

Biosorptive Removal of Toxic Contaminant Lead from Wastewater

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Lead is one of the most toxic heavy metals due to its high bioaccumulation potential. This paper reports a native biomass (*Vanda* species) for the effective and rapid removal of lead from aqueous solution. The parameters studied under the batch removal process were lead concentration, biomass dose, contact time and pH. The identified plant originated biomass was found to remove more than 90 % of available Pb(II) within 5 min and the complete removal was observed within 1 h. The optimum pH identified was 3.5, contact time was 60 min and the optimum biomass dose was 1 g. To identify chemical functional group responsible for effective adsorption the FTIR analysis was carried out which indicated the involvement of amine, hydroxyl and carboxyl group for the adsorption. The SEM and EDX analyses showed high uptake of Pb(II) which got accumulated with in the cell structure. The adsorption isotherms studied were Langmuir and Freundlich isotherms. The maximum adsorption capacity as derived by Langmuir adsorption isotherm was 51 mg/g. In continuous flow removal experiments the break through point was observed on passing 17.8 L of 100 mg/L Pb(II), the exhaust point was noted at 50 L and the uptake capacity was 83.33 mg/g. Based on the result the identified biomass (*Vanda* species) is rapid effective and possess high removal capacity. The biomass is amenable for industrial application also.

Keywords: Pb(II), Biosorption, Water, Removal, *Vanda* species.

INTRODUCTION

Industrial metals such as cadmium, chromium, copper, mercury, nickel, lead and zinc have vastly contaminated the earth since the mid-1900s [1]. Mobilization of these metals and their increased deposition in aquatic environment occur through dumping of industrial waste, mine tailings and landfill run off [2]. Over the years it was realized that the lead was the most toxic heavy metals due to its persistent nature in the environment which increases the health risk [3]. Lead is being introduced into the environment from since the beginning of industrialization. The past human activities include the combustion of fossil fuels like leaded gasoline and use of lead based paint. Currently lead and lead compounds have been used in a wide variety of products including paints, ceramics pipes, plumbing materials, shoulders, gasoline, batteries and cosmetics [4,5]. Lead is particularly dangerous to the young children. In their book, 'Lead Wars', Rosner and Markowitz [6] described the ways in which even small exposure can interfere with a child's brain development and cause lasting learning challenges. Lead, even at microscopic levels in drinking water causes anemia, hepatitis and nephritic syndrome [7]. Severe

damage to kidneys, nervous system, reproductive system, liver and brain are one of the cause of lead poisoning [8]. The upper limit for lead in drinking water recommended by WHO is 0.05 mg/L [9].

Several conventional methods have been used to remove metal ions from aqueous solutions, including oxidation, reduction, precipitation, membrane filtration, ion exchange and sorption [10]. The biosorption has many advantages over the conventional method such as the reusability of biomaterial, low operating cost, selectivity for specific metal, short operation time and little chemical sludge [11]. The distinct advantages of the biosorption technology are its low-cost and the effectiveness in quickly reducing the concentration of heavy metals ions to very low levels with high efficiency [12]. Several adsorbent have been used for the removal of lead in aqueous solution such as barley straws [13], *Symphori carpusalbus* [14], *Caulerpa lentilifera* [15], *Moringa oleifera* [16], Pine bark [17], Sugar beet pulp [18], *Sargassam ilicifolium* [19], *Lactarius scrobiculatus* [1] and saw dust [20].

For identifying better biosorbent *Vanda* sp. plants were collected from the forest area of Chhattisgarh state, India. This *Vanda* sp. prefer full sun exposure and bright light with warm

temperatures and high humidity levels. The long trailing roots of these *Vanda* sp. are capable of drawing nutrients and the required moisture from the atmosphere. Height of the plant is maximum 30-40 cm and it blooms grey flower during rainy season.

The complete plant was used for the experiment. To obtain optimum condition, batch studies for different parameters were performed like pH, contact time, biosorbent density, initial metal ion concentration and presence of competing ions. Characterization of biosorbent was done to explain adsorption. Adsorption isotherm studies were carried to understand the potential of metal biosorption. The continuous flow method was employed to identify the potential of the biosorbent for industrial application.

EXPERIMENTAL

Preparation: The *Vanda* sp. was collected from the *Basia latifolia* tree from forest area. It was sun dried for one month and then dried in hot air oven. After adequate drying the biomass was grinded and passes by the standard sieve size of 1.18 mm. The sieved biomass was pre-treated with 0.01 N HCl and then washed with double distilled water until the filtrate attained the pH of 6.8. The treated biomass was then dried in hot air oven for 24 h at 60 °C. This process removed external impurities and primed the biomass for the better adsorption. This prepared biosorbent material were kept in air tight container.

Batch study procedure: The batch equilibration method was conducted in 250 mL of Erlenmeyer flask containing 50 mL of Pb(II) solution having concentration between 10 to 1000 mg/L. Adsorbent dosage investigated was between 1 to 5 g. The pH of the solution was adjusted by adding either 0.01 M NaOH or 0.01 M HCl acid solution. During the equilibration process, the solution was agitated between 5 to 180 min at room temperature (25 to 30 °C). The whole process was simultaneously conducted in double distilled water as a blank sample. After due equilibration the agitated solution was filtered. The initial and final concentration of the analyst was determined by a fast sequential atomic absorption spectrophotometer (AAS240FS, Varian). After optimization the effect of interfering radicals was also studied.

Column study procedure: The continuous flow mode study was carried out in a glass column packed with the pre-treated biomass. The internal diameter and height of glass column was 5 and 70 cm, respectively. The plant biomass (60 g) was loaded in the glass column which was supported by the glass beads. Lead(II) solution having an initial concentration of 100 mg/L was pumped through the column in an up-flow mode with a fixed flow rate of 13 mL/min using peristaltic pump. The influent solution was maintained at optimized pH using 0.01 M NaOH and 0.01 M HNO₃. The effluent solution were collected and analyzed for Pb(II) concentration by fast sequential atomic absorption spectrometer (AA240FS, Varian).

RESULTS AND DISCUSSION

The use of orchid biosorbent (*Vanda* sp.) for the removal of lead ion from the aqueous solution gave favorable results. The individual parameters studied under the batch mode are also made.

Effect of pH: The pH of the solution is reported to play a significant part in metal biosorption process. It is because the metal ion solubility and biosorbent total charge are two aspects which affects biosorption process, since protons can be adsorbed or released [21]. This behaviour is reported to depend on the functional groups present on the adsorbent. To understand the same, the effect of hydrogen ion concentration was studied between 2 to 5 pH. The results show a marked impact of the pH. Accordingly, the adsorption capacity of biomass was found 62.5 % at pH 2. At pH 3, the adsorption capacity of biomass was found to increase to 99.25 %. On further increase of pH the removal percentage was found to decrease to 78 and 74 % at pH 4 and 5, respectively (Fig. 1).

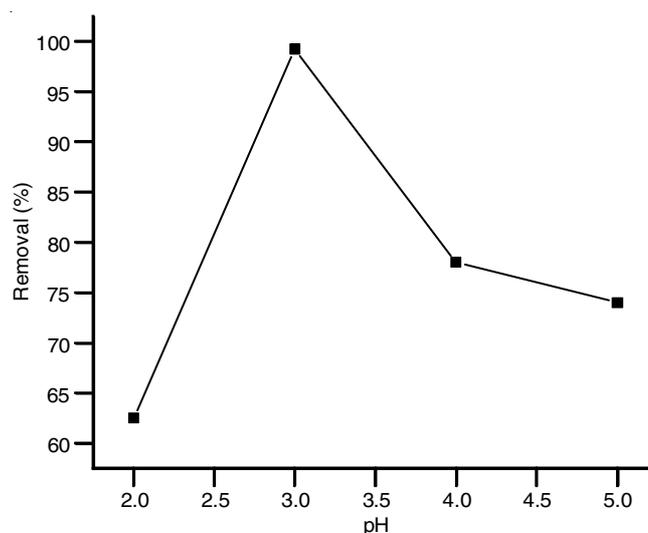


Fig. 1. Effect of pH in the removal of Pb(II)

This behaviour can be explained as a role of the pH in adsorption process. At the lower pH, the biosorption was low due to the higher density of H⁺ ion which inhibit the attraction between metal ion and functional group present in surface of biosorbent. The initial increase in pH decreases H⁺ ion which gives a scope for the adsorption of biomass. However, beyond the pH of 3.5 a decreasing adsorption trend was observed. Higher pH values were not studied to avoid the formation of metal hydroxide. Based on the above, the optimum pH value for removal capacity of biosorbent was taken as 3.5.

Effect of biosorbent dose: The amount of biosorbent used for the treatment studies is an important parameter, which determines the potential of biosorbent to remove metal ions at a given initial concentration [22]. To study the effect of biosorbent dose on removal capacity, the experiments were carried out by using biosorbent dose from 1-5 g biomass on a fixed initial concentration of Pb(II) at 50 mg/L, and the optimized pH of 3.5.

Results show that the percentage removal capacity increased from 99.19 to 99.89 % on increasing the dose from 1 to 4 g of adsorbent (Fig. 2). This is explainable because of the greater availability of the binding sites. Based on the results the optimum dose of adsorbent to remove metal ion was fixed as 1 g/50 mL solution.

Effect of contact time: To attain the maximum adsorption equilibrium condition the effect of contact time was studied by

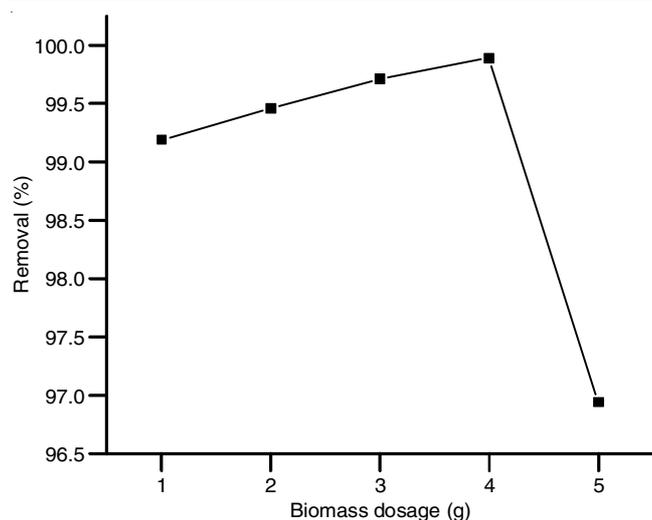


Fig. 2. Effect of biosorbent dose in the removal of Pb(II)

varying the time from 5 min to overnight. The results showed that the equilibrium was attained rapidly within 5 min with a removal of 91.84 % of the lead. The adsorption increased to 99.27 % within 60 min of contact time (Fig. 3). Beyond 60 min only a marginal increase was noted. On leaving the contents overnight it was noted that there was desorption of the adsorbed lead ions. Therefore, the optimum contact time was taken as 60 min. This behaviour of a rapid adsorption initially can be attributed to the abundant availability of active sites present in surface area of adsorbent. These active sites are rapidly occupied in the beginning of the equilibration time. The gradual increase of adsorption capacity appears to be due to the secondary adsorption sites located in the inner surface area of the adsorbent. These sites are slow in reacting with the metal ion [23].

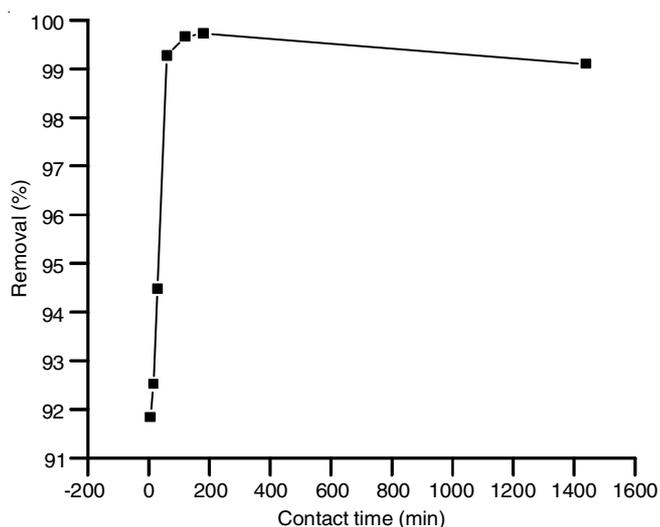


Fig. 3. Effect of contact time in the removal of Pb(II)

Effect of initial Pb(II) ion concentration: Effect of initial concentration of Pb(II) was studied by varying the Pb(II) concentration from 10 to 1000 mg/L at a constant pH of 3.5, contact time of 60 min and 1 g of biosorbent dose. The result showed that the initial concentration of Pb(II) ion in the solution play a major role in the adsorption process. This acted

as a driving force to overcome the mass transfer resistance between the aqueous and solid phase as reported by Dang *et al.* [24].

At the initial concentration of 1 to 10 mg/L the percentage of removal was 100 %. The adsorption remained almost complete till 50 mg/L (> 99.8 %). On increasing the lead concentration from 50 to 100 mg/L, the percentage of removal was found to decrease from 99.8 to 99.2 %. Even on a very high concentration of 1000 mg/L this biosorbent was able to remove 91.55 % of Pb(II) (Fig. 4). These data indicate that initially there is complete removal due to the higher interaction between Pb(II) ion concentration in solution and the active adsorption sites in the biomass. However on higher Pb(II) concentration the saturation of binding sites results in lower removal percentage but the overall biosorption uptake capacity increases. Thus, this biosorbent can be expected to perform well even on occasional spikes in the influent concentration during the column mode experiments.

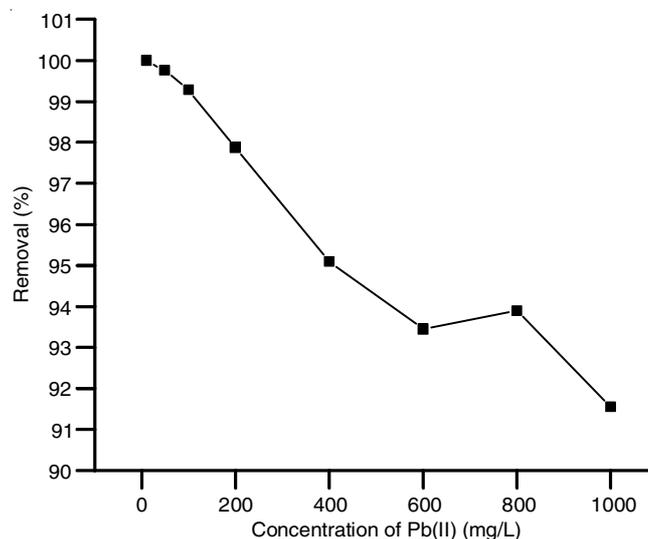


Fig. 4. Effect of initial Pb(II) concentration in the removal of Pb(II)

The biosorption capacity was calculated by the following equation:

$$Q = \frac{(C_i - C_f) \times V}{M} \quad (1)$$

where, Q is the biosorption uptake capacity (mg/g), C_i is the initial concentration of Pb(II) (mg/L), C_f is the final concentration of Pb(II) (mg/L), V is the volume of solution (L) and M is the weight of biomass taken for adsorption (g). Using this equation on multiple replicates the final biosorption capacity of this biosorbent was established as 45 mg/g for Pb(II) ions.

Effect of common co-ions: The effect of presence of co-ions in removal of Pb(II) by biosorbent was investigated by varying the concentration of various co-ions. In cationic co-ions the concentration was varied from 100 to 500 mg/L at 3.5 pH. The result shows that the presence of Cu(II) and Cr(IV) does not affect the removal of Pb(II) ion at concentration as high as 500 mg/L. The other cations *i.e.* Cd(II), Ni(II), Mn(II), Fe(II), Zn(II), also had a negligible effect on the removal Pb(II) (less than 0.5 %).

In case of anions a fixed high concentration (500 mg/L) of anions was used in all experiments. Here it was also observed that all commonly occurring anions *viz.* chloride, nitrate, fluoride, sulphate and phosphate had a very small effect on the Pb(II) removal percentage ($> 0.1\%$). Hence, it can be concluded that *Vanda* sp. can be considered as one of the best biosorbent for the removal of Pb(II) in the presence of any interfering ions even at a high concentrations which can be normally encountered in natural water or even in industrial water.

FTIR analysis: Fourier transform infrared (FTIR) spectroscopy is a versatile tool used to detect the presence of functional group in a biomass. FTIR is also used to determine the significant changes that may take place in a fresh (unexposed) and exhausted biomass. Accordingly the FTIR was used to identify the changes in IR adsorption patterns. Fig. 5(a) shows the spectrum of fresh biomass. There it can be observed that a strong broad band was at 3430.79 cm^{-1} . This corresponds to the presence of -OH stretch band of phenols. A medium peak was observed at 2920.78 cm^{-1} which can be attributed to stretching OH band of primary or secondary amines. Another strong band observed at 1629.30 cm^{-1} is due to carbonyls (C=O) stretching. The medium band located at 1514.05 cm^{-1} was characteristic of stretching primary amines (N-H) or stretching of aromatics (C-C) in ring. The medium peak observed at 1036.82 cm^{-1} could be assigned to stretching of aliphatic amines (C-N). The medium band observed at 555.16 cm^{-1} was due to the presence of stretching of alkyl halides (C-Br).

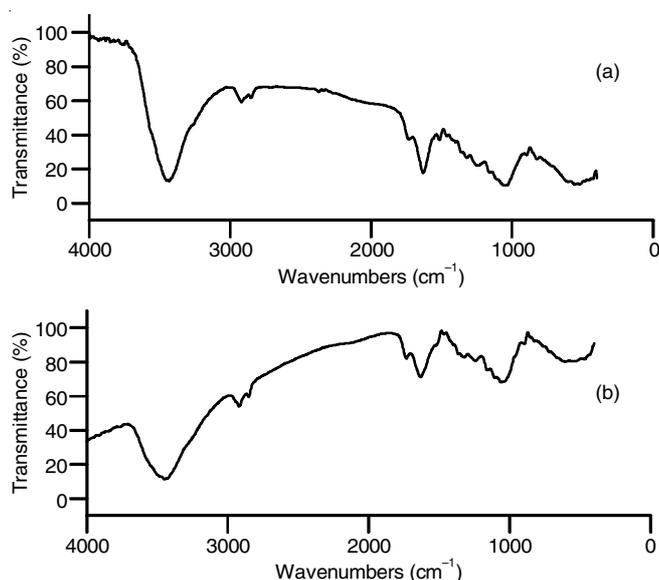


Fig. 5. FTIR spectrum of *Vanda* sp. (a) Fresh biosorbent (b) Exhausted biosorbent

Significant variation was observed in the FTIR spectrum of exhausted biomass (Fig. 5b). Pandey *et al.* [25] reported that the functional groups located on the surface of biomass are responsible in metal chelation. Accordingly, it was observed that the OH band of phenol was shifted from 3430.79 and 2920.78 cm^{-1} to 3445.65 and 2919.82 cm^{-1} , respectively. This shifting indicates that the hydroxyl group was changed from multimer to monopolymer or even to dissociative state [26]. Hence it can be concluded that the degree of the hydroxyl polymerization in *Vanda* sp. biomass gets decreased by binding

with Pb(II) ions and hence a displacement of -OH can be considered as a major binding mechanism operating here. The band observed at 1732.33 and 1631.47 cm^{-1} in the lead exposed biomass and the shifting of these bands further indicated the involvement of N-H of amines and carboxylic groups in the adsorption process. This shift in the wavelength is due to metal binding process taking place on the surface of the biomass [27].

Hence, it was concluded that the hydroxyl, amine and carboxylic group are the main functional groups present in *Vanda* sp. Which are responsible for the Pb(II) adsorption.

SEM analysis: Scanning electron microscopy (SEM) determines multi-phase and high resolution to evaluate surface structure of a sample. The SEM analysis was determined by SEM Model-ZEISS EVO 18. SEM images for texture analysis and morphology of biomass of before and after adsorption of biomass are shown in Figs. 6 and 7. Fig. 7 exhibits that the fresh biomass contains large number of vacant alveoli and uneven surface texture. On lead exposure the SEM image showed that the vacant alveoli get completely filled and appear stretched also on account of high adsorption of Pb(II). Based on this fact it can be suggested that the active adsorption of Pb(II) adsorption and its subsequent deposition in the alveoli is a mechanism of defense of the biomass to protect itself from the toxic effects of Pb(II).

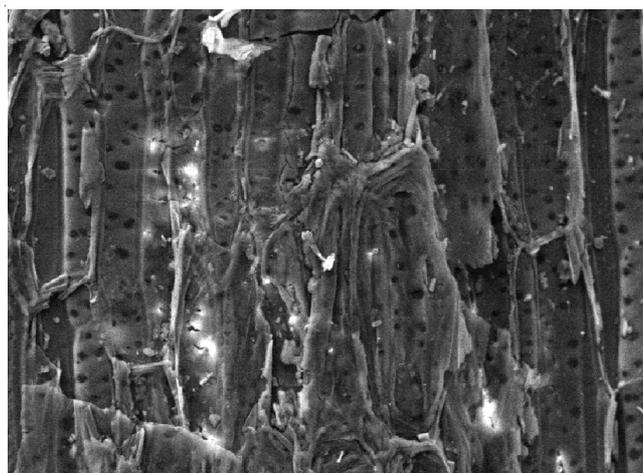


Fig. 6. SEM structure of fresh *Vanda* sp. biosorbent

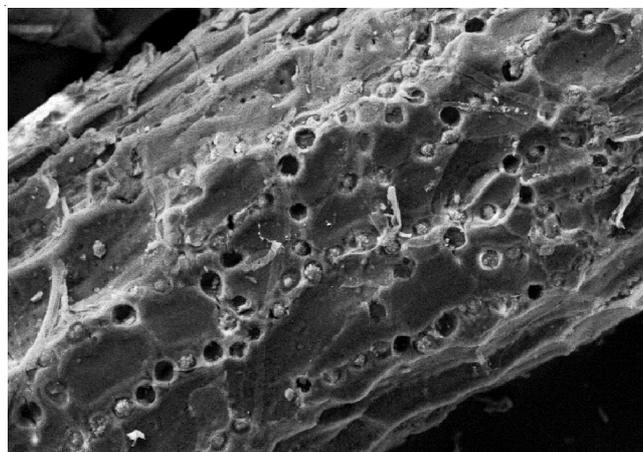


Fig. 7. SEM structure of Pb(II) exhausted *Vanda* sp. biosorbent

The efficient biosorption and better fit to the Freundlich isotherm further supports the multilayer biosorption of Pb(II) by *Vanda* sp.

Energy dispersive X-ray analysis of biosorbent: Energy dispersive X-ray (EDX) analysis gives the elemental information about the compositional structure of a sample. EDX analysis can provide the chemical proof for the incorporation of the adsorbate in the molecular structure of the adsorbent. Accordingly the EDX analysis was performed on un-exposed and exhausted biomass after Pb(II) adsorption. The results of the EDX analysis are shown in Figs. 8 and 9. The results clearly show that Pb(II) signal was absent in the fresh biomass. The exhausted biomass show a dominant presence of the Pb(II) by its characteristic signal of Pb(II) in the exhausted biomass. This confirms that the Pb(II) ion are actively adsorbed by *Vanda* biosorbent. Clear signals corresponding to the Ca^{2+} and Mg^{2+} were detected in fresh biomass and but the same were absent in the exhausted biomass. This establishes a selective exchange of Ca and Mg ions by *Vanda* sp. to chemisorb the Pb(II) ions. Such type of ion exchange mechanism was also explained by Iqbal *et al.* [28,29] for Pb(II) ion and for Zn(II).

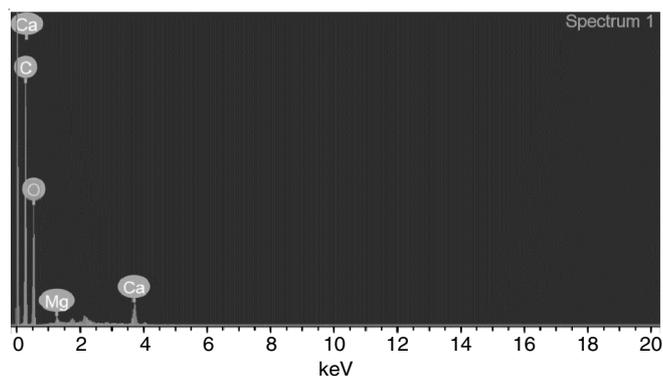


Fig. 8. EDX analysis of fresh biosorbent

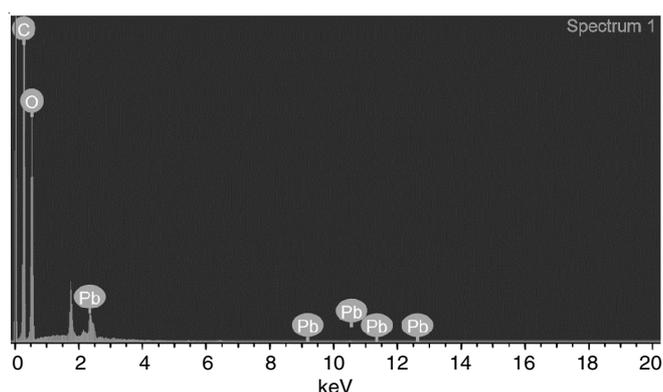


Fig. 9. EDX analysis of Pb(II) exhausted biosorbent

Adsorption isotherm: The efficacy and governing principles for the transfer of Pb(II) from the liquid phase to solid phase was studied under the adsorption isotherm determination. The transport process involved in adsorption was studied under optimized batch adsorption as described in the material and methods.

The Langmuir model uses a simple model of the solid surface adsorption to derive an equation for an isotherm. This model assumes that adsorptive solid possesses a uniform surface

with specific adsorbent sites. It further assumes that these sites do not react further with adsorbed molecules and thus form a monolayer of adsorbed molecules. The data of the equilibrium studies for adsorption of Pb(II) ions may follow the following form of Langmuir model [30] which states

$$\frac{C_e}{q_e} = \frac{C_e}{Q_o} + \frac{1}{Q_o b} \quad (2)$$

where, C_e is the equilibrium concentration (mg/L) and q_e is the amount adsorbed per specified amount of adsorbent (mg/g), Q_o is the equilibrium constant and b is the amount of adsorbate required to form a monolayer. Hence, a plot of C_e/q_e vs. C_e should be a straight line with a slope $(1/b)$ and an intercept as $1/Q_o b$.

The Freundlich isotherm is an empirical equation that is based in the adsorption of metal uptake on the heterogeneous surface of adsorbent is given by equation [31]

$$q = K_f C_e^n \quad (3)$$

Taking log and rearranging equation:

$$\log q = \log K_f + n \log C \quad (4)$$

where K_f and n are the Freundlich constants characteristic. K_f and n are indicators of biosorption capacity and biosorption intensity, respectively. A plot of $\log q$ versus $\log C_e$ should be straight line with a slope $1/n$ and an intercept of $\log K_f$ (Fig. 10). The Langmuir and Freundlich isotherm equation were calculated and given in Table-1. Accordingly in this experiment, the Langmuir isotherms value of Q_o was 51.02 mg/g and constant value of b was 0.05 L/mg. The value of $1/n$ and K in Freundlich isotherm was 0.43 and 116.14 mg/g, respectively. This specifies that Pb(II) ion adsorbed more strongly by biosorbent. Multilayer adsorption process indicated by the result as the correlation coefficient value of Freundlich isotherm equation ($R^2 = 0.98$) was higher than Langmuir isotherm equation ($R^2 = 0.82$).

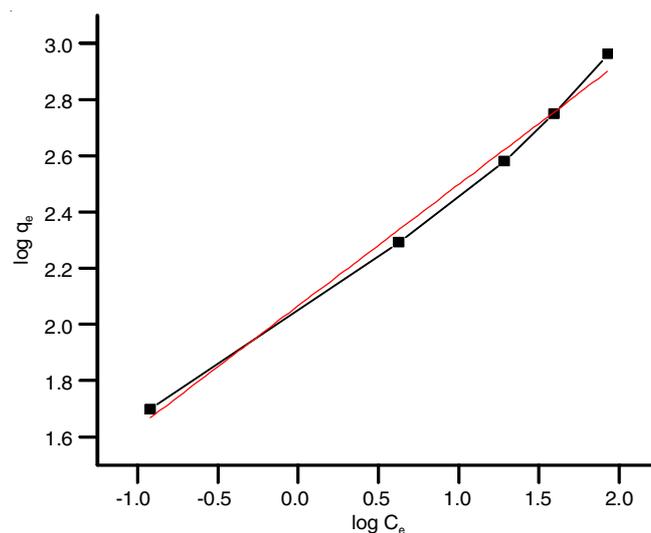


Fig. 10. Freundlich isotherm equation for adsorption of Pb(II)

TABLE-1 LANGMUIR AND FREUNDLICH ISOTHERM CONSTANT VALUES					
Langmuir isotherm			Freundlich isotherm		
Q_o (mg/g)	b (L/mg)	R^2	K (mg/g)	$1/n$	R^2
51.05	0.052	0.82	116.14	0.043	0.98

Efficacy of the *Vanda* sp. biosorbent for continuous flow mode removal of lead(II): Study of the target biosorbent for its suitability for continuous flow mode removal is important for large scale removal application. Accordingly continuous flow experiment was conducted to verify the efficiency of the biosorbent for the removal of Pb(II). The packed bed column (PBC) is generally used to improve the contact between the stationary and mobile phases. Hence, in this research also the packed bed column was used. In packed bed column experiments the empty bed contact time (EBCT) is an important property which informs about the time lag between the start of the influent and obtaining the effluent at the other end of the packed bed column. This time is useful in determining the window period when treated water shall not be available in a new column. In this experiment the empty bed contact time was calculated by dividing the bed volume with flow rate. Accordingly, the calculated bed volume ($\pi r^2 L$) of column was 651.74 mL and effluent run in fixed flow rate was 13 mL/min. From above, the empty bed contact time calculated was 50 min.

The breakthrough point indicates that the concentration of effluent passes through column reached beyond the described permissible limit. The exhausted point was that where concentration of influent and effluent was same. The breakthrough curve and exhaust curve was plotted accordingly to the results (Fig. 11), which shows that 100 % of Pb(II) was removed from 17 L water. The breakthrough point was observed on passing 17.8 L of 100 mg/L Pb(II). The breakthrough point biosorption capacity (BPBC) was calculated by:

$$BPBC = \frac{C_o \times V_b}{M} \quad (5)$$

where, C_o is the initial concentration of Pb(II) ion (mg/L), V_b is the breakthrough point volume of water passed (L) and M is the total mass of the biomass taken for packed column (g). The exhaust point reached after passing 50 L of 100 mg/L Pb(II) effluent from packed column. The exhausted point biosorption capacity (EPBC) was calculated by:

$$EPBC = \frac{C_o \times V_e}{M} \quad (6)$$

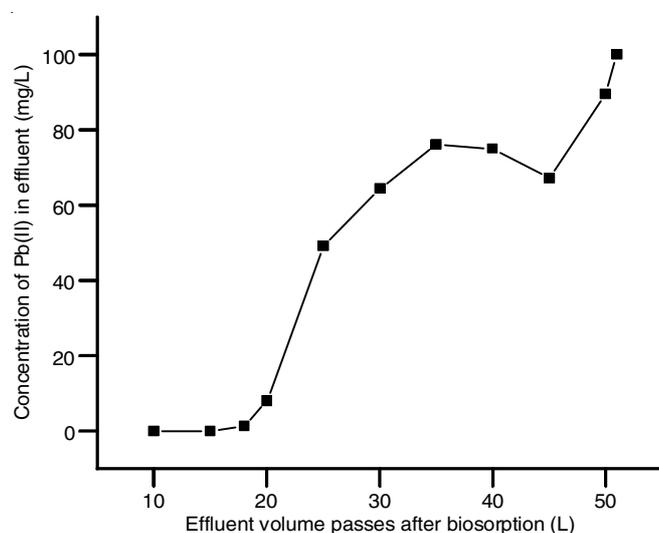


Fig. 11. Breakthrough curve and exhaust point graph of continuous flow study

where, C_o is the initial concentration of Pb(II) ion (mg/L), V_e is the exhausted point volume of water passed (L) and M is the total mass of biomass taken in packed column (g). The results obtained by above calculation for exhausted point biosorption capacity up to 17.8 L effluent was 29.66 mg/g and the exhausted point biosorption capacity up to 50 L effluent was 83.33 mg/g. Compare to batch adsorption, the exhausted point biosorption capacity for Pb(II) by *Vanda* sp. was much higher.

Application: The application of *Vanda* Sp. for the removal of Pb(II) from industrial electrolyte wastewater was taken. *Vanda* sp. shows higher sorption in low pH and it was applicable to the electrolyte wastewater as its pH level is low. The experiment were carried out where concentration of Pb(II) in wastewater before biosorption was 4.5 mg/L and pH value was 3.0. After biosorption, the result showed that Pb(II) was removed more than 99.5 % from wastewater using biosorbent. The concentration of Pb(II) in wastewater after biosorption was in permissible limit. The biosorbent was efficient for the removal of industrial electrolyte waste water.

Comparison of uptake capacity with other biosorbent: Various bio adsorbents have been reported for the removal of Pb(II) from water and wastewater. The comparison between maximum uptake capacities of recently published work to this research work is given in Table-2. As compared to the list of the given recently used bio adsorbents, *Vanda* sp. is greater efficient for the adsorption of Pb(II). Most of the biosorbent was reported the work in which the biosorption was studied by taking the pH value in neutral and mild acid range but this present study of biosorbent gives better adsorption in acidic medium that is 3.5. Usually wastewater from lead industries carried low pH value so it is one of best part of this biosorbent and can be applicable for the removal of Pb(II) containing industrial waste water.

TABLE-2
A COMPARISON OF THE UPTAKE CAPACITY FOR THE BIOSORPTION OF Pb(II) BY DIFFERENT BIOSORBENTS

Biosorbents	Uptake capacity		Ref.
	(mg/g)	pH	
Black cumin	8.08	5.1	[32]
<i>Ficus carica</i> leaves	34.36	6.0	[33]
<i>Ficus benghalensis</i> L.	28.60	6.0	[34]
<i>Araucaria cookii</i>	37.03	5.0	[35]
<i>Delftia tsuruhatensis</i>	44.40	5.0	[36]
Groundnut hull	31.54	5.0	[37]
<i>Pongamia pinnata</i>	11.16	6.0	[38]
<i>Caryocar brasiliense</i> camb.	16.78	–	[39]
<i>Vanda</i> Sp.	51.04	3.5	Present work

Conclusion

Vanda sp. showed significant removal capacity of Pb(II) with 51 mg/g and 83.33 mg/g in batch and column mode study. Different batch parameters were performed successfully to obtain optimum condition. 1 g of biomass gives maximum adsorption in pH 3.5 at contact time of 60 min. FTIR analysis confirms that hydroxyl, amine and carboxylic group are responsible for the Pb(II) sorption. SEM and EDX-ray analysis were carried out for the characterization of biomass, where

positive changes were obtained in fresh and exhausted biomass confirms the adsorption in biomass. EDX analysis showed Pb(II) characteristics signal after adsorption. The adsorption isotherm studies shows the higher correlation coefficient in Freundlich isotherm ($R^2 = 0.98$) was the prime mode of multi-layer adsorption between Pb(II) and adsorbent. This method is ecofriendly, efficient and low cost process to remove Pb(II). Removal percentage of Pb(II) was unaffected in the presence of background cations and anions. *Vanda* sp. works in low pH range and gives higher removal capacity for Pb(II) compare to other biosorbent. *Vanda* sp. has potential to remove Pb(II) ion from water and can be also use for industrial application.

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