/l and 180 rpm)

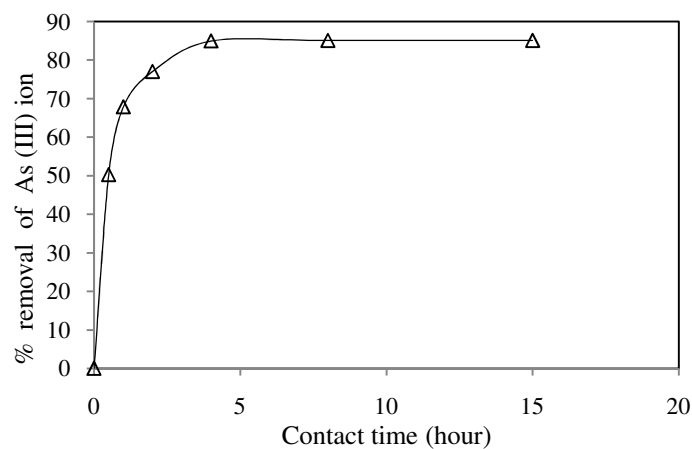
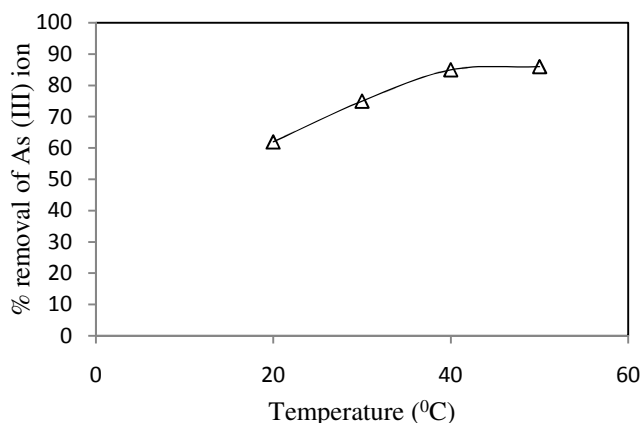
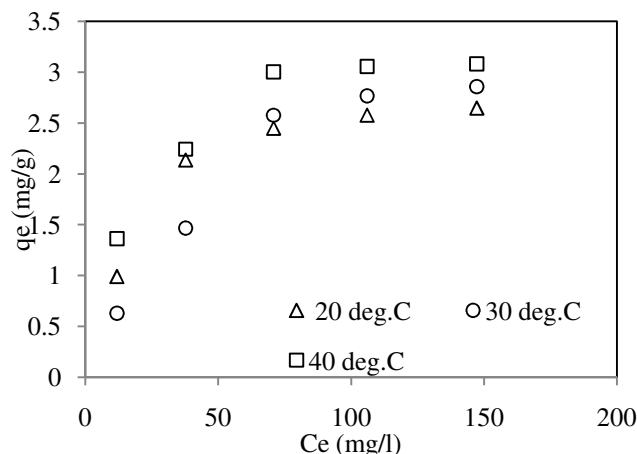


Figure-4

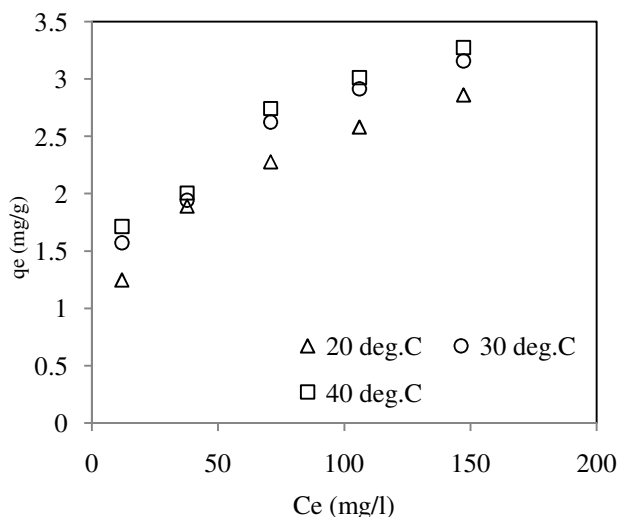
Effect of contact time on sorption capacity of maize leaf for removal of As (III) ion (wt. = 0.1 g,  $C_0 = 50$  mg/l and 180 rpm)



**Figure-5**  
Effect of temperature on sorption capacity of maize leaf for removal of As (III) ion (wt. = 0.1 g, C<sub>0</sub> = 50 mg/l and 180 rpm)



**Figure-6**  
Study of Langmuir isotherm curves for As (III) ion sorption onto maize leaves surface at different temperatures



**Figure-7**  
Study of Freundlich isotherm curves for As (III) ion sorption onto maize leaves surface at different temperatures

where  $K_f$  and  $n$  are constants for adsorption capacity (l/g) and affinity constant, respectively. Linear form of isotherm equation 5 can be expressed as

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \dots\dots\dots (6)$$

The experimental data were fitted with the selected isotherm model. Figure 6 and 7 represent the isotherm plot for sorption of As (III) ion onto the biosorbent. Constant parameters and correlation coefficient values of each model obtained are given in table 1. From the correlation coefficient ( $R^2$ ) values of all isotherm equations, Freundlich model provides a better understanding of As (III) ion biosorption with linear correlation coefficients ( $R^2$ ) near to unity. However, Langmuir model did not fit

**Table-1**  
Parameters of Langmuir and Freundlich models for the sorption of As (III) ion onto the surface of maize leaves

Isotherm model	Temperature (°C)	Model parameters
Langmuir model	20	$K_L = 2.032$ ; $b = 0.029$ ; $R^2 = 0.978$
	30	$K_L = 2.731$ ; $b = 0.172$ ; $R^2 = 0.963$
	40	$K_L = 3.811$ ; $b = 0.232$ ; $R^2 = 0.952$
Freundlich model	20	$K_f = 0.38$ ; $n = 2.558$ ; $R^2 = 0.990$
	30	$K_f = 1.116$ ; $n = 5.329$ ; $R^2 = 0.992$
	40	$K_f = 1.199$ ; $n = 4.691$ ; $R^2 = 0.994$

well experimental data. The maximum uptake capacity of As (III) ion was 85.6 mg/g, it is showed that maize leaves have a potential to be a good biosorbent for removal of heavy metals from aqueous solutions due to its high sorption capacity and availability.

**Biosorption Kinetic Studies:** Kinetics study reveals the rate and mechanism of reaction coupled with determination of rate controlling step<sup>19</sup>. In the present investigation, pseudo-first-order and pseudo-second-order models have been studied to find out the rate limiting step of As (III) ion biosorption across liquid phase<sup>20</sup>. Pseudo first order reaction model has been represented in equation (7)

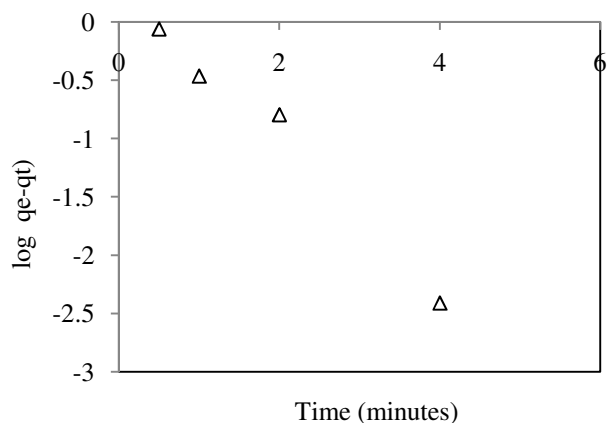
$$\frac{dq_t}{dt} = K_1(q_e - q_t) \dots\dots\dots (7)$$

where  $q_e$  and  $q_t$  uptake capacities of metal ion per unit of adsorbent at equilibrium and at time  $t$  (min) and  $K_1$  the As (III) ion biosorption constant. On integrating at boundary conditions the following equation obtained

$$\log(q_e - q_t) = \log q_e - \frac{K_1 t}{2.303} \dots\dots\dots (8)$$

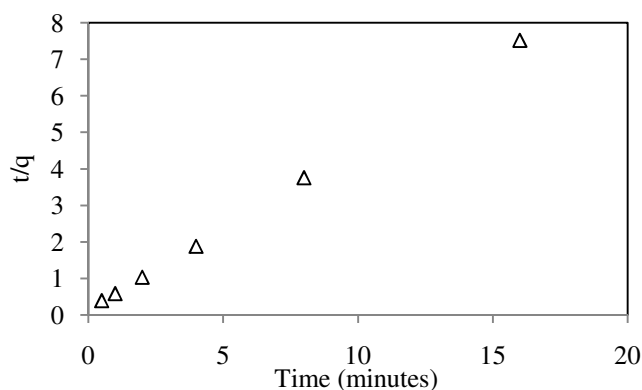
**Table-2**  
**Summary of kinetic model parameters for the sorption of As (III) ion onto the surface of maize leaves**

Metal ion	Experimental $q_e$ (mg/g)	Pseudo first order model			Pseudo second order model		
		Calculated $q_e$ (mg/g)	$K_1$ ( $\text{min}^{-1}$ )	$R^2$	Calculated $q_e$ (mg/g)	$K_2$ (g/mg.min)	$R^2$
As (III)	0.812	0.935	0.912	0.978	0.725	0.321	0.997



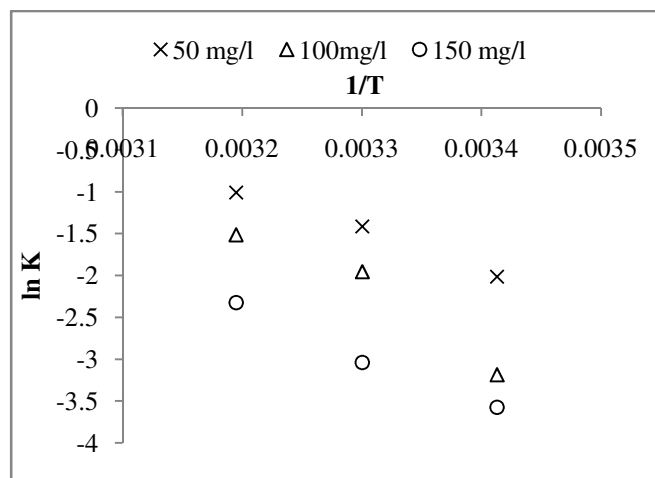
**Figure-8**

Study of pseudo first order reaction at 40°C, 50 mg/l, 180 rpm and dose 1 g/l



**Figure-9**

Study of pseudo second order model reaction at 40°C, 50 mg/l, 180 rpm and dose 1 g/l



**Figure-10**

Study of thermodynamic parameters for As (III) ion sorption onto maize leaves surface at different temperatures

The values of  $K_1$  ( $\text{min}^{-1}$ ) and  $q_e$  (mg/g) can be calculated from the intercept and slope of the plots of  $\log(q_e - q_t)$  versus  $t$ . Results of pseudo first order model has been represented in figure 8 and table 2.

The Pseudo second order model can be represented in equation (9)<sup>21</sup>

$$\frac{d q_t}{d t} = K_2 (q_e - q_t)^2 \dots \dots \dots (9)$$

Integrating above equation at boundary conditions the following equation obtained<sup>22</sup>

$$\frac{t}{d t} = \frac{1}{(K_2 \times q_e^2)} + \frac{t}{q_e} \dots \dots \dots (10)$$

Extrapolation of the plot between  $t/q_t$  and  $t$  calculates the value of  $K_2$  and  $q_e$  as intercept and slope of the curve respectively. The graph of pseudo second order model has been represented in figure 9 and the constants of kinetic models are given in table 2. Pseudo second order model gives better prediction of uptake rate with linear correlation coefficients ( $R^2 = 0.997$ ).

**Thermodynamic Study:** Thermodynamic biosorption parameters such as the change of adsorption enthalpy ( $\Delta H^\circ$ ), entropy ( $\Delta S^\circ$ ), and free energy change ( $\Delta G^\circ$ ) are of necessity to resolve the magnitude of parameter change during the biosorption process due to the transfer of a unit mole of solute from solution to the solid liquid interface and solid surface<sup>23</sup>. In present investigation, thermodynamics feasibility of As (III) ion adsorption was calculated in terms of  $\Delta G^\circ$  (kJ/mol),  $\Delta H^\circ$  (kJ/mol) and  $\Delta S^\circ$  (kJ/mol.K).

$$\Delta G^\circ = -RT \ln K \dots \dots \dots (11)$$

$$\ln K = \frac{C_a}{C_e} \dots \dots \dots (12)$$

where,  $C_a$  = As (III) ion adsorbed per litre (mg/l),  $C_e$  = equilibrium concentration of As (III) ion (mg/l),  $R$  = gas constant ( $8.314 \text{ J ml}^{-1} \text{ K}^{-1}$ ) and  $T$  = temperature (K)

$$\ln K = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \dots\dots\dots (13)$$

Plot of  $\ln K$  vs  $1/T$  gives the thermodynamic parameters  $\Delta H^\circ$  (kJ/mol) and  $\Delta S^\circ$  (kJ/mol. K) respectively.

Thermodynamics parameters study and process feasibility has been represented. Thermodynamic studies on experimental data have been done and the constants were reported in table 3. Results show that the negative value of Gibbs free energy ( $\Delta G^\circ$ ) represents the feasibility and spontaneous nature of biosorption onto the surface of grounded and sieved maize leaves<sup>24</sup>. The positive sign for change in enthalpy ( $\Delta H^\circ$ ) and entropy ( $\Delta S^\circ$ ) indicated that As (III) ion biosorption was endothermic in nature coupled with increase in randomness at the solid liquid interface during the sorption process<sup>25</sup>.

**Table-3**  
**Summary of thermodynamic parameters for As (III) ion onto the surface of maize leaves**

Description	Temp. (°C)	Thermodynamic parameters		
		$\Delta G^\circ$ (kJ/mol)	$\Delta H^\circ$ (kJ/mol)	$\Delta S^\circ$ (kJ/mol.K)
Maize leaves	40	-117.24		
	30	-113.09	63.97	0.171
	20	-110.94		

## Conclusion

The present investigation indicated that the potential of maize leaves biomass as an economically feasible biosorbent for the removal of As (III) ion from aqueous solutions. Maximum percentage removal of As (III) ion was obtained as 84.98% at temperature of 40°C, contact time of 4 hrs dose of 1 g, pH 8 and 180 rpm, respectively. The sorption of As (III) ion was found to be highly pH dependent. Functional groups presented on the surface of maize leaves biomass significantly affect the biosorption process. For equilibrium studies, Freundlich isotherm was found to be more favourable compare to Langmuir model. The uptake of As (III) ion onto solid surface of maize leaves was studied by using kinetic models. The pseudo second-order kinetic model fits very well with linear correlation coefficients ( $R^2 = 0.997$ ). The negative Gibbs free energy ( $\Delta G^\circ$ ) value obtained from the experimental results confirmed the feasibility and spontaneous nature of the biosorption. Positive values of both enthalpy  $\Delta H^\circ$  and  $\Delta S^\circ$  entropy indicates the endothermic nature of the process with increased randomness at liquid solid interface during As (III) ion adsorption in batch studies.

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