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Artificial Neural Network Modeling of Biosorptive Removal of Arsenic(V) by a Low-cost Biomass

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Abstract

The presence of arsenic in drinking water has been recognized as a serious community health problem because of their toxic nature and therefore, its removal is highly essential. This paper deals with batch biosorption study for the removal of pentavalent arsenic ions from aqueous solutions using finely ground (250 µm) *Azadirachta indica* (neem) bark powder (AiBP) as a low-cost biosorbent. Employing the batch experimental setup, the effect of operational variables such as initial concentration of As(V), pH, biosorbent dose, contact time, temperature and agitation speed on the As(V) removal process were studied. Under optimized batch conditions, the AiBP could remove up to 86.6% of As(V) from contaminated water. The biosorbent dose had the most significant impact on the biosorption process. The artificial neural network (ANN) model developed from batch experimental data sets, provided reasonable predictive performance (R^2 = 0.951; 0.967) of arsenic biosorption. The study on equilibrium biosorption of batch operation revealed that Freundlich isotherm model gave the best fit to experimental data. The nature of biosorption of As(V) by AiBP was physisorption as inferred from the D–R isotherm model. The biosorption is pseudo second–order, exothermic and spontaneous.

Abbreviations

AiBP: *Azadirachta indica* bark powder;
ANN: artificial neural network;
D–R: Dubinin–Radushkevich;
SDDC: silver diethyl dithiocarbamate;
SEM: scanning electron microscopy.

1. Introduction

Groundwater enriched with arsenic in the form of arsenate [As(V)] and arsenite [As(III)] has emerged as a major concern on a global scale [1–6]. Exposure to arsenic through drinking water sourced from groundwater [1,2,7] poses a serious health hazards in several developing regions [2,8]. As high as, the WHO provisional guideline of 10 µg/L of arsenic in drinking water is now recognized as a worldwide problem in many countries, especially in the Southeast of Asia, including India, Bangladesh, and China [1,3]. A largest segment of population currently is at risk in the Bengal Basin area of Bangladesh and West Bengal in India [4,9,10]; however, it is remarkable that these two countries have retained the earlier WHO guideline of 50 µg/L as their standard of arsenic in drinking water [11,12]. About 70 million people are suffering from arsenic problem alone in these regions; this is perhaps the largest poisoning in world's history [13,14]. Today, in West Bengal, the arsenic contamination in groundwater, and eventually in drinking water, has been reported in the range from 50 to 3600 µg/L, with predominance of As(V) [9,10,15], over 111 blocks in 12 districts of the state [8,16]; affecting more than 34 million people [15]. Thus, the removal of As(V) from drinking water has received significant attention and major concern to many water utilities and governmental agencies.

Among a variety of adsorbents for arsenic removal, there has been an increase in the use of biomasses (biosorbents) because they are relatively low-cost, readily available [9,17–19], and therefore may easily be used as column fillings in small-scale treatment plants. Of late, biosorption has emerged as a promising remediation technology [4,6], which uses dead or alive biomasses [14] to remove arsenic from contaminated environment. However, only a limited number of biosorbents (without chemical modification) have been examined for their efficacy to remove arsenic from contaminated solutions [2,4–6,9–14,18–23].

The powder form of brown colored mature stem bark of the *Azadirachta indica* (neem) tree is an example of such type of a low-cost biosorbent. In the past few years, this low-cost biosorbent has drawn considerable attention to the scientific community due to its wide availability all over the world, especially in the Southeast Asia [17,24]. The biosorptive removal behavior of Zn(II) [17,25–27], Cd(II) [27,28], Cr(VI) [24,29] and dyes [30–32] from aqueous solutions on the *Azadirachta indica* bark powder (AiBP) had previously been investigated but no information is, however, available in literature on the removal of arsenic by AiBP; except our earlier reports on arsenic(III) [23].

In view of these attributes, it is our present interest to investigate the application of AiBP for the removal of arsenic from aqueous solutions. With this fundamental goal in mind, the present study deals with batch experiment to assess the potentiality of AiBP for removal of arsenic(V) from aqueous environment. For sensitivity analysis, the batch biosorption experiment has statistically been modeled using artificial neural network (ANN). The model is also applied to study the individual effect of different variables influencing the biosorption process. Results obtained from this study are presented and discussed.

2. Material and Methods

2.1. Reagents and apparatus

Chemicals of analytical grade were procured from M/S, Merck India Ltd., and used in the study without further purification. All reagents and standards were prepared using double distilled water. Sodium arsenate hydrate ($Na_2HAsO_4 \cdot 7H_2O$) was used for the preparation of standard As(V) solution and the required As(V) concentrations to prepare contaminated water for experiments were prepared by serial dilution of standard solution on daily basis. All borosil glassware were cleaned by being soaked in 15% HNO_3 and rinsed with double distilled water. Different pH of As(V) solution was generated by the addition of 0.1 N HCl or NaOH solutions whenever necessary.

2.2. Instrumental and software

The shaking in batch experiments were conducted in a temperature controlled magnetic stirrer (Spinot, Cat No. 6040). A UV–visible spectrophotometer (Systronics, Vis double beam Spectro 1203) with a 1–cm quartz cell was used for quantitative determination of As(V) in solution. pH was measured by a digital pH meter (Systronic–335) with an accuracy of ± 0.01 unit. A high precision electrical balance (Sartorius–312) was used for weighing. Surface morphology of the biosorbent was studied by a Hitachi, S–530 (Make: Eiko Engineering, Ltd., Japan) scanning electron microscope.

SPSS–17 (SPSS Inc., Chicago, USA) software was applied for the data analysis and to study the effect of different variables influencing the batch process.

2.3. Preparation of AiBP

The brown colored neem (*Azadirachta indica*) barks, used in the investigation, were collected from the University campus, Burdwan University, Burdwan. The collected barks were thoroughly washed with double distilled water to remove adhering muddy materials and soaked in 0.1 N NaOH to remove lignin based color materials followed by 0.1 N H_2SO_4 [26,27,32]. The washed neem barks were dried in sun for fifteen days. The dried barks were then cut into small pieces and ground to powder with kitchen grinder and finally sieved to obtain a constant size of 250 µm prior to use as adsorbent (biosorbent) [23]. The physiochemical characterizations of the biosorbent, *Azadirachta indica* bark powder (AiBP), has been reported elsewhere [31,32].

2.4. Analytical determination

Quantitative determination of arsenic was carried out spectrophotometrically by silver diethyl dithiocarbamate (SDDC) method [8,12,15] with minimum detectable quantity of 1 µg [15,33]. Each sample was analyzed in triplicate and the results were found reproducible within ± 3% error limit. Calibration was carried out daily with a freshly prepared arsenic standard, before analysis. Blank experiments were conducted to ensure that no biosorption was taking place on the walls of the apparatus used [23]. Experiment done with control biomass indicates no release of arsenic by the biomass.

2.5. Batch experimental setup

In the batch operation, the effect of different variables (i.e., pH, contact time, initial concentration of As(V), dose of biosorbent, agitation speed and temperature) on biosorption of As(V) was studied. The solution of 100 mL As(V) was taken in 250–mL Erlenmeyer flask. After pH adjustment, a known quantity of dried AiBP biosorbent was added into each flask and the As(V) bearing suspensions was kept under magnetic stirring until the equilibrium condition was reached. Then, the flasks were taken out from the stirrer at pre–determined intervals and the content was filtered using Whatman–42 grade filter paper to separate the biosorbent and filtrate. 25 mL filtrate of each batch experiment was taken for analysis and residual arsenic was determined using SDDC method. The arsenic concentrations before and after biosorption were recorded, and the percent of arsenic biosorption (removal efficiency) by the biosorbent was computed by using the following equation:

$$\%\ \text{Biosorption (Removal efficiency)} = \frac{(C_i - C_e)\times 100}{C_i}$$

where C_i and C_e are the initial and final concentration of As(V) in the solution.

The arsenic uptake loading capacity (q_e= µg/g) of AiBP for different concentration of As(V) at equilibrium was also determined by using the equation; $q_e = \frac{(C_i - C_e)\times V}{M}$.

where, V is the volume of solution (L) and M is the mass of the biosorbent (g) used.

The experimental datasets obtained from the batch studies, were used as inputs to the ANN model to provide the reasonable predictive performance of the biosorbent.

3. Results and discussion

3.1. Effect of initial arsenic concentration

The biosorption behavior of arsenic(V) was studied in the arsenic concentration range of 50–500 µg/L, initially at pH 6.0. In general, the removal percentage of As(V) on AiBP was initially increased with the increasing initial concentration of arsenic reaching the optimum level of 66.6% at 100 µg/L. Thereafter, the percentage of removal showed little decrease (Figure 1). At higher concentrations, lower biosorption yield is probably due to the saturation of free biosorption sites. Though an increase in arsenic uptake, with decrease in percentage of biosorption, was attributed to lack of available active sites on the biosorbent surface to accommodate much more arsenic available in the solution [9,26,33].

3.2. Effect of pH

Figure 2 represents the percentage removal (or uptake capacity) as a function of the pH at optimum concentration (100 µg/L) of As(V). In the experimental pH range of 4.0–10.0, the predominant arsenate species are usually $H_2AsO_4^-$ and $HAsO_4^{2-}$. It has been well documented that at pH range of 4–6 As(V) presents mainly in the form of $H_2AsO_4^-$; whereas, the divalent anion $HAsO_4^{2-}$ dominates at higher pH of 8–10. Also in the intermediate pH range of 6–8, both species co–exist. The optimum removal 66.6% (44.4 µg/g) was obtained at the pH of 6.0. At optimum pH of 6.0, it seems possible that arsenate can be biosorbed through specific biosorption between the monovalent anionic species ($H_2AsO_4^-$) and positively charged surface sites [15] (as the zeta potential; pH_{zpc} of AiBP = 6.80 [31]); via coordination with the hydroxyl groups present in the surface of the biosorbent [18,24,25]. The low biosorption shown at higher pH might be due to the reason that the density of OH^- ion becomes dominant at an alkaline pH and this ion competes with anionic arsenic species. In addition, the

carboxyl, hydroxyl, and amide groups [24] of the biomass would be negatively charged at alkaline conditions. Therefore, there is a repulsive force between the negatively charged biomass and the anions ($H_2AsO_4^-$ and $HAsO_4^{2-}$) resulting in lower biosorption efficiency [18,25].

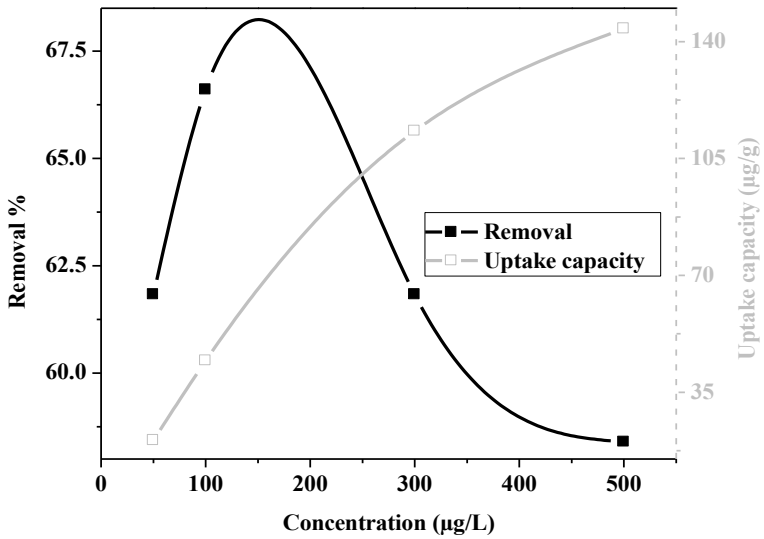

Figure 1: Effect of initial arsenic concentration on the biosorption of As(V) (Experimental conditions: dose = 0.15 g; contact time = 20 min; pH = 6.0; temperature = 25 ºC; agitation speed = 300 rpm).

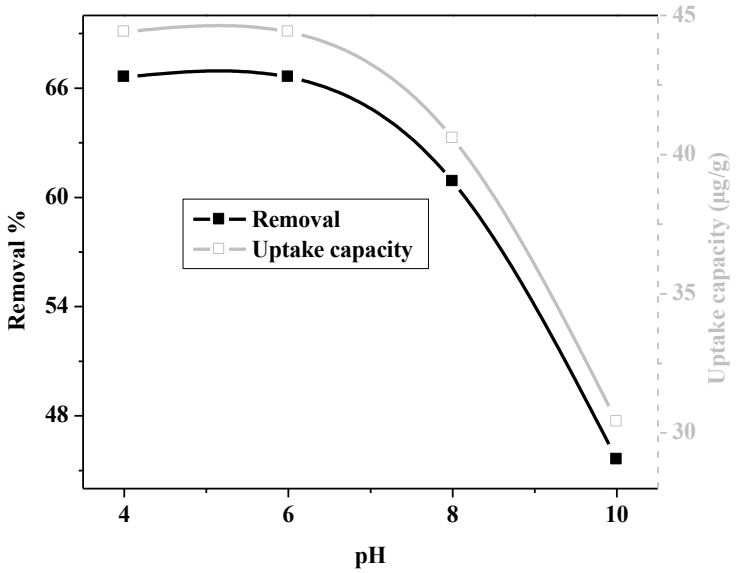

Figure 2: Effect of pH on the biosorption of As(V) (Experimental conditions: initial arsenic concentration = 100 µg/L; dose = 0.15 g; contact time = 20 min; temperature = 25 ºC; agitation speed = 300 rpm).

3.3. Effect of biosorbent dose

While observing the influence of the dry weight of AiBP biomass on the removal efficiency at pH 6.0, it was found that the removal efficiency of arsenic(V) increased (Figure 3) with increasing biosorbent doses (0.05–0.30 g). No remarkable improvement in the removal efficiency was found on further increasing the biomass dosages from 0.25 g onwards. Increasing removal efficiency with biosorbent dose seems to be attributed simply to an increase in biosorbent surface area and availability of more active sites [21,26,34]. The higher removal might also be due to the presence of suitable functional groups on AiBP surface, which help in the biosorption of arsenic [24,25].

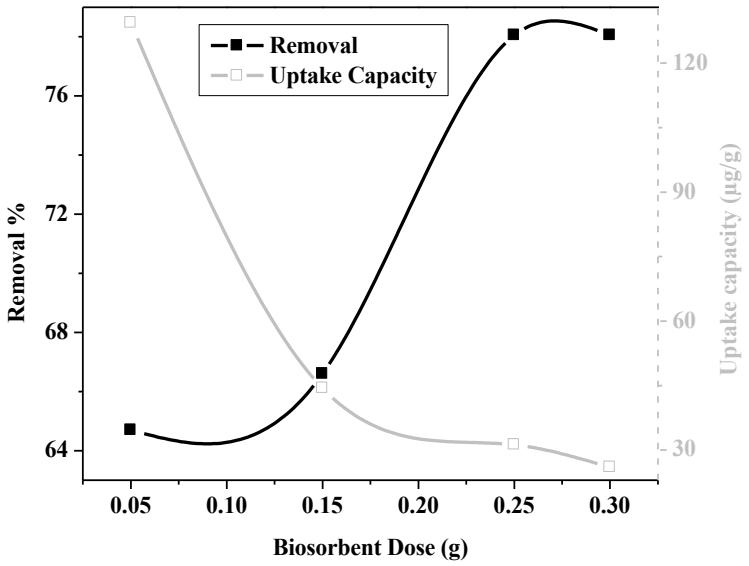

Figure 3: Effect of biosorbent dose on the biosorption of As(V) (Experimental conditions: initial arsenic concentration = 100 µg/L; contact time = 20 min; pH = 6.0; temperature = 25 ºC; agitation speed = 300 rpm).

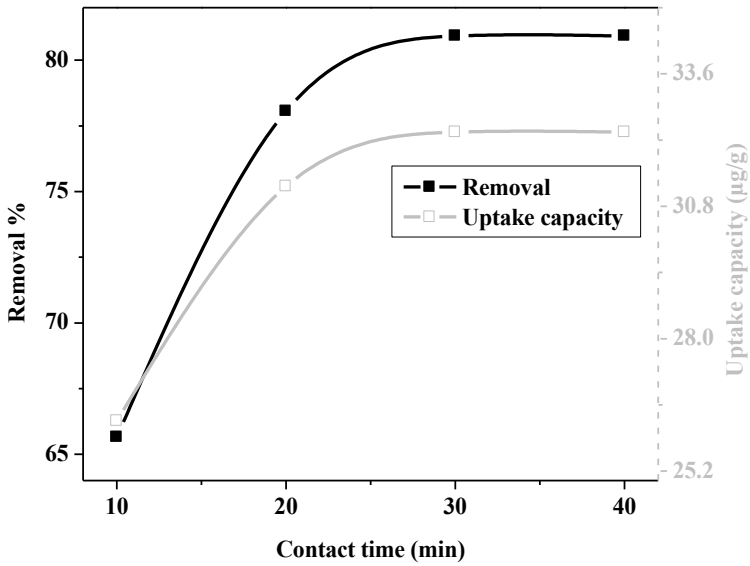

Figure 4: Effect of contact time on the biosorption of As(V) (Experimental conditions: initial arsenic concentration = 100 µg/L; dose = 0.25 g; pH = 6.0; temperature = 25 ºC; agitation speed = 300 rpm).

3.4. Effect of contact time

The biosorption of arsenic(V) on AiBP with different time interval (10–40 min) at optimum value of arsenic concentrations (100 µg/L) at pH 6.0 and 0.25 g of biosorbent dosage is shown in Figure 4. The percentage removal of As(V) on AiBP showed a rapid initial increment up to 30 min, which gradually reached at equilibrium thereafter. Further increase in interval time up to 40 min does not affect the equilibrium of arsenic biosorption at all. The fast biosorption rate at the initial stage is probably due to abundant availability of active biosorption sites on biosorbent surface [22,28,34]. With a lapse of time, the remaining unsaturated sites are difficult to occupy because of repulsive forces between the solute (arsenic ions) on the solid and bulk phases [8,35].

3.5. Effect of temperature

The influence of temperature on the removal of arsenic(V) by AiBP was measured at different temperatures ranging from 25 to 55 °C (Figure 5). The percent removal of As(V) was increased from 80.9 to 83.8% when temperature was increased from 25 to 35 °C and thereafter it was decreased. The decrease in biosorption might occur due to the fact that at high temperature, solute move with greater speed, therefore, less time of interaction along with the biosorbent active sites was available for them [36,37].

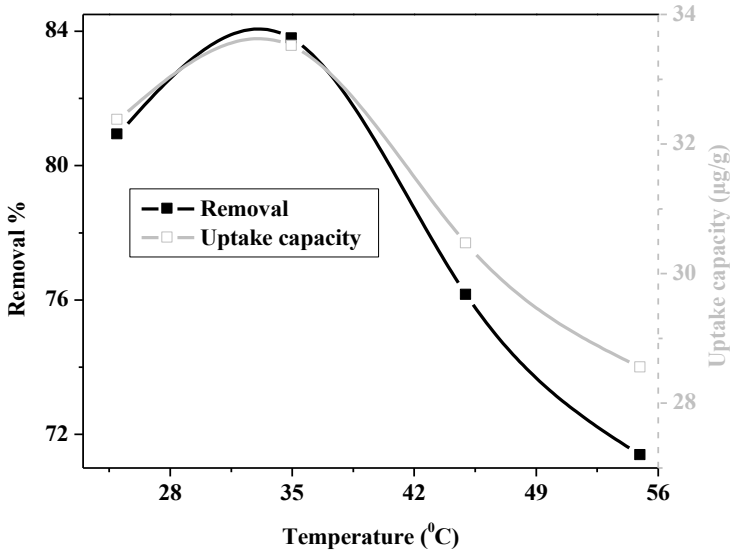

Figure 5: Effect of temperature on the biosorption of As(V) (Experimental conditions: initial arsenic concentration = 100 µg/L; dose = 0.25 g; contact time = 30 min; pH = 6.0; agitation speed = 300 rpm).

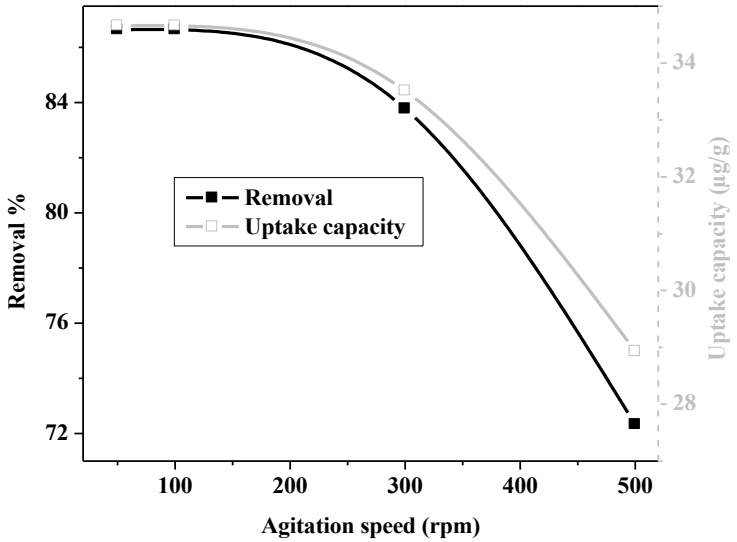

Figure 6: Effect of agitation speed on the biosorption of As(V) (Experimental conditions: initial arsenic concentration = 100 µg/L; dose = 0.25 g; contact time = 30 min; pH = 6.0; temperature = 35 ºC).

3.6. Effect of agitation speed

The effect of different agitation speed varying from 50 to 500 rpm was examined, and it appeared that the percentage of arsenic(V) biosorption was increased with the decrease in agitation speed (Figure 6). Probably an increase of speed did not give sufficient time for arsenic ions and biosorbent surfaces to interact with each other

and also resulted in detachment of loosely bounded ions. At 100 rpm, maximum removal occurred at the level of 86.6% for As(V); but below 100 rpm there was no significant increment in the rate of removal. This is because all the active binding sites have been utilized and no binding sites were left for further biosorption [23,37].

3.7. Artificial neural network (ANN) modeling

ANN is an advance mathematical or computational modeling procedure which is similar to that of biological neural networks. ANN can map a set of input patterns onto a corresponding set of output patterns after a series of previously processed data from a given system have been acquired, without knowing the intricate relationship among them [20,22,23]. Moreover, ANN can be trained to identify patterns and extract trends in imprecise and complicated non–linear data [8,19,20]. As biosorption is a complex non–linear process, neural network are found suitable for prediction of arsenic biosorption properties. Neural network toolbox of SPSS–17 mathematical software was used to predict the biosorption process of AiBP under batch studies. The topology of ANN architecture of this study is illustrated in Figure 7.

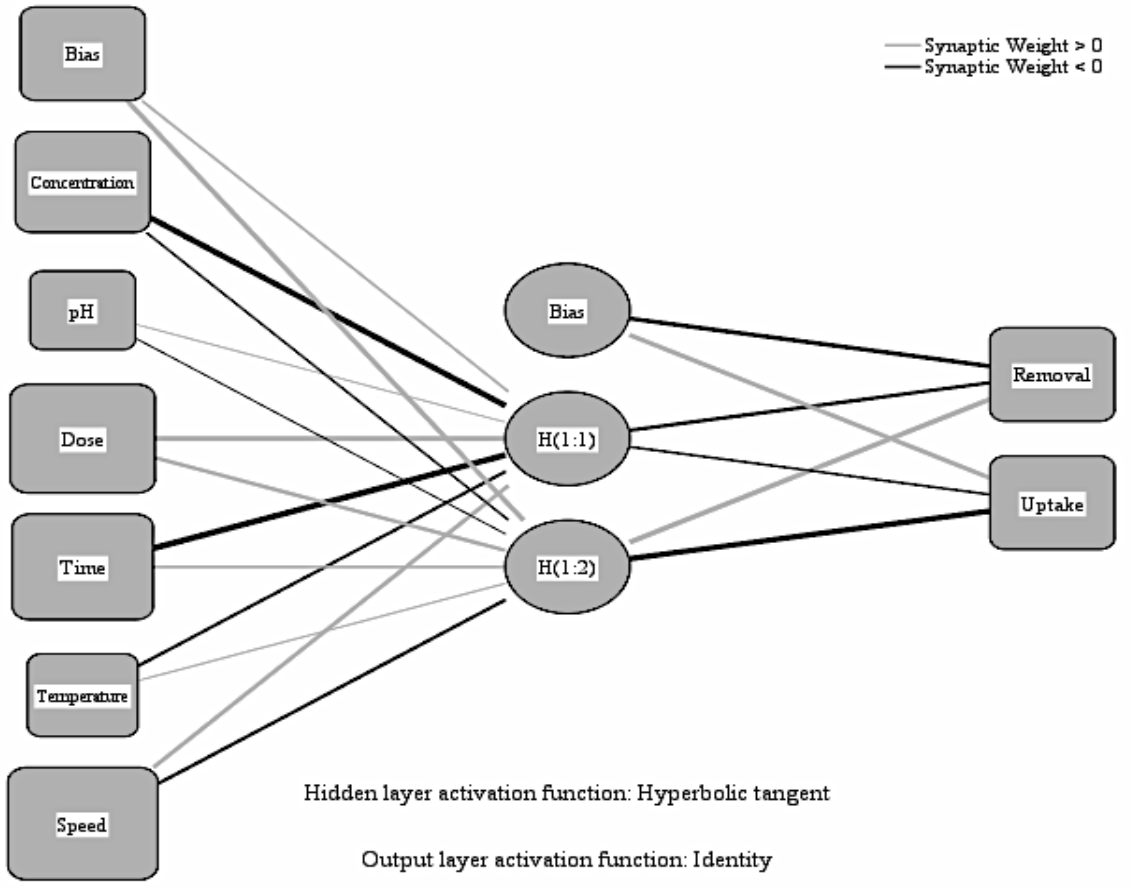

Figure 7: Neural network architecture of As(V) biosorption.

A total of 24 experimental datasets, which were obtained from batch biosorption experiments, were used to develop a three–layer feed–forward neural network model by applying hyperbolic tangent function under the standardized method for scale dependents. Out of these 24 datasets, 75% were used to train the network and remaining 25% were used for testing and validation of the ANN model. There were six neurons (viz., pH, initial As(V) concentration, biosorbent dose, contact time, agitation speed and temperature) in the input layer whereas two neurons in the output layer (removal efficiency and uptake capacity). The 7–3–2 ANN (including bias neuron) model is found to be working satisfactorily with an average relative error of 0.224 and sum square error of 0.588 during testing phase, indicating that the model is able to predict the biosorption efficiency with reasonable accuracy.

The performances of optimized ANN are shown in Figures 8 and 9. In these figures, experimental and ANN–predicted values are compared for both removal efficiency and uptake capacity of As(V); respectively. The values of R^2 (0.951; 0.967) are very close to 1 for each case, shows an excellent agreement between the experimental and the ANN–predicted values.

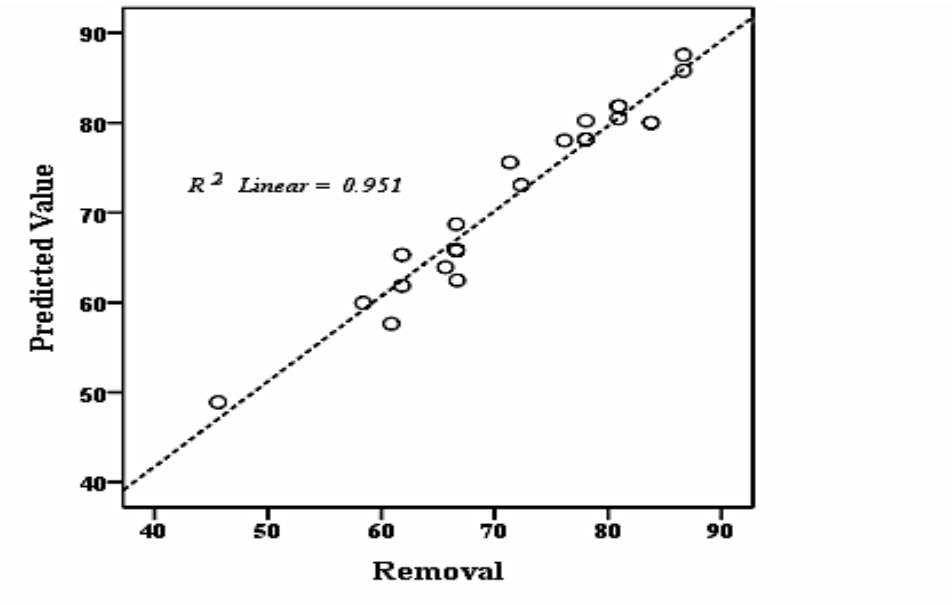

Figure 8: Comparison of the experimental data with those predicted by ANN model [As(V) removal].

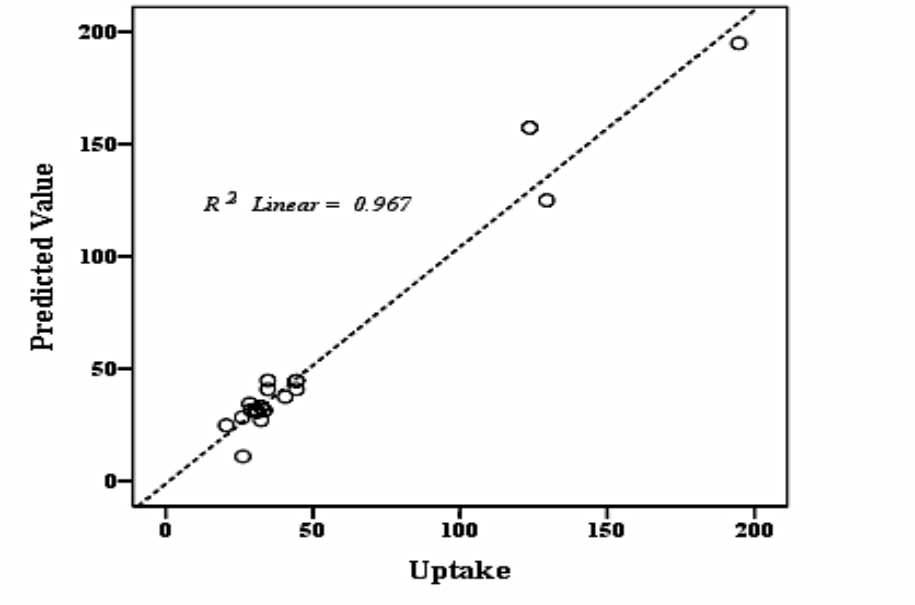

Figure 9: Comparison of the experimental data with those predicted by ANN model [As(V) uptake capacity].

3.8. Importance analysis of the developed ANN

An importance analysis for the developed network was also performed to assess the relative effectiveness of the various operating (input) variables on the output variables [20,33]. In the present scenario (Figure 10), the degree of effectiveness of the input variables on the output variables was found to be in the order of biosorbent dose > initial concentration > agitation speed > solution pH > contact time > temperature for As(V) biosorption. The influence percentages of these variables on the output were 100.0, 99.9, 93.0, 80.6, 65.5 and 16.4%; respectively.

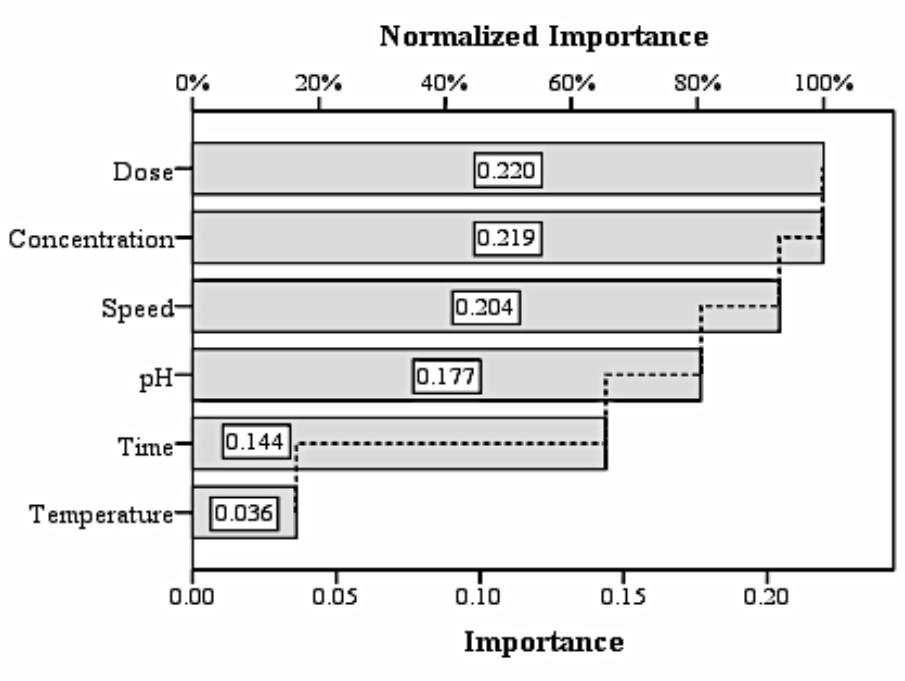

Figure 10: Effect of various experimental (input) parameters on biosorption efficiency.

3.9. Adsorption isotherms and kinetics study

An adsorption (biosorption) isotherm represents the equilibrium relationship between the adsorbate concentration in the liquid phase and that on the adsorbents surface at a given condition. A number of isotherms have been developed to describe equilibrium relationships. In the present study, the Langmuir, Freundlich, Temkin and Dubinin–Radushkevich (D–R) models were used to describe the equilibrium isotherms. The summarization of the isotherm models is shown in Table 1.

From Table 1, it is observed that the Freundlich isotherm showed good fit to the experimental equilibrium data than the Langmuir, Temkin and Dubinin–Radushkevich isotherm equation for arsenic(V) biosorption according to the values of R^2. It is also seen from Table 1 that the Langmuir maximum biosorption capacity q_{max} (µg/g) is 140.8 and the equilibrium constant K_L (L/µg) is 0.0065. The separation factor (R_L) values are 0.755, 0.606, 0.339 and 0.235 while initial As(V) concentrations are 50, 100, 300 and 500 µg/L; respectively. All the R_L values were found to be less than one and greater than zero indicating the favorable biosorption of As(V) by AiBP. The Freundlich constant K_F indicates the adsorption capacity of the biosorbent and the value of K_F is 1.339 µg/g. Furthermore, the value of 'n' at equilibrium is 1.071. The value of n lies in between 1 and 10 also represents a favorable biosorption. From Temkin constant, b_T related to adsorption binding energy for As(V) is found 35.39 J/mol, clearly denying to follow the ion–exchange mechanism. In addition, D–R isotherm shows the value of the adsorption energy (E) of 61.95 J/mol. The estimated value of E (< 8 kJ/mol) has been indicated towards the physisorption process [5,33,34].

The pseudo first–order and pseudo second–order kinetic models were tested to investigate the rate of biosorption of As(V) by AiBP. The linearized form of adsorption kinetics and their constants are presented in Table 2.

From the table, it is confirmed that the arsenic(V) biosorption followed the pseudo second–order reaction. It is also clear from the Table 2 that the pseudo second–order kinetic model showed excellent linearity with high correlation coefficient (R^2 = 0.998) at 100 µg/L As(V) concentration in comparison to the first–order kinetic model. Furthermore, the calculated q_e value also agrees with the experimental value (34.66 µg/g) in the case of pseudo second–order kinetic model.

Table 1: Isotherm data for biosorption of As(V) by AiBP.

Adsorption isotherms	Equations	Parameters (unit)	Values	R^2
Langmuir isotherm	$\frac{1}{q_e} = \frac{1}{q_{\max} K_L C_e} + \frac{1}{q_{\max}}$	$q_{\max}$ (µg/g) K_L (L/µg)	140.8 0.0079	0.982
Freundlich isotherm	$\log q_e = \log K_F + \frac{1}{n} \log C_e$	K_F (µg/g) n	1.399 1.071	0.995
Temkin isotherm	$q_e = \frac{RT}{b_T} (\ln A_T + \ln C_e)$	A_T (L/µg) b_T (J/mol))	272.1 35.39	0.971
D–R isotherm	$\ln q_e = \ln q_{\max} - \frac{1}{2E^2} \times \left[RT \ln \left(1 + \frac{1}{C_e} \right) \right]^2$	$q_{\max}$ (µg/g) E (J/mol)	143.7 61.95	0.911

where q_{max} is the maximum biosorption capacity; K_L, K_F, A_T and b_T are different biosorption constants; n is the heterogeneity factor; E is the mean free energy of biosorption per mole of the adsorbate; T is the temperature (K), and R is the ideal gas constant (8.3145 J/mol.K).

Table 2: Kinetic parameters for biosorption of As(V) by AiBP.

Kinetic Models	Equations	Parameters (unit)	Values	R^2
Pseudo first-order	$\ln(q_e - q_t) = \ln q_e - k_1 t$	q_e (µg/g) k_1(min^{-1})	8.29 0.191	0.822
Pseudo second-order	$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}$	q_e (µg/g) k_2(g/µg.min)	33.3 0.425	0.998

where q_t is the biosorption capacity at time t; k_1 and k_2 are the first-order and second-order rate constant, respectively.

3.10. Thermodynamic study

In order to describe thermodynamic properties of the biosorption of arsenic by AiBP, enthalpy change (ΔHº), Gibbs free energy change (ΔGº) and entropy change (ΔSº) were calculated by using equations shown in Table 3.

Table 3: Thermodynamic parameters for biosorption of As(V) by AiBP.

Thermodynamic parameters	Equations	Values (kJ/mol)
Standard free energy	$\Delta G^0 = -RT \ln K_C$	
298 K		– 3.579
303 K		– 3.889
308 K		– 4.204
313 K		– 4.220
318 K		– 3.069
323 K		– 2.753
328 K		– 2.491
Standard enthalpy change	$\ln K_C = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT}$	– 17.76
Standard entropy change		-45.7×10^{-3}

where K_C is the equilibrium constant; T and R are as defined in previous equation.

From Table 3, it is clear that the reaction is spontaneous in nature as △Gº values are negative at all the temperature studied. Increase in value of △Gº with increase in temperature suggests that lower temperature makes the biosorption easier. Again negative △Hº value implies that the biosorption is exothermic in nature. The type of biosorption can be explained in terms of the magnitude of △Hº. The enthalpy or the heat of adsorption ranging from 2.1 to 20.9 kJ/mol corresponds to physical adsorption whereas ranging from 20.9 to

418 kJ/mol is regarded as chemical adsorption. Therefore, the △Hº value (17.76 kJ/mol) confirms that the biosorption process of As(V) by AiBP occurred due to physisorptions. Furthermore, the negative △Sº suggests a decrease in the randomness at the solid or solution interface during the biosorption process [5,10,23].

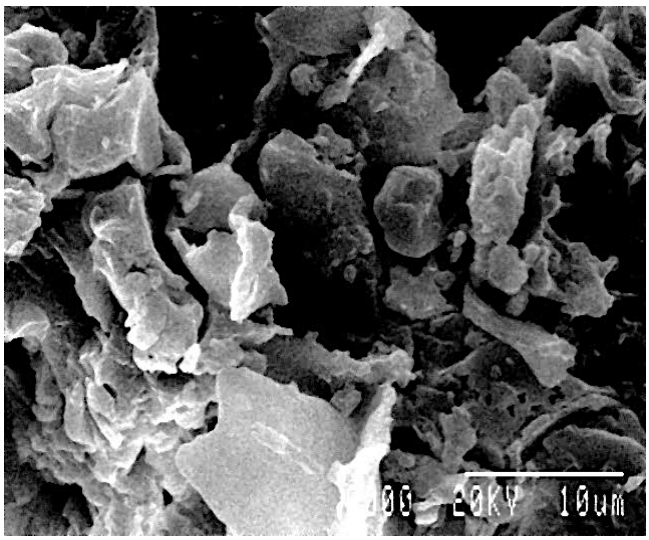

Figure 11: SEM micrograph of AiBP before treatment.

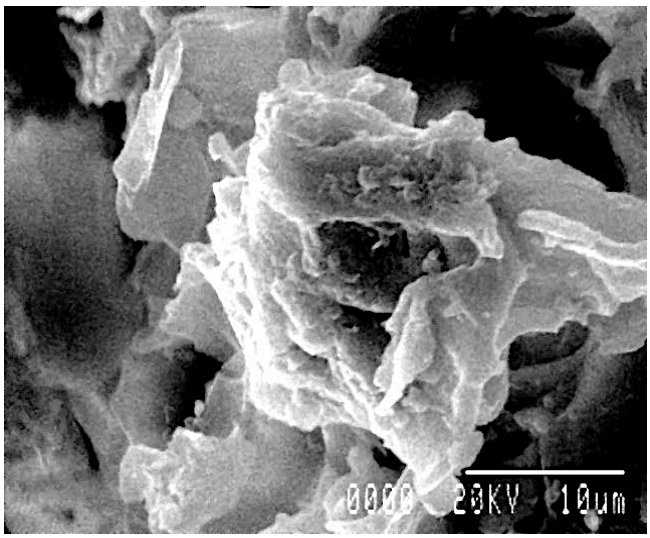

Figure 12: SEM micrograph of AiBP after treatment.

3.11. Scanning electron microscopy (SEM) analysis

SEM analysis is a useful tool for the study of the surface morphology of the biosorbent. The SEM micrographs (20 kV; 10 µm) at 1000 magnifications for AiBP surface before and after batch operation are shown in Figures 11 and 12; respectively. Figure 11 clearly shows the presence of porous, rough, and irregular surface morphology of the unloaded AiBP. The surface roughness of an unloaded biosorbent indicated the availability of a tremendous surface area of AiBP meant for high adsorption capacity in the biosorption process [8,12,33,34]. Contrary to this, after biosorption, the biosorbent surface seemed to have decreased porosity due to the heavy impregnation of As(V) onto the surface of the AiBP biomass (Figure 12).

Conclusion

AiBP was found to be efficacious in the removal of arsenic(V) from aqueous solution. The effect of different process variables (i.e., solution pH, contact time, initial concentration of As(V), AiBP dose, agitation speed and temperature) in batch operations were studied for the biosorption of As(V) on AiBP. Under optimized conditions (AiBP dose 0.25 g, initial arsenic concentration 100 µg/L, contact time 30 min, pH 6.0, temperature 35 ºC, agitation speed 100 rpm); the maximum removal of As(V) was 86.6%. ANN model was applied upon batch experimental values to provide the reasonable predictive performance of the biosorbent. The findings indicate that the model provide reasonable predictive performance (R^2 = 0.951; 0.967) of As(V) biosorption. The model is also found to be working satisfactorily with an average relative error of 0.224 and sum square error of 0.588 during testing phase to predict the biosorption process with reasonable accuracy. In the importance analysis of the developed ANN, it can be observed that the biosorbent dose was the most significant parameter followed initial concentration, agitation speed, solution pH, contact time and temperature. The rate of the biosorption process followed pseudo second–order kinetics while equilibrium data well fitted to the Freundlich isotherm model. The thermodynamic parameters were calculated and it was found that the biosorption process was spontaneous, feasible, and exothermic. The nature of biosorption was physisorption as inferred from the D–R isotherm model. SEM examination also showed that high As(V) uptake favored the physisorption process onto the surface of AiBP.

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