



# Valorization of aquaculture waste in removal of cadmium from aqueous solution: optimization by kinetics and ANN analysis

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## Abstract

Cadmium is one of the most hazardous heavy metal concerning human health and aquatic pollution. The removal of cadmium through biosorption is a feasible option for restoration of the ecosystem health of the contaminated freshwater ecosystems. In compliance with this proposition and considering the efficiency of calcium carbonate as biosorbent, the shell dust of the economically important snail *Bellamya bengalensis* was tested for the removal of cadmium from aqueous medium. Following use of the flesh as a cheap source of protein, the shells of *B. bengalensis* made up of  $\text{CaCO}_3$  are discarded as aquaculture waste. The biosorption was assessed through batch sorption studies along with studies to characterize the morphology and surface structures of waste shell dust. The data on the biosorption were subjected to the artificial neural network (ANN) model for optimization of the process. The biosorption process changed as functions of pH of the solution, concentration of heavy metal, biomass of the adsorbent and time of exposure. The kinetic process was well represented by pseudo second order ( $R^2=0.998$ ), and Langmuir equilibrium ( $R^2=0.995$ ) had better fits in the equilibrium process with  $30.33 \text{ mg g}^{-1}$  of maximum sorption capacity. The regression equation ( $R^2=0.948$ ) in the ANN model supports predicted values of Cd removal satisfactorily. The normalized importance analysis in ANN predicts  $\text{Cd}^{2+}$  concentration, and pH has the most influence in removal than biomass dose and time. The SEM and EDX studies show clear peaks for Cd confirming the biosorption process while the FTIR study depicts the main functional groups ( $-\text{OH}$ ,  $\text{C}-\text{H}$ ,  $\text{C}=\text{O}$ ,  $\text{C}=\text{C}$ ) responsible for the biosorption process. The study indicated that the waste shell dust can be used as an efficient, low cost, environment friendly, sustainable adsorbent for the removal of cadmium from aqueous solution.

**Keywords** Cadmium · Shell · Valorization · Kinetics · ANN

## Abbreviations

AAS	Atomic absorption spectroscopy
ANN	Artificial neural network
SEM	Scanning electron microscope
BSD	<i>Bellamya</i> shell dust
FT-IR	Fourier transformed infrared
EDX	Energy dispersive X-ray
BCF	Bio-concentration factor

## Introduction

The heavy metals in aquatic ecosystem can be detrimental to different organisms depending on the tolerance levels and the complexity of the food web. In course of the different industrial and agricultural processes, the heavy metals are released and eventually contaminate the aquatic ecosystems. For instance, cadmium, considered as one of the important heavy metal pollutant, is added to the aquatic ecosystems through activities in mining, electroplating, battery, paint and ceramic industries, in addition to the natural deposits. The disposal of heavy metals in aquatic systems concentrates as a cascading effect in the trophic levels, frequently recognized in the form of bioaccumulation and biomagnifications (Brooks et al. 2004; Tao et al. 2012). Subsequent entry of heavy metals in the living system can create a chain of physiological, biochemical and genetic changes that are concern from public health viewpoint. In case of cadmium, entry in the human body can damage the kidney, substitute

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calcium in the bones, can cause liver damage, cancer and hypertension (Godt et al. 2006; Bernard 2008). Accumulation of cadmium in the food web causes damage to the wild life and their diversity (Johri et al. 2010; Bernard 2008). As a consequence, the removal of cadmium and similar heavy metals are given priority for the sustenance of the ecological functions of the aquatic ecosystem.

The heavy metal pollutants are non-biodegradable and thus adsorption is considered as one of the convenient way of removal from the aquatic ecosystems. In recent past, several methods based on the principle of ion exchange, chemical precipitation, coagulation, activated charcoal, electrochemical processes and membrane technology alone or in combination have been promoted for the purpose of the heavy metal removal (Du et al. 2011; Liu et al. 2009). However, many of these processes increase the risk of generating secondary pollutants, and therefore pose a concern from ecosystem health and public health viewpoints. As a consequence, the use of biological materials for the removal of these heavy metals is being promoted to minimize the secondary pollutant level in the system as well as cost-effect benefit (Gifford et al. 2006). Application of the biological materials that are component of the ecosystem reduces the possibility of yielding unwanted secondary pollutants. These are substantiated in the observations on the metal adsorption ability of different microorganisms (Hetzer et al. 2006; Sari and Tuzen 2009) and hydrophytes (Sinha et al. 2007) many of which are hyperaccumulator of heavy metals. Application of the aquatic animals in the removal of the heavy metals from the aquatic system has been tested as evident from the studies on living freshwater bivalve (Jana and Das 1997), bivalve shell (Du et al. 2011; Liu et al. 2009; Pena-Rodriguez et al. 2010) crab and arca shell biomass (Dahiya et al. 2008). However, the unregulated use of the living forms of the biological resources may facilitate species invasion, such as many of the plant hyperaccumulators (Sinha et al. 2007). In other instances, such as the case of the freshwater bivalve, despite the metal removal being high (Jana and Das 1997), their potential as aquaculture resources limits their use in bioremediation. Recent studies have demonstrated that calcium carbonate and its derivatives may act as biosorbents of different heavy metals (Du et al. 2011; Pena-Rodriguez et al. 2010). The shells of different molluscs are composed of 95–99% calcite and/or aragonite (principally made up of calcium carbonate) oriented in a matrix of proteins (0.1–5%), and act as natural ceramic with excellent strength and toughness (Kaplan 1998; Boro et al. 2012). The shells of aquatic gastropods can be considered as cheap and available source of calcium carbonate (Hossain and Aditya 2013) and thus can be considered as low-cost biosorbent of heavy metals. In compliance with this proposition, the present study was aimed at evaluation of cadmium removal capacity of shell dust of freshwater gastropod *Bellamya bengalensis*

(Mollusca: Gastropoda: Viviparidae). A common inhabitant of ponds, ditches, rivers and varied types of freshwater wetlands, the operculate snail *B. bengalensis* a prolific breeder and can easily be cultured (Khan and Choudhury 1984). The flesh of the snail is used as a cheap source of protein and the shells are discarded. The discarded shells that are aquacultural waste may be used as cheap source of calcium carbonate and the *Bellamya* shell dust (BSD) can be used in the removal of cadmium from aquatic system.

To promote the biosorption as an effective process, the solution for the optimization is required since the process is influenced by several factors (Witek-Krowiak et al. 2014). Improving the performance of the cadmium biosorption in terms of the process efficiency can be achieved through the optimization, which provides an idea about the best operational condition to yield the best possible response (adsorption) (Witek-Krowiak et al. 2011). Apart from the adsorption kinetics which forms the basis for the selection of the conditions, the modeling of the adsorption process, including those of heavy metals such as cadmium, is carried out to portray the changes in the efficiency depending on the various physico-chemical parameters (Çelekli and Geyik 2011; Çelekli et al. 2013). Since the biosorption process is linked with different variables in a non-linear manner and the mechanism of the process is multifaceted, it is difficult to model using conventional mathematical simulation. In recent past, artificial neural network (ANN) model has been used in describing problems in different fields of chemical and environmental engineering (Çelekli et al. 2012; Hossain et al. 2015; Maghsoudi et al. 2015). In a generalized ANN, the outputs provide an overview of the relative importance of the input parameters (factors influencing the biosorption process) that drive the sorption process (Çelekli and Geyik 2011; Çelekli et al. 2013; Ahmad et al. 2014; Witek-Krowiak et al. 2014). In the present instance, the application of the ANN was meant for deducing the efficacy of the *B. bengalensis* shell dust (BSD) as biosorbent of cadmium, under varied physico-chemical conditions that influence the adsorption process. Thus, the cadmium absorption efficiency of BSD was judged through multilayer perceptron in ANN model, apart from description of the equilibrium and kinetic models. The results will be useful in supplementing the required information in utilizing the BSD, a waste generated from aquaculture as a biosorbent and thus in bioremediation of heavy metal contaminated aquatic ecosystem.

## Materials and methods

### Preparation of the metal solution

The experiments were initiated through preparation of a cadmium chloride stock solution of 1000 mg L<sup>-1</sup> using double

distilled water and further working solutions were prepared by appropriate dilution. The pH of the solution was adjusted by adding  $\text{HNO}_3$  (0.1 N) and  $\text{NaOH}$  (0.1 N) as required in course of the experiments. All the inorganic chemicals that have been used in these experiments were purchased from Merck India Ltd., India.

### Preparation of the *Bellamyia* shell dust

The waste shells of the snail *B. bengalensis* were collected from local fish markets in Burdwan, India. Following procurement, the remnant tissue portion and dusts in the shells were cleared in warm water and dried in sunlight for 2 days. Next, the shells were placed in hot air oven for 2 days at  $60^\circ\text{C}$  and kept in plastic zipper bags. For preparation of the shell dust, the shells were pulverized in mortar and pestle to fine granules and dried in oven for 2 days at  $60^\circ\text{C}$ . The granules were then sieved through  $500\ \mu\text{m}$  and consequently through  $200\ \mu\text{m}$  net, to yield two different sized granules, respectively,  $500\text{--}200\ \mu\text{m}$  and  $\geq 200\ \mu\text{m}$ . Initially, the larger-sized granules adsorbed less amount of cadmium and thus were not studied further. The ash content of the dust was  $72.34\%$  and the water content capacity was  $1859.0\ \text{mg g}^{-1}$ . The specific surface area, pore volume and pore diameter were determined by the Brunauer–Emmett–Teller (BET) method using Quantachrome Autosorb automate with nitrogen gas (version 4.0). The surface area of the biosorbent was  $10.143\ \text{m}^2\ \text{g}^{-1}$ , while the pore volume was  $0.079\ \text{cc g}^{-1}$  and the pore diameter was  $3.336\ \text{nm}$ .

### Batch sorption procedure

The batch sorption experiments were performed in a 250-mL Erlenmeyer's flask that contained 100 mL solution of the particular cadmium ion concentration at required pH and relevant amount of snail shell dust (BSD). The flasks were sealed with wax paper and shaken in a shaking incubator (Lab Companion, SI-300R, India) at a required temperature at 150 rpm for required time. After shaking for particular time, the solution of the flasks was centrifuged at 2000 rpm for 15 min and the supernatant was taken for estimation of metal concentration by atomic absorption spectroscopy (GBC Avanta 1.3, India). The influence of different levels of pH on biosorption equilibrium was studied through changing the pH of the solution in range of 2–7. The effect of contact times between solution and the BSD were monitored by varying it from 10 to 80 min. For equilibrium studies, the metal ion concentrations were used in a varied concentration of  $25\text{--}1000\ \text{mg L}^{-1}$ , and for optimum biosorption study, the BSD biomass was varied between 200 and 1000 mg. The amount of Cadmium ion adsorbed on the BSD was estimated following the equation (Sharma et al. 2011):

$$q_e = (c_o - c_e) v/m,$$

where  $q_e$  is the amount of metal adsorbed,  $v$  is the volume of solution,  $m$  is the mass of adsorbent,  $c_o$  is the initial concentration of the Cd solution and  $c_e$  is the equilibrium concentration of the Cd solution.

### Estimation of the metal content in tissues and shells by AAS

For estimation of metal, required amount of *Bellamyia bengalensis* tissue/shell of a particular dose was taken into Teflon container for microwave digestion. 4 mL of *aqua regia* was added and container was placed in microwave oven at 450 W and the sample was digested for 7 min. 4 mL of hydrogen peroxide was then added to the mixture and was again digested for another 7 min. It was diluted with distilled water, and filtered in a 25-mL volumetric flask. Final volume was made up with distilled water. Reagent blank was prepared in the same way. The final samples were then estimated for cadmium by atomic absorption spectroscopy (GBC Avanta 1.3, India) at  $228.8\ \text{nm}$  wavelength.

### Analyses of FT-IR absorbance spectra of BSD

IR spectra of protonated or  $\text{Cd}^{2+}$  loaded BSDs were recorded in a (Perkin-Elmer FTIR, Model RX1) Fourier transform infrared (FT-IR) spectroscopy. Samples of 100 mg of KBr disks contain 1% of the finally ground powder of each sample were prepared less than 24 h before recording.

### The scanning electron micrograph (SEM) study of BSD

Raw and metal-adsorbed BSDs were dried and prepared for scanning electron microscopic studies. The samples were attached with the stubs by both side cello tapes and gold plated in a sputter coater before use in the SEM. Electron acceleration potential of 20 kV was used for the microscopic observations. Photographs were taken in a HITACHI S530 scanning electron microscope at  $800\times$  magnification.

### Energy dispersive X-ray (EDX) analysis

For energy dispersive X-ray analysis, the samples were studied by field emission gun-based scanning electron microscope with energy dispersive X-ray analysis (SEM/EDX) (Quanta model by FEI Co., the Netherlands) for the morphological as well as for the presence of elemental information on the samples. The SEM studies were performed at 30 kV in the low vacuum mode. EDX spectra were taken from the area corresponding to the SEM image shown in the in-set. The EDX spectra were taken in the 'region mode' with the bombardment of energetic electrons for duration of electron of 100 s.

## Equilibrium modeling

The adsorption equilibria were studied for the estimation of maximum cadmium biosorption by the BSD. For the equilibrium study, the experiments were performed at different initial cadmium ion concentration (25–1000 mg g<sup>-1</sup>).

Langmuir and Freundlich isotherms were used in describing the equilibrium between adsorbed cadmium ions on the BSD ( $q_e$ ) and in solution ( $c_e$ ) at a particular temperature. The parameters of the Langmuir equation (Langmuir 1918) were determined from a linear form of the following equation:

$$c_e/q_e = (c_e/a) + 1/ab,$$

where  $a$  is the maximum amount of metal ions/unit mass of adsorbent to form a monolayer and  $b$  is the equilibrium constant. The correlation coefficient of the linear plot of  $c_e/q_e$  against  $c_e$  indicates the applicability of the Langmuir modeling in the experiment (Tan and Hameed 2017).

Freundlich equation (Freundlich 1906) is described as below:

$$q_e = k_f c_e^{1/n},$$

where  $k_f$  and  $1/n$  are the Freundlich constants indicating the adsorption capacity and intensity, respectively. The linear model of the isotherm can be expressed logarithmically as in the following:

$$\log q_e = 1/n(\log c_e) + \log K_F,$$

where the value of  $K_F$  and  $n$  can be determined from the y-intercept and slope of the plot of  $\log c_e$  against  $\log q_e$ . The linear plot of  $\log q_e$  against  $\log c_e$  indicates the applicability of the Freundlich modeling in the experiment.

Temkin's isotherm model is an empirical equation comprises a factor that clearly indicates the adsorbent–adsorbate interactions. Through overlooking the lower and higher value of concentrations, it assumes that heat of adsorption of the interacting molecules in the layer would decrease linearly rather than logarithmically. The model is given by the following equation (Foo and Hameed 2010; Tanzifi et al. 2017; 2018; Ghaedi and Vafaei 2017):

$$q_e = RT/b_T \ln (A_T c_e),$$

or,

$$q_e = RT/b_T \ln A_T + RT/b_T \ln c_e,$$

where  $A_T$  is the Temkin isotherm equilibrium binding constant (L g<sup>-1</sup>),  $b_T$  is the Temkin isotherm constant,  $R$  is the universal gas constant (8.314 J<sup>-1</sup> mol<sup>-1</sup> K), and  $T$  is the temperature at 298 K.  $A_T$  and  $B_T$  values are obtained from  $q_e$  versus  $\ln c_e$  plot for the adsorbent.

Dubinin–Radushkevich (D–R) isotherm was used in investigation of Cd adsorption nature based on the equilibrium data. The isotherm is expressed in linear form as:

$$\ln (q_e) = \ln (x_m) - \beta \epsilon^2,$$

where  $\beta$  and  $x_m$  (maximum Cd adsorption capacity) are the constants obtained from the plot of  $\ln q_e$  vs.  $\epsilon^2$  (Polanyi potential). ' $\epsilon$ ' is calculated from the following equation:

$$\epsilon = RT \ln (1 + 1/c_e),$$

where  $R$  is the universal gas constant (J mol<sup>-1</sup>) and  $T$  is the absolute temperature in Kelvin and ' $\beta$ ' is associated to the adsorption free energy ( $E$ ). Adsorption energy is calculated with the following equation (Tanzifi et al. 2017):

$$E = \sqrt{1/ - 2\beta}.$$

## Kinetic modeling

The pseudo second order model for prediction of biosorption is not suitable for a long period of adsorption process (Ho and McKay 1999) and the pseudo second order equation:

$$dq_t/dt = k_2 (q_e - q_t)^2,$$

or

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

where  $q_e$  is the amount of adsorbed metal ion on biosorbent at equilibrium,  $q_t$  is the amount of adsorbed metal ion on biosorbent at time ' $t$ ', and  $k_2$  is the second order rate constant (g mg<sup>-1</sup> min<sup>-1</sup>). A linear plot of  $t/q$  vs.  $t$  indicates whether this model of biosorption is applicable for this case.

## Artificial neural network (ANN) model

ANN is a non-linear mathematical model that is inspired by the structural and functional aspects of neuron in exploration of a group of input data (through training and testing) as function of output data (including relative error) and to envisage the performance of the given system (Cavas et al. 2011). A three-layered ANN model is used in the analyses of the complex interactions in finding the pattern of experimental data. Multilayer perceptron uses supervised learning technique in relating output responses to the expected responses by regulating the loads of the input signals and remind it. The memory is used in the next time of data feed when the output response will be much closer to the wanted one after the same input signals are delivered (Shanmugaparakash and Sivakumar 2013). The weight of any input signal is calculated by the following equation:

$$W_i = \sum_{j=1}^n w_{ij} x_j,$$

where  $w_{ij}$  is the corresponding load of connection between each neuron ‘ $j$ ’ in input layer and each neuron ‘ $i$ ’ at hidden layer and  $x_j$  is the value of input signal ‘ $j$ ’ at input layer (Çelekli et al. 2013). For a particular function, the output is predicted from the following equation:

$$y_k = f(w_i + b_j),$$

where  $w_i$  is the sum weight of each neuron and  $b_j$  is the bias, i.e., constant weight of a neuron representing the relative error (Çelekli and Geyik 2011). In predicting the biosorption of cadmium (output), the combined effect of the variables—(1) pH, (2)  $\text{Cd}^{2+}$  concentration, (3) biosorbent dose and (4) contact time were used in the analysis. Sixty-nine experimental sets were used in training, validation and testing the ANN model by SPSS® 20, trial version.

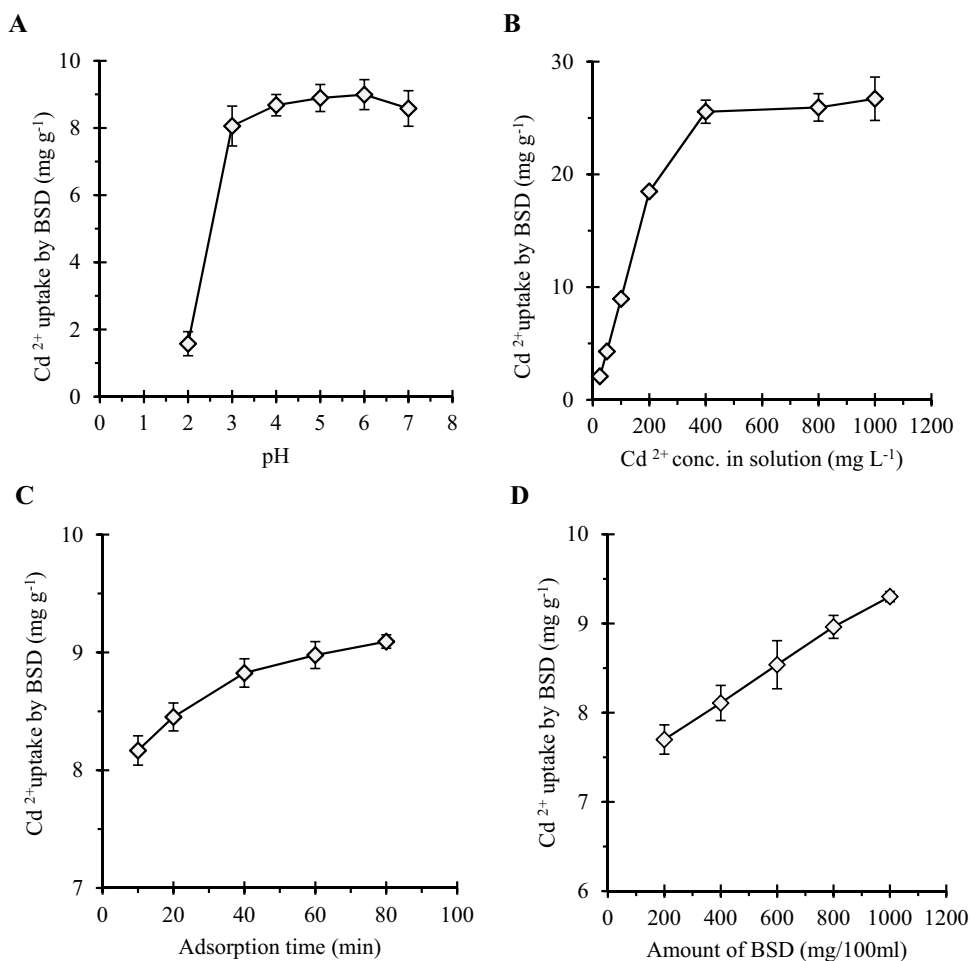
## Results and discussion

### Bioaccumulation of cadmium on shell dust

#### Effect of pH of the solution on batch sorption

The biosorption procedure was maintained over the experimental range of pH 2–7. The sorption procedure was affected by the pH of the medium in two ways—metal solubility and total charge of the functional groups of the biosorbent. The optimum pH, at which the procedure shows the highest adsorption for the biosorbent was estimated to be 6 which is nearer to the environmental pH of most of the freshwater aquatic system. At high pH, that is, at alkaline condition, precipitation of the metal takes place, and at low pH due to high protonation, metal sorption capacity decreases (Guo et al. 2008). The experiment was carried out in 100 mL of solution having  $100 \text{ mg L}^{-1}$   $\text{Cd}^{2+}$  and 100 mg of the BSD at  $30^\circ\text{C}$  in reference to varying pH of the solution. Figure 1a indicates the pH-dependent adsorption of the metal ion by BSD as the metal sorption was negligible at pH 2 and it

**Fig. 1** **a** Effect of pH of the solution on biosorption of cadmium on BSD at  $30^\circ\text{C}$ , initial cadmium ion concentration of  $100 \text{ mg L}^{-1}$ , contact time of 60 min and biosorbent dose of 1 g per 100 mL solution. **b** Effect of initial cadmium ion concentration of solution on biosorption on BSD at  $30^\circ\text{C}$ , pH 6, contact time of 60 min and biosorbent dose of 1 g per 100 mL solution. **c** Effect of time on biosorption of cadmium on BSD at  $30^\circ\text{C}$ , pH 6, initial cadmium ion concentration of  $100 \text{ mg L}^{-1}$ , and biosorbent dose of 1 g per 100 mL solution. **d** Effect of biosorbent dose on biosorption of cadmium on BSD at  $30^\circ\text{C}$ , pH 6, initial cadmium ion concentration of  $100 \text{ mg L}^{-1}$ , contact time of 60 min





increases dramatically as increase in pH and at pH 6 it shows the highest adsorption. The metal sorption declined as the pH increase further. In a particular pH range, most metal sorption is enhanced with pH, increasing to a certain value followed by a reduction on further pH increase (Guo et al. 2008; Pavasant et al. 2006; Semerciöz et al. 2017).

#### Effect of initial metal ion concentration on batch sorption

Increase in metal ion concentration added on metal adsorption as concentration of the ion increases. 25–1000 mg L<sup>-1</sup> ion concentrations were used for the study taking seven different doses in series. The 25 mg L<sup>-1</sup> initial metal ion concentration showed the lowest adsorption while the 400 mg L<sup>-1</sup> concentration showed the highest adsorption and the adsorption remained same in further increase in Cd<sup>2+</sup> ion increase in the solution (Fig. 1b). The initial metal ion concentration can modify the metal removal efficiency over a combination of factors, the availability of specific surface functional groups and ability of surface functional groups to bind metal ions (Taty-Costodes et al. 2003; Pino et al. 2006)

#### Effect of contact time on batch sorption

The sorption potentials of the BSD over time were monitored from 10 min to 20, 40, 60, 80 min using 100 mL of 100 mg L<sup>-1</sup> Cd<sup>2+</sup> at pH 6 (Fig. 1c). At the beginning, metal adsorption was less due to more binding sites remained free when treated for the short period of time and increased rapidly as the treatment time increases. It showed lowest adsorption when treated for 10 min and increased over time to saturate at 60 min and after that the uptake remained almost same. The variation in uptake of the cadmium ions with time was used in fitting the kinetic models.

#### Influence of the biosorbent dose on batch sorption

The cadmium biosorption potential of the BSD augmented over its amount increased in treating the metal solution. The more the amount of biosorbent the more the free binding sites or exchanging group to adsorb the metal ion from the solution. For a 100 mg L<sup>-1</sup> metal ion concentration, the increase in biosorbent resulted increase in metal ion adsorption and above a certain dose it remained same or slightly higher due to comparatively higher number of free sites and lesser number of metal ions (Fig. 1d) (Al-Anber and Matouq 2008; Ghodbane et al. 2008).

#### FT-IR study of the shell dust

To study the mechanism of cadmium removal and the main functional groups responsible for Cd<sup>2+</sup> binding, the FTIR

spectrum of the BSD was performed. IR spectra of protonated and cadmium loaded BSD are shown in Fig. 2a, b, respectively. A peak at 3424 cm<sup>-1</sup> is indicating the presence of hydroxyl (–OH) groups. Strong band at 2921 cm<sup>-1</sup> is due to C–H stretching frequency (Semerciöz et al. 2017) and peak at 1634 cm<sup>-1</sup> is due to C=O stretching mode of the primary and secondary amides (Reddy et al. 2010). Weak band at 1462 cm<sup>-1</sup> is attributed to aromatic C=C and the strong band at 1041 cm<sup>-1</sup> is due to C–O stretching of alcoholic groups (Blazquez et al. 2011). FTIR study reveals that OH, CH, CO, and C=C may be the responsible groups in the biosorption process. The FTIR of metal loaded BSD shows that distinct shift of the above mentioned bands as well as change in intensity informs some ion exchange behavior of the BSD.

#### The scanning electron micrograph (SEM) analysis of the shell dust

The surface structure of the free and cadmium-loaded BSD was analyzed under scanning electron microscope. The scanning electron micrographs of the dried BSD before and after the Cd<sup>2+</sup> treatment at 800× magnification are shown in Fig. 3a, b, respectively. It indicates the irregular morphological structure of the particles and lamellar stratified surface of the BSD. The SEM image of the biosorbent after exposure to the Cd<sup>2+</sup> shows a spongy layer indicating surface precipitation occurred during the sorption (Du et al. 2011). In case of cadmium sorption by CaCO<sub>3</sub> compound generally follows surface precipitation due to similar ionic radii (Prieto et al. 2003; Perez-Garrido et al. 2007) of divalent calcium and cadmium.

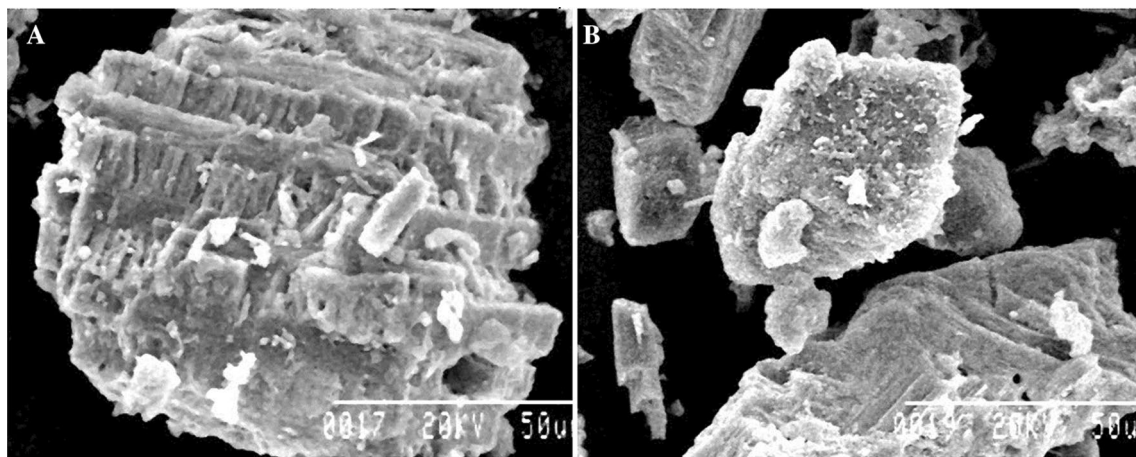
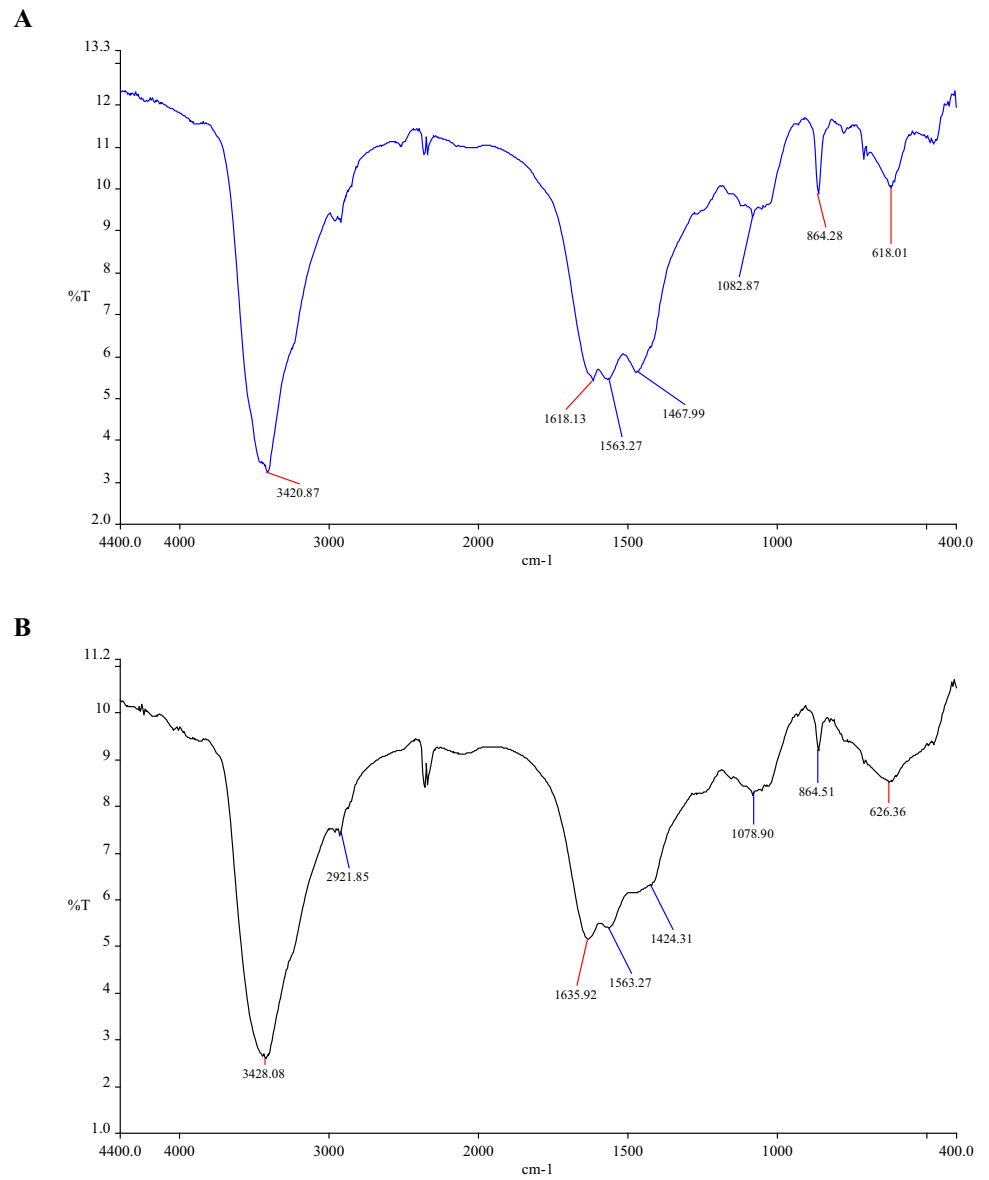
#### Energy dispersive X-ray (EDX) analysis

The surface structure of the shell dust makes accessible a large unadsorbed surface area for the cadmium ion of the solution. The elemental profile of the BSD before and after the treatment of the cadmium solution was estimated using the energy dispersive X-ray analysis. The projecting peaks in the EDX spectra correspond to CKα, OKα, AlKα, SiKα, PKα, CaKα, CaKβ, FeKα, etc., in the untreated BSD and furthermore to it a CdLα peak in treated BSD (Fig. 4a, b). Both the treated and untreated BSD display strong peaks corresponding to calcium. In addition, the calcium of BSD may facilitate adsorption of cadmium because of the similarities in ionic radii that enhance ion exchange (Purkayastha et al. 2014).

#### Equilibrium modeling

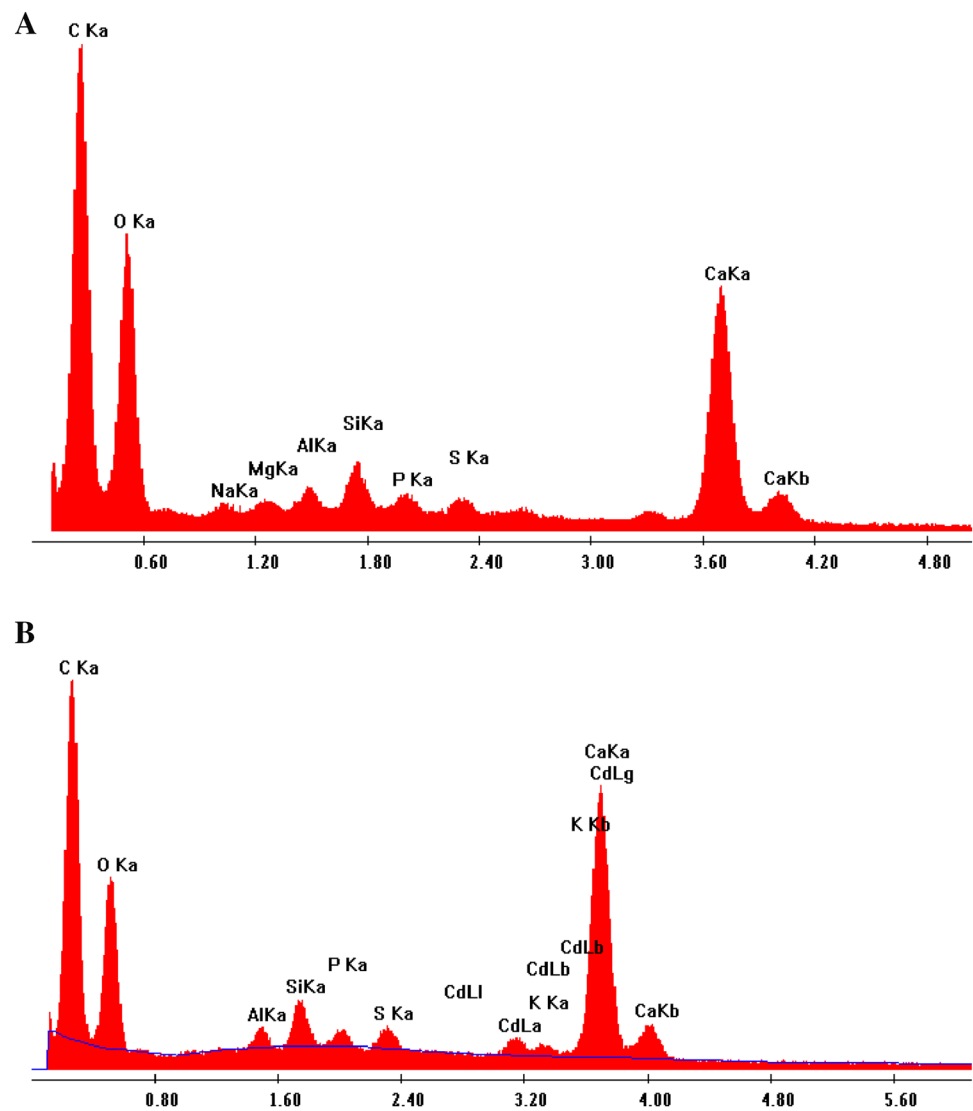
The biosorption isotherm is important in waste water treatment as it implies estimation of biosorption capacity of

**Fig. 2** FTIR absorbance spectra of snail shell dust (BSD) before (a) and after (b) biosorption of cadmium



**Fig. 3** Scanning electron micrograph (SEM) of snail shell dust (BSD) before (a) and after (b) the biosorption of cadmium (magnification at ×800)

**Fig. 4** EDX analyses for elemental composition in the snail shell dust for BSD before (a) and after (b) biosorption of cadmium clearly showing the peaks for cadmium, in the treated shell dust, indicating the adsorption of the metal on the shell dust

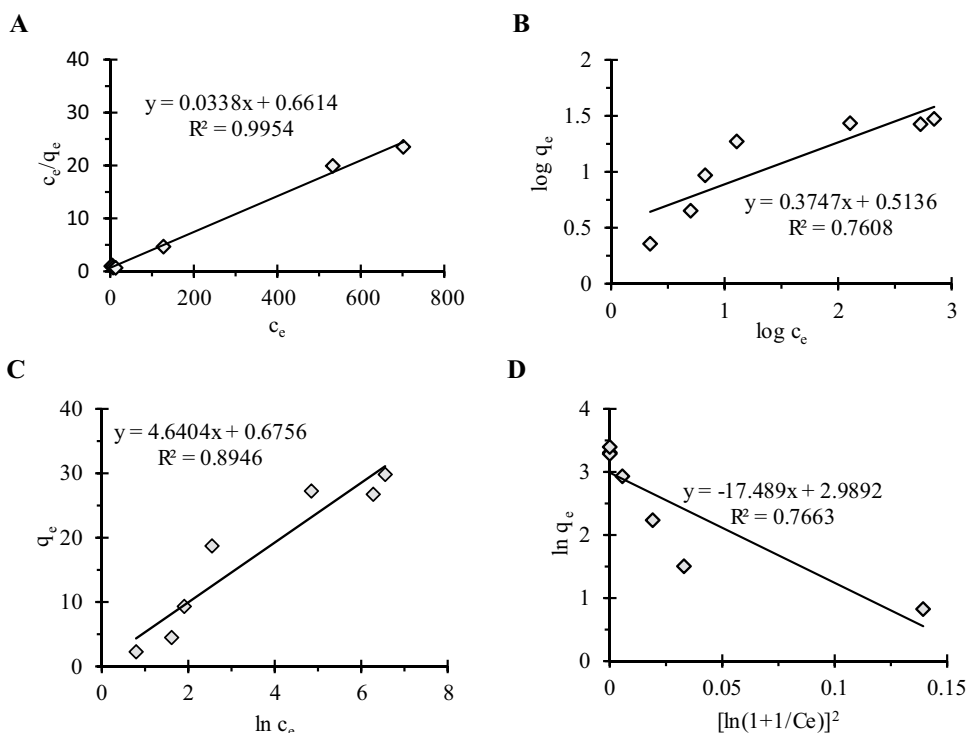


the adsorbent. The linear representations of Langmuir and Freundlich isotherm of cadmium adsorption at 30 °C are given in Fig. 5a, b, respectively. The correlation coefficient and constants obtained from the equations are presented in Table 1. The correlation of determination is high in Langmuir equation ( $R^2=0.995$ ) contrast to Freundlich equation ( $R^2=0.760$ ) (Fig. 5). It indicates that Langmuir model is more suitable for describing the biosorption equilibrium of  $\text{Cd}^{2+}$  on the snail shell dust. High  $q_{\text{max}}$  value ( $30.33 \text{ mg g}^{-1}$ ) from Langmuir equation indicates the biosorption potential of the material. The value of  $b$  (0.04) (Table 1) indicates  $b$  the affinity of the binding sites and the energy of adsorption (L/mg) (Blazquez et al. 2011). The equilibrium models of

cadmium biosorption support that under optimum conditions (pH 6, biosorbent dose of 1 g,  $\text{Cd}^{2+}$  concentration of  $100 \text{ mg L}^{-1}$  and 60 min time period)  $30.33 \text{ mg g}^{-1}$  is the maximum biosorption capacity of BSD. The Temkin isotherm shows  $B_T=533$ ,  $T_A=1.15$  and the  $R^2=0.896$  showing moderate fit. The R–D equation ( $R^2=0.766$ ) for the biosorption shows the value of the adsorption energy ( $E$ ) is  $13.89 \text{ J/mol}$  that corresponds to chemisorption type of uptake based on ion exchange (Wu et al. 2012; Markou et al. 2016). Maximum cadmium biosorption capacities of similar low cost biosorbents are shown in Table 2. BSD is mainly composed of calcium carbonate, degradation of which, if any, will not yield unwanted compound to the ecosystem.



**Fig. 5** **a** Langmuir isotherm, **b** Freundlich isotherm, **c** Temkin isotherm and **d** Dubinin–Radushkevich isotherm plot for biosorption of cadmium on BSD (pH 6, temperature 30 °C, biosorbent = 1 g, cadmium ion concentration = 100 mg g<sup>-1</sup>)



**Table 1** Coefficients of the Langmuir, Freundlich, Temkin isotherm and Dubinin–Radushkevich models for cadmium biosorption by BSD

Langmuir coefficients			Freundlich coefficients			Temkin coefficients			D–R coefficients		
$q_{max}$ (mg g <sup>-1</sup> )	$b$ (L mg <sup>-1</sup> )	$R^2$	$n$	$k_F$	$R^2$	$T_A$ (L mg <sup>-1</sup> )	$b_T$	$R^2$	$q_{max}$ (mg g <sup>-1</sup> )	$E$	$R^2$
30.33	0.049	0.995	2.67	1.67	0.760	1.15	533	0.894	9.211	13.89	0.766

**Kinetic modeling**

The kinetic model is necessary for determination of optimal condition of the biosorption process. For the evaluation of differences in sorption process, the kinetics of metal uptake were described by pseudo second order model (Ho and McKay 1999). The linear plots obtained from pseudo second order model at 100 mg L<sup>-1</sup> initial Cd<sup>2+</sup> concentration, pH 6 and at studied temperature are shown in Fig. 6a, b, respectively. The rate constants, expected metal uptake and correlation coefficients have been described in Table 3. The pseudo second order reaction in biosorption is based on the sorption capacity on the solid phase. From Fig. 6b and Table 3, the correlation coefficient in the pseudo second order reaction ( $R^2 = 0.998$ ) is high and the calculated metal

adsorption (9.26 mg g<sup>-1</sup>) is much nearer to the expected value (9.302 mg g<sup>-1</sup>). These suggest that the biosorption process is based on the pseudo second order model.

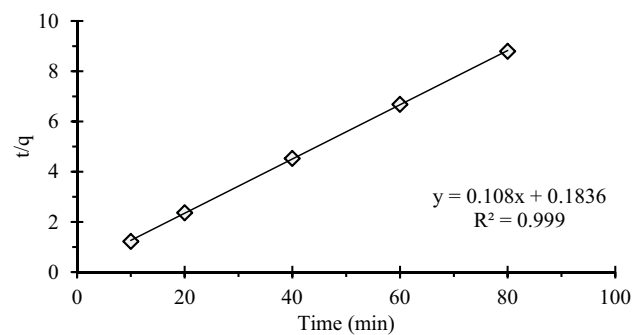
**Optimization results through ANN model**

The ANN model established in this study comprised three layers as shown in Fig. 7a consisting of a hyperbolic tangent transfer function at hidden layer and a linear transfer function at output layer. The input layers have four predictors, viz. biomass, pH, Cd concentration and time, the hidden layer and the output layer have one response variable, the amount of Cd absorbed (Table 4) (Çelekli et al. 2012, 2016). The weight of the neurons was used in the study of the relative influence of each of the input variables on

**Table 2** Comparative data of biosorption capacities ( $q_{\max}$ —maximum metal uptake capacity) for cadmium by different biosorbents

Adsorbent	$q_{\max}$ (mg g <sup>-1</sup> )	References
Raw corn stalk	3.39	Zheng et al. (2010)
Olive waste	6.55	Azouaou et al. (2008)
Brewer's yeast	10.17	Cui et al. (2010)
Corn cob	4.73	Ramos et al. (2005)
Rice straw	13.9	Ding et al. (2012)
Wheat bran	15.71	Nouri et al. (2007)
Castor seed hull	6.98	Sen et al. (2010)
Bamboo charcoal	12.08	Wang et al. (2010)
Walnut tree sawdust	5.76	Yasemin and Zek (2007)
Chitosan/bentonite	12.05	Arvand and Pakseresh (2012)
Coconut copra meal	4.99	Ho and Ofomaja (2006)
<i>S. platensis</i>	73.64	Çelekli and Bozkurt (2011)
Sweet potato	18.00	Asuquo and Martin (2016)
<i>P. pubescens</i> biochars	14.70	Zhang et al. (2017)
<i>Bellamyia</i> shell dust (BSD)	30.33	The present study

the biosorption process, i.e., on output layer. In the present instance, the sum of squares error (SSE error, a measure of the predicted and the observed values; Çelekli and Geyik 2011) is 0.467; and the relative error being 0.056, justifying the suitability of the multilayer perceptron ANN model. The regression equation of the output (predicted adsorption through ANN model,  $y$ ) and the corresponding target (observed through the experiments,  $x$ ) complied with the linear form as  $y = 0.37 + 0.95x$  (Fig. 7b). The high ( $R^2 = 0.943$ ) coefficient of determination fits well with the experimental dataset with that of the ANN model of the system (Yang et al. 2011; Khataee et al. 2011). Upon comparison, the data complied well with both the pseudo second order (Fig. 6) as well as the ANN model (Fig. 7b), with high values of coefficient of determination. Among the predictors, the pH of the solution (0.347) appeared to be comparable to the cadmium concentration (0.339) in terms of the relative importance in shaping the adsorption process, similar to those observed for the dye removal by *Chara contraria* (Çelekli and Geyik 2011). The contact time and the biomass bear comparative low influence in the adsorption, contrast to those observed for the dye removal by the walnut husk (Çelekli et al. 2016).

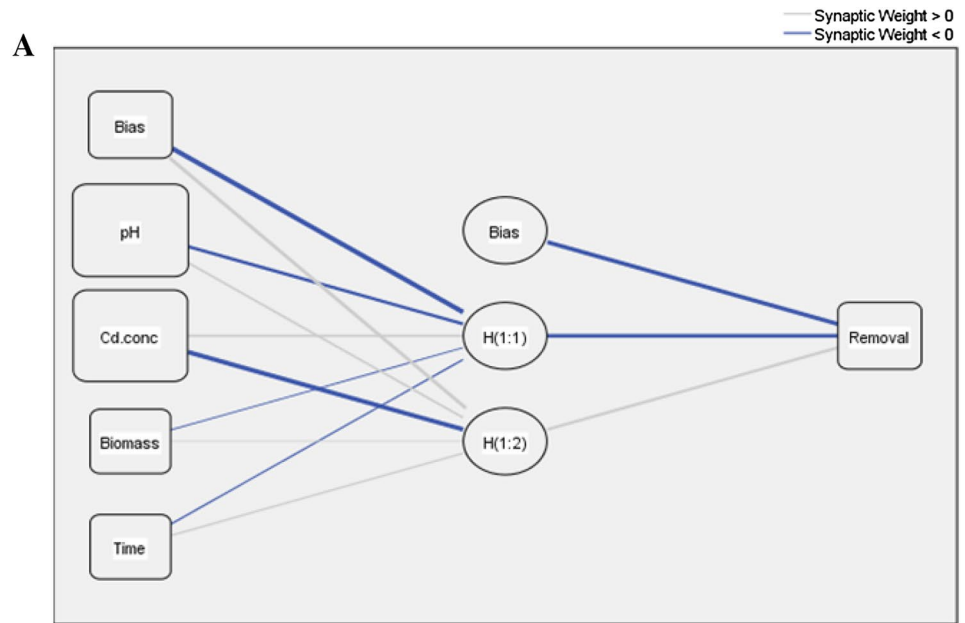
**Fig. 6** Pseudo second order plot for biosorption of cadmium on BSD (pH 6, temperature 30 °C, biosorbent = 1 g, cadmium ion concentration = 100 mg g<sup>-1</sup>)**Table 3** Rate constant and equilibrium uptake for cadmium binding by BSD at pH 6 and initial metal ion concentration of 100 mg L<sup>-1</sup>

Metal	Second order model		
	$k_2$ (g mg <sup>-1</sup> min <sup>-1</sup> )	$q_{e \text{ cal.}}$ (mg g <sup>-1</sup> )	$R^2$
Cadmium	0.064	9.26	0.998

## Conclusion

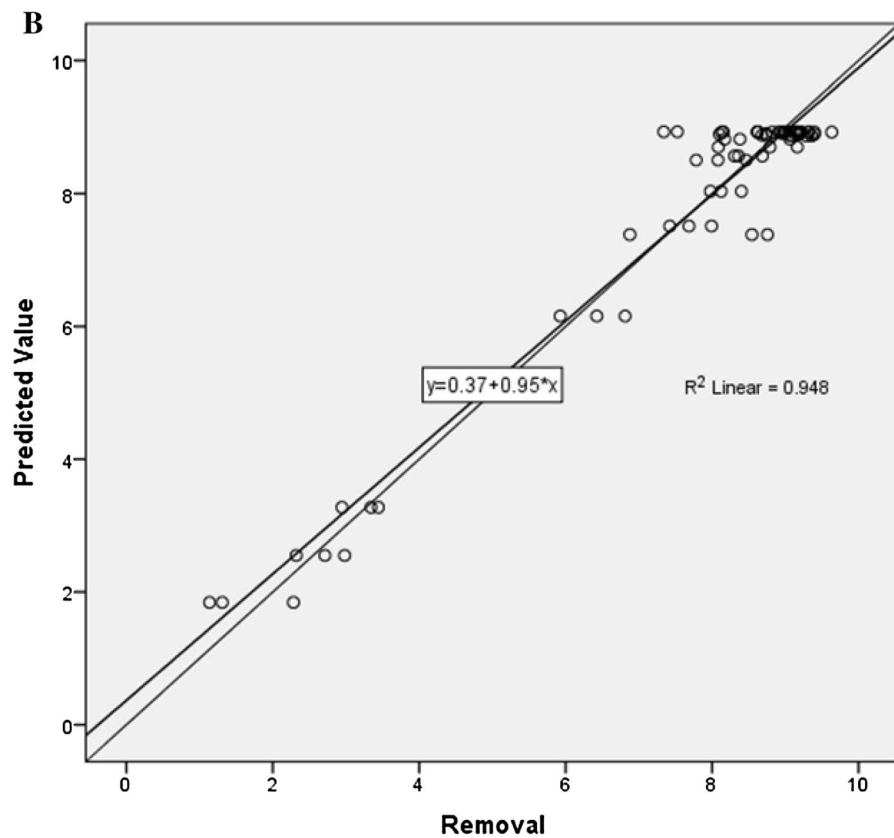
The biosorption process works as function of pH of the solution, biomass, Cd ion concentration and time of contact. The biosorption study indicates that at pH 6, the maximum cadmium adsorption capacity of BSD is 30.33 mg g<sup>-1</sup>. The isotherm model follows Langmuir model ( $R^2 = 0.995$ ) better than Temkin isotherm ( $R^2 = 0.896$ ), R–D ( $R^2 = 0.766$ ) and Freundlich model ( $R^2 = 0.760$ ). The main functional group responsible for chelation is OH, C=O, C=C and C–C, as supported by FTIR analysis. EDX study shows clear peaks for cadmium in the treated biomaterial, conveying further support of clear adsorption. The biosorption process followed pseudo second order ( $R^2 = 0.998$ ) kinetics. A multilayer perceptron model in artificial neural network (ANN) model successfully portrayed biosorption of cadmium ion on the LSD with high correlation coefficient ( $R^2 = 0.943$ ) between predicted and observed removal. BSD can be considered as efficient, low cost and environment friendly biosorbent for cadmium bioremediation, opening a new aspect of the economic value of shells of the freshwater snail *B. bengalensis*, apart from its value as cheap protein source.

**Fig. 7** Basic design (a) of the multilayer perceptron artificial neural network (ANN) model used in the study along with the regression line (b) endorsing the linearity of predicted and observed cadmium removal by BSD



Hidden layer activation function: Hyperbolic tangent

Output layer activation function: Identity



**Table 4** Basic information of the artificial neural network used in describing the biosorption of cadmium by BSD

Network information			
Input layer	Covariates		
	1	pH	
	2	Cd concentration	
	3	Biomass	
	4	Time	
	Number of units <sup>a</sup>	4	
	Rescaling method for covariates	Standardized	
	Hidden layer(s)	Number of hidden layers	1
		Number of units in hidden layer 1 <sup>a</sup>	2
		Activation function	Hyperbolic tangent
Output layer	Dependent variables		
	1	Removal	
	Number of units	1	
	Rescaling method for scale dependents	Standardized	
	Activation function	Identity	
	Error function	Sum of squares	

<sup>a</sup>Excluding the bias unit

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