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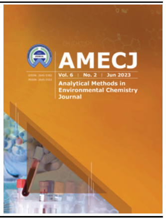

Removal of Fe (II) from aqueous solution by Calotropis Procera: Kinetics, isotherm studies, and measurement of competitive adsorption with UV-Visible spectrophotometer

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ABSTRACT

Iron is essential heavy metal in trace quantities, but its excessive concentration as Fe^{2+} is present in effluents from steel mills, iron ore mines, and metal processing industries, which pollute the groundwater. Among other conventional methods, sorption by natural biomass is a low-cost alternative for iron sequestration from an aqueous solution. The root of a native weed plant *Calotropis Procera* was used to optimize the adsorption parameters like pH, contact time, sorbent dose, and initial adsorbate concentration. Competitive adsorption of Fe^{2+} in the presence of cations (Ni^{2+}, Cd^{2+}, Cr^{3+}, Zn^{2+}, Ca^{2+}, Mg^{2+}, As^{3+}) and anions (Cl^-, SO_4^{2-} , F^-) was also studied. Batch adsorption studies were carried out to evaluate adsorption isotherm by Langmuir and Freundlich isotherm models. Leaching of biomass significantly improved iron uptake capacity from 15 mg g^{-1} to 80 mg g^{-1}. The kinetics of the reaction was fast, with equilibrium conditions attaining in 30 minutes. FTIR study of the biomass revealed the presence of -COOH, -NH groups responsible for the metal binding mechanism. The biomass could be successfully regenerated with 0.1 M HNO_3 for further use. Successful removal of iron from simulated acidic water was done under optimum conditions. In this study, absorbance was measured by a UV-Visible spectrophotometer at 523 nm.

1. Introduction

The management of groundwater resources in emerging countries is being recognized as a critical concern due to rising population, urbanization, and rapid industrialization [1]. Toxic heavy metal concentrations (Cd, As, Fe, Cr, Zn, Cu, Mn, Pb, Ni, and others) in soil, surface, and groundwater have been documented in several Asian and European countries [2,3]. The Soil, rock, and water flow regulate the metal concentration in surface water. Metals on the surface of the soil are carried away by their course, which leads to sewage and reservoirs [4]. Landfill leachates, deep good contamination disposal, industrial hazardous wastes, and other factors have contaminated groundwater [5]. The rainwater gets contaminated while passing through the atmosphere. Water resources get contaminated by the discharge of various industrial effluents into it. Identification of heavy metals is of utmost importance in the view of the management of water resources, as groundwater gets contaminated by them and thereby human beings.

Iron contamination in groundwater is an essential topic of concern as groundwater is a vital resource

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for the living and food security of the population in a country. There are two types of iron: soluble ferrous (Fe^{2+}) and insoluble ferric (Fe^{3+}). The disintegration of rocks and minerals, acid mine drainage, and landfill leachate are all possible sources of iron in natural water. To some extent, groundwater contribution might be due to its contact with the broken well casing, improper pump assemblies, leaking pipes, and storage tanks. At pH under four and relatively low oxygen in water, the oxidation process to the ferric iron is slow, so almost all of the iron is in the dissolved state [6]. Iron in drinking water is undesirable because it tastes bitter even at low concentrations (0.3 mg L^{-1}) and stains clothing and plumbing fixtures. Different methods like chlorine oxidation, filtration by activated carbon, supercritical fluid extraction method, precipitation with limestone [7], and electrocoagulation [8] have been adopted so far for the removal of Fe(II) from aqueous solution. Biosorption, a physicochemical process for the accumulation of metals by biomass, can be used as a cost-effective process for treating metal-contaminated industrial effluents. Bioremediation is an environmentally friendly approach that offers significant advantages over other decontamination methods [9]. Biosorption is a collective term including passive accumulation processes, physical or chemical adsorption [10], ion exchange, complexation [11], chelation, and precipitation for the accumulation of heavy metals. The chemical composition of metal ions, type of biomass, ambient circumstances, and competitive organic and inorganic chelators may all influence how metal ions are bound by the adsorbent [12-14]. Thus, we may take advantage of this ability of plants to uptake metals by using their dead tissues and taking advantage of their functional groups to recover metal ions from aqueous solutions. Adsorption potential of various biosorbents like a crab shell, tree bark, coir fiber, wooden charcoal [15], chitosan activated carbon composite [16], pomegranate peel carbon [17], duckweed [18], rice husk ash [19] has been identified for the removal of Fe (II) from aqueous solution. *Calotropis procera* is a common wasteland weed found mostly in East-Asian countries, abundant in the sub-tropics and tropics, and is harvested for its medicinal properties. The root of the plant was used in the adsorption study.

This study aimed to investigate the kinetics and mechanism of iron adsorption with natural biomass through the acquisition of adsorption isotherms models. The suitability of the biomass for sorption under various environmental conditions was examined through multi-metal sorption studies. Experimental investigations to improve the indigenous plant Calotropis procera's adsorption capacity were done for its technological use in the wastewater treatment process.

2. Experimental

2.1. Instrument

The UV-Visible spectrophotometer (Tungsten Halogen Lamp/Deuterium Lamp; Systronics brand 117; Microcontroller-based single beam, India) based on a wavelength range of 1100 nm, a spectral bandwidth of 1.0 nm, and solid-state silicon photodiode detector with a photometric resolution (0.001 Abs) was used for determination Fe(II) in water solution at 523 nm. A fast sequential Atomic Absorption spectrophotometer (Varian AA240FS) was used to determine heavy metals like Ni^{2+}, Cd^{2+}, Cr^{3+}, Zn^{2+}, and As^{3+}. Ca^{2+} and Mg^{2+} were determined by the titration method. The FTIR spectra of the leached and un-leached biomass were recorded in the frequency range of 400 to 4000 to analyze the functional groups responsible for adsorption using Thermo Nicolet Nexus 670 spectrophotometer.

2.2. Reagents

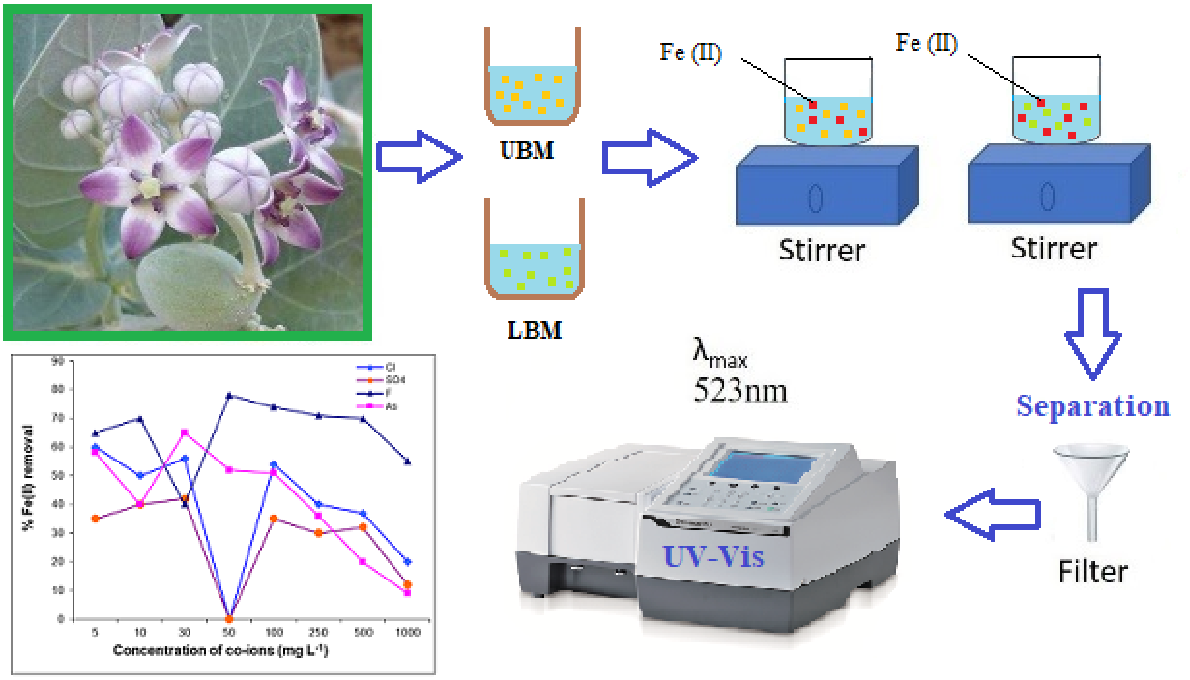
All reagents were purchased from Merck, Sigma, Germany. HNO_3 (CAS N.: 7697-37-2), H_2SO_4 (CAS N.: 7664-93-9), NaOH (CAS N.: 1310-73-2), and KOH (CAS N.: 1310-58-3) were prepared from Merck. The deionized water (DW) was prepared from Millipore (Sigma, Germany). The standard solution of Fe(II), Iron(II) ethylene-diammonium sulfate (CAS N.:113193-60-5) was purchased from Merck, Germany. Other standard metal solutions (Ni^{2+}, Cd^{2+}, Zn^{2+}, Cr^{2+}, As^{3+}, Ca^{2+}, Mg^{2+}) were prepared from Sigma, Germany (500 mL; Trace analysis for AAS).

2.3. Adsorption Procedure

Calotropis procera was hand collected, washed thoroughly with tap water, and rinsed with de-ionized water to remove foreign particles on its surface. The root was dried under sunlight and then in the oven at 60°C for 2-3 days. It was ground and screened through a 250-micron mesh after complete drying to obtain a fine biomass powder for further use in adsorption studies. The biomass was leached by soaking it in dilute HNO_3 overnight, then washed and dried in the oven again. Double distilled water was used to make stock solutions. Colorimetric determination of Fe(II) was done by the α-α bipyridyl method [20]. Absorbance was measured by a UV-Visible spectrophotometer at 523 nm. Batch adsorption studies were performed to study the effect of major influencing parameters such as adsorbent dosage, pH, initial Fe(II) concentration, contact time, and co-existing ions on the amount of adsorption capacity. In a 250 ml Erlenmeyer flask, a specific weight of leached biomass was added to 100 ml of Fe(II) solution for each experiment. The solution was stirred with a magnetic stirrer at 180 r min^{-1}. During the study, the adsorbent dosage was varied from 0.625 g L^{-1} to 5.0 g L^{-1}, the pH from 1.0 to 6.5, the initial Fe(II) concentration from 1 to 1000 mg $L^{-1,}$ and the contact time from 5 to 240 min. The pH of the solution was adjusted using either HCl (0.1 N) or NaOH (0.1 N) solutions. After stirring, the solutions were filtered through a Whatman No. 42 filter paper and used for further experimental runs. The adsorption procedure is shown in Schema 1.

2.4. Batch adsorption studies

The experimental batch method optimized the parameters for maximum Fe(II) sorption from an aqueous solution. To study the competitive effect of different ions present in groundwater on the sorption process, experimental analysis was performed on a binary solution of iron in the presence of cations (Ni^{2+}, Cd^{2+}, Zn^{2+}, Cr^{2+}, As^{3+}, Ca^{2+}, Mg^{2+}) and anions (Cl^-, SO_4^{2-}, F^-) in varying concentrations from 5 to 1000 mg L^{-1}. Experimental solutions conical flask was kept under constant stirring conditions until equilibrium time was reached. The biomass was separated from the solution by filtration, and the spectrophotometric method determined Fe^{2+} in the residual solution.



Schema1. Schematic presentation of the adsorption process

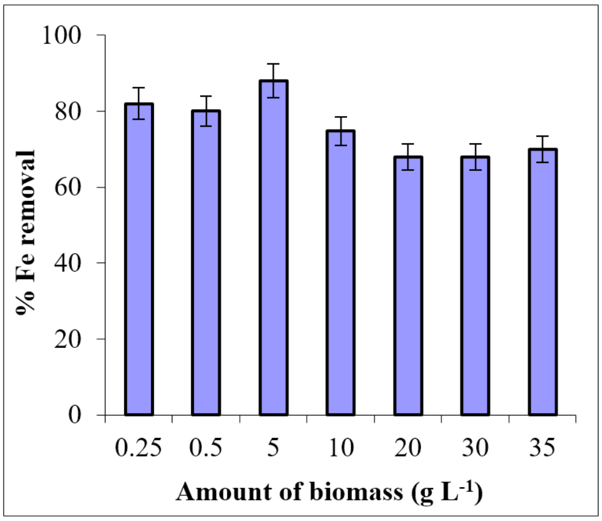


Fig. 1. Effect of adsorbent dose on Fe(II) removal

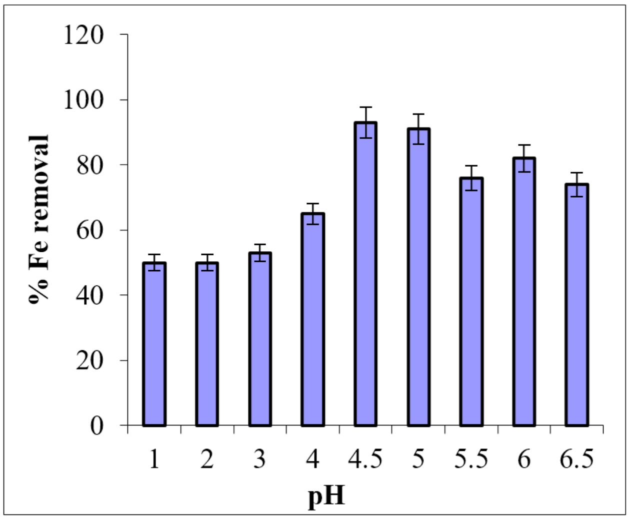


Fig. 2. Effect of pH on Fe (II) removal

3. Results and discussion

3.1. Effect of Adsorbent dose

The percentage removal of 5 mg L^{-1} iron increased from 75% to 88% when the amount of biomass varied from 0.625 g L^{-1} to 5 g L^{-1} (Fig. 1). No appreciable change in the percentage removal of Fe^{2+} was observed on further increasing biomass dose due to the saturation of binding sites in the biomass with an adsorbed metal ion, which prohibited further uptake of metal from aqueous solution [21]. Hence 5 g of the biomass was considered the optimum dose for continuing experimental studies.

3.2. Effect of pH

The most critical variable that controls metal adsorption on biosorbent is pH. It affects metal ion speciation and the ionization of surface functional groups [22,23]. The experimental result showing the adsorption of Fe at varying pH (1-6.5) is presented in Figure 2. The pH beyond seven was not investigated to avoid metal precipitation, which may interfere with and/or be indistinguishable from adsorption. The reduction in metal uptake at increased pH beyond its optimum value is attributed to reduced solubility and precipitation [24]. Experimental results showed the maximum sorption (92%) observed at pH 4.5. As a result, pH 4.5 was chosen to optimize other variables.

3.3. Kinetic study

It was observed that adsorption equilibrium reached within 30 minutes which remained constant afterward. The metal ion's maximum removal efficiency, i.e., 98%, was observed within 30 minutes of contact time (Fig.3). Further, an increase in contact time showed no change in the adsorption behavior. It means that most of the Fe(II) binding sites in *Calotropis procera* are located on the biomass's exterior surface, thereby eliminating intercellular diffusion, which is a relatively slow process [25].

3.4. Effect of initial metal ion concentration

Adsorption experiments with fresh or un-leached biomass (UBM) showed high adsorption characteristics at lower metal concentrations, i.e., 94% removal was observed for 5 mg L^{-1} Fe^{2+} which markedly reduced upon increasing iron concentration. At higher concentrations, i.e., 500 mg L^{-1} and 1000 mg L^{-1} UBM observed no adsorption. This may be due to many organic and inorganic constituents in UBM, which reduced the binding efficiency of the biomass. By contrast, the leached biomass (LBM) showed a higher percentage removal efficiency [26] over a much larger concentration range (1 to 50 mg L^{-1}), as shown in Figure 4. LBM could adsorb 98% of 50 mg L^{-1} iron within 30 minutes of contact time. At higher concentrations (1000 mg L^{-1}), the adsorption capacity of the biomass was reduced to 60%. Upon leaching by acid, all the endogenous metals and bio-molecules were removed from the biomass [27]. This enhanced the % metal uptake due to increased vacant sites on the biomass surface.

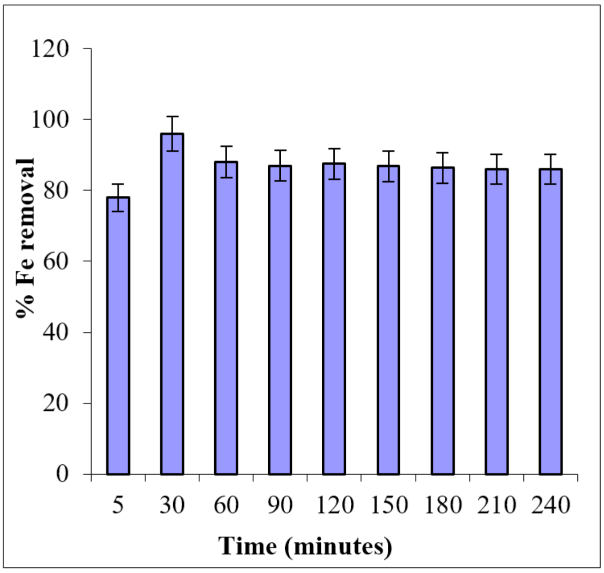


Fig. 3. Effect of contact time on Fe(II) removal

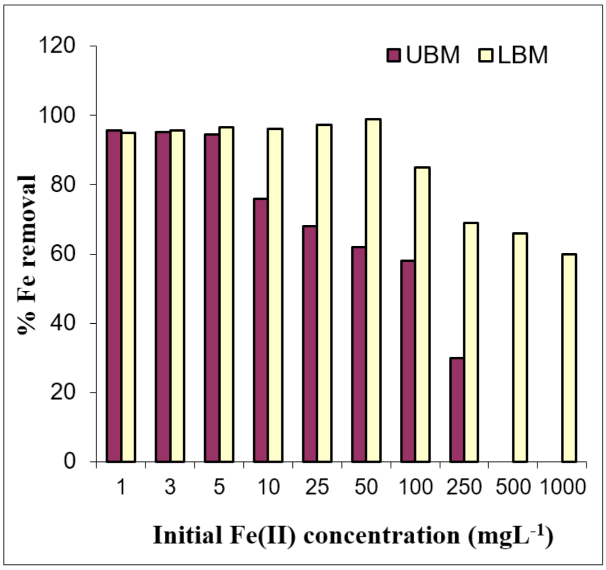


Fig. 4. Effect of metal ion concentration

3.5. Effect of co-occurring ions:

Wastewater effluents generally comprise multiple toxic and light metal ions, reducing the biomaterial's capacity to adsorb the targeted ion from the solution. The co-occurring ions present in the groundwater may also compete with the surface binding sites of the biomass and thus can influence iron adsorption [28]. Therefore, the ions considered for the study were Cl^-, SO_4^{2-}, F^-, Ca^{2+}, Mg^{2+}, As^{3+}, Ni^{2+}, Cr^{3+}, Cd^{2+}, and Zn^{2+} at various concentrations in an aqueous solution. Figure 5 displays the percentage removal of Fe(II) in the presence of anions ranging from 5 to 1000 mg L^{-1}, keeping the initial concentration of Fe^{2+} at 5 mg L^{-1} and pH at 4.5. There was a significant decrease in the metal uptake process in the presence of Cl^- and $SO_4^{2-}$with a marked inhibition at a ratio of 1:10. As^{3+} and F^- had a similar effect at lower concentrations, i.e., at 1:2 and 1:6, therefore the concentration of these two ions must be targeted for effective adsorption of iron from an aqueous solution. Zn(II) and Ni(II) showed an inhibitory effect on the Fe(II) sorption at a ratio of 1:4. At the same time, the presence of the Cd(II) did not affect the percentage of Fe(II) removal even at the higher concentrations (Fig. 6a). The decrease in metal uptake at increasing concentrations was assumed to be a reaction to increased competition for binding sites between similarly charged species [29]. Due to smaller ionic radii of Zn^{2+} and Ni^{2+} (Table 1), these ions are preferentially adsorbed onto the binding sites compared to Fe^{2+}. The Cd^{2+} has ionic radii greater than iron, so it did not significantly interfere with adsorption, as the ionic radii of Cr^{3+} are greater than iron. Hence, the presence of Cr^{3+} ions had no antagonistic effect on the adsorption efficiency of the biomass. The presence of Ca^{2+} and Mg^{2+} showed a significant reduction in metal uptake at 1:4, as shown in Figure (6b).

These alkaline metals in concentrations greater than Fe^{2+} were responsible for the dissolution of iron already present in the biomass at higher pH [30].

3.6. Biosorption isotherm

Adsorption isotherms such as the Langmuir and Freundlich isotherms are commonly used to describe adsorption data. The Langmuir model was one of the first theoretical treatments of linear sorption [31]. This has been applied successfully to a wide range of systems with limiting or maximum sorption capacities. The model assumes uniform adsorption on the surface until an equilibrium between adsorbent and adsorbate has been attained on one molecular layer. The Langmuir isotherm is given by Equation 1.

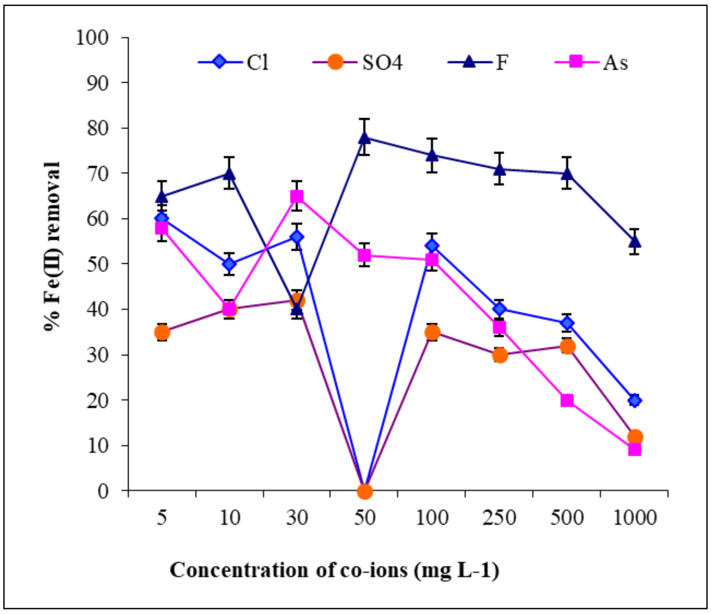


Fig. 5. Effect of competing anions on iron removal from water samples

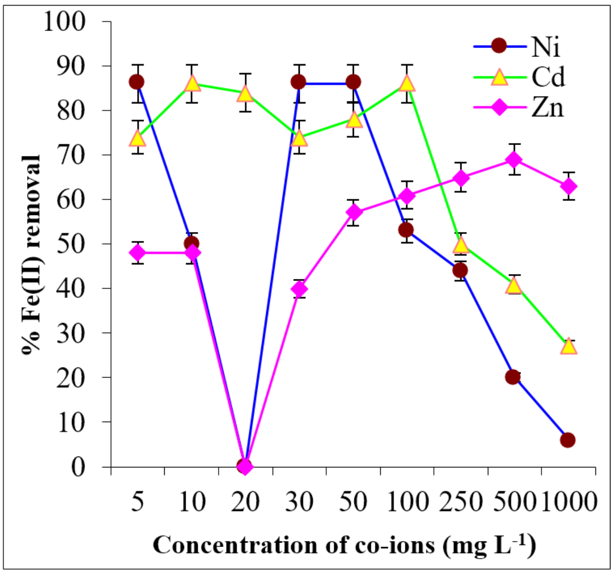


Fig. 6a. Effect of competing cations

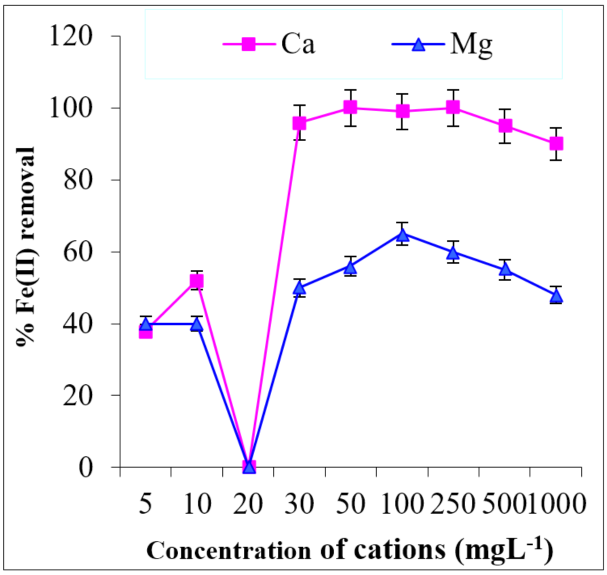


Fig. 6b. Effect of competing cations

Table 1. Comparison of ionic radii of cations

Sample	Cation	ionic charge	ionic radius(pm)
1	Fe	+2	77
2	Ni^{2+}	+2	70
3	Zn^{2+}	+2	74
4	Cd^{2+}	+2	95

$$Ce/qe = 1/[qmKe] + Ce/q_m \qquad \textbf{(Eq.1)}$$

where C_e is the concentration of adsorbate at equilibrium (mg g^{-1}), q_e is the amount of the adsorbate at equilibrium (mg g^{-1}), q_m and K_e are Langmuir constants related to maximum adsorption capacity (mg g^{-1}), and adsorption intensity (L mg^{-1}) of the biomass respectively.

The linear form of the Freundlich model is given by Equation 2.

$$\log q_e = \log K_F + 1/n \log C_e \qquad \textbf{(Eq. 2)}$$

where K_F relates to the sorption capacity and 1/ n to sorption intensity.

Equations (1) and (2) are usually used to analyze equilibrium sorption data for Langmuir and Freundlich isotherms, respectively. Each model gives the same results at low concentrations [32]. Langmuir and Freundlich plots for Fe(II) adsorption on *Calotropis procera* are shown in Figures 7 and 8, respectively. The Freundlich and the Langmuir constant values are calculated from the linear least-squares fitting. Table 2 depicts the value of these constants; the higher value of q_m for UBM shows that at a lower concentration monolayer mechanism is followed where Fe(II) gets bounded with the active sites electrostatically. With the increase in metal ion concentration, adsorption capacity increased due to the multilayered adsorption process in LBM. This is verified by a greater correlation coefficient value obtained for the Freundlich isotherm. Based on the values of correlation coefficients (r^2) for the different isotherm plots, biomass was found to fit better with the Freundlich isotherm, which explains the adsorption on the heterogeneous surface of the adsorbent. The biomass uptake of the metal ion was calculated using Equation 3.

$$q = V (C_o\text{-}C_f)/ M \qquad \textbf{(Eq. 3)}$$

Where q is the uptake, $C_{o,}$ and C_f are the initial and final concentrations of adsorbate, V is the volume of the solution, and M is the mass of the adsorbent used [33]. The uptake capacity of unleached biomass of *Calotropis procera* for iron was 15 mg g^{-1} from an aqueous solution. In contrast, a marked increase in uptake of 80 mg g^{-1} was observed by leached biomass.

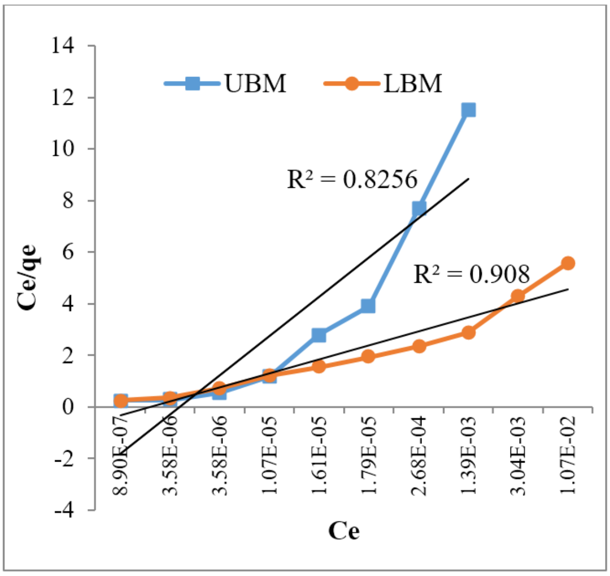


Fig. 7. Langmuir isotherm plot for Fe adsorption

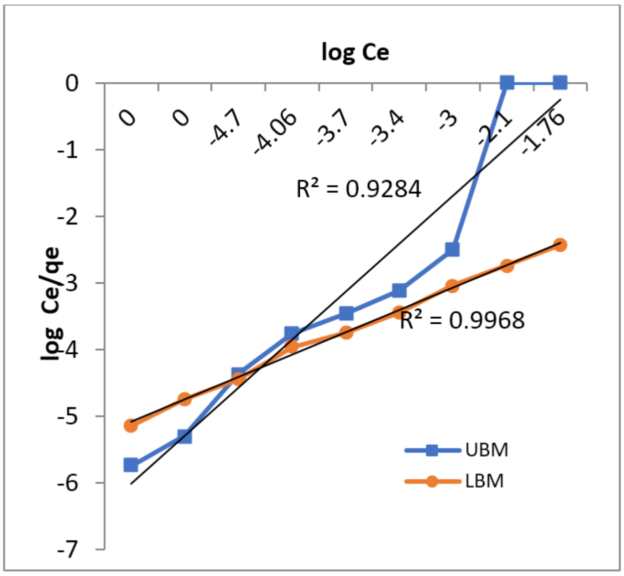


Fig. 8. Freundlich isotherm plot for Fe adsorption

Table 2. Summary of the isotherm parameters

	Langmuir isotherm			Freundlich isotherm		
Adsorbent	**K_L(L mg^{-1})**	**q_m(mg g^{-1})**	**R^2**	**K_F**	**1/n**	**R^2**
Calotropis procera (UBM)	0.458	2.18	0.825	1.40	0.719	0.928
Calotropis procera (LBM)	0.621	1.83	0.908	1.52	0.335	0.996

3.7. Regeneration of exhausted biomass

The preceding experiments on the effect of co-ions indicated that the Fe uptake by the biomass was inhibited in the presence of chloride and sulfate. These showed that acidic ligands might make regeneration/elution of Fe-loaded biomass feasible. Therefore, an exploratory regeneration experiment for Fe-loaded biomass was conducted under batch conditions. The Fe(II) loaded biomass was connected with different eluents, i.e., 0.1M HCl, 0.1M H_2SO_4, 0.1M HNO_3, 0.2M NaCl, 0.05M Na_2CO_3, and 0.05M NaOH for 60 minutes. Metal ion recovery was calculated by Equation 4 [34].

$$M(rec) = \frac{\text{Amount of metal ion desorbed}}{\text{Amount of metal ion adsorbed}} \text{x } 100$$

(Eq. 4)

The results showed that regeneration of exhausted biomass, i.e., the recovery of metal ions, was very effective in the presence of 0.1M HNO_3. All three acidic reagents, HNO_3, HCl, and H_2SO_4, were effective in regenerating more than 95% of the Fe adsorbed on the biomass (Fig. 9). Among them, HNO_3 exhibited maximum elution efficiency, i.e., 99% removal of total iron adsorbed on the surface of biomass.

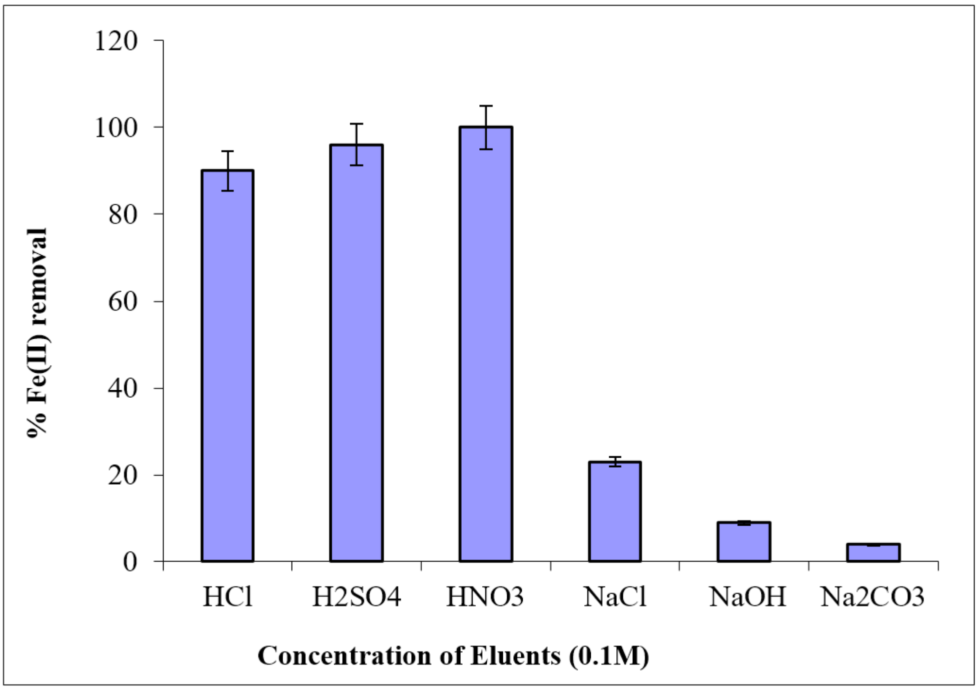


Fig. 9. Effect of eluents on Fe(II) recovery

3.8. Metal-binding mechanism

Infrared spectroscopy is used to detect the functional group responsible for metal binding in biomass. This technique helps to give information about the type of bond formation between the metal ion and functional groups of the adsorbent. Figures 10 and 11 represent the FTIR spectra of UBM and LBM. The strong absorption peak at 3416 cm^{-1}in UBM is attributed to the presence of the alcoholic -OH & -NH group of a primary aliphatic amine, which shifted to 3489 cm^{-1}in LBM. It signifies the significant involvement of these functional groups in the metal-binding behavior [35]. The intensity of absorbance peaks at 1250 cm^{-1} and 1158 cm^{-1} represents a -CO functional group of carboxylic acid [36]. A major shift in the peak from 1373 cm^{-1} to 1639 cm^{-1} shows the involvement of NO_2 groups of the LBM. A change in the frequency from 859 cm^{-1} to 1639 cm^{-1} shows an appreciable change in the chemical structure of the biomass upon leaching [37]. This frequency is related to phenyl ring substitution in the biomass. Table 3 represents the absorption peak frequencies of corresponding functional groups present in the biomass. It also indicates that these functional groups are the basic constituent of *Calotropis procera* which are responsible for binding Fe^{2+} ions from an aqueous solution. Also, many methodologies based on nanotechnology were used for metal removal from various matrixes [38-40].

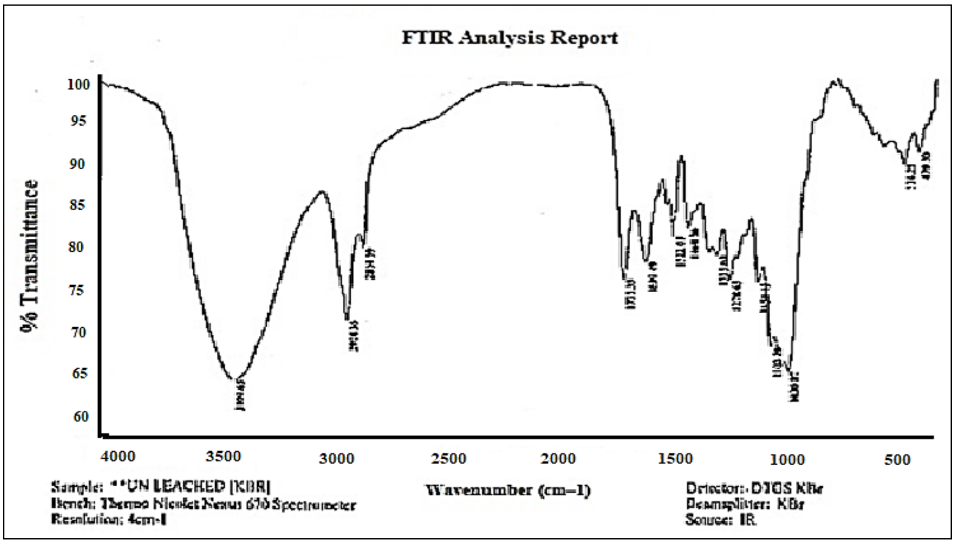


Fig. 10. FT-IR spectra of Unleached biomass (UBM)

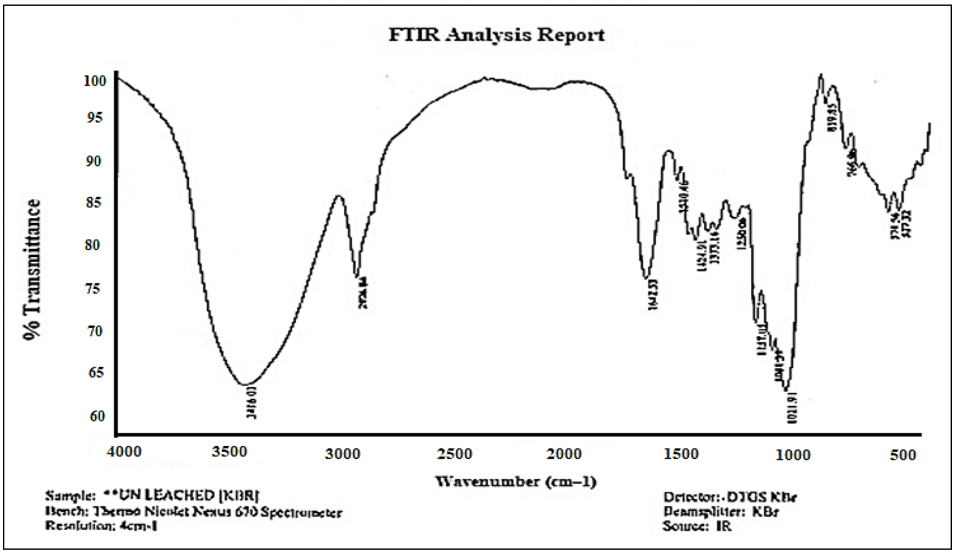


Fig. 11. FT-IR spectra of leached biomass (LBM)

Table 3. IR absorption band and corresponding functional groups

Sample	Absorption peak in UBM(cm^{-1})	Absorption peak in LBM (cm^{-1})	Change Observed	Region of spectrum(cm^{-1})	Functional groups involved
1	3416	3409	Shift	3600-3200	O-H stretching in alcohol, NH stretching in aliphatic amine
2	2926	2924	Shift	2956-1350	O-H stretching, C-H stretching vibration in Alkanes
3	None	2854	Additional peak	2956-1350	Alkanes –C-H stretching
4	1373	1639	Shift	1660-1000	NO_2stretchings
5	1250	1158	Shift	1260-1000	C-O stretchings, Carboxalic acid
6	859	1639	Shift	1750-750	Phenyl ring substitution band

3.9. Application of biomass for removal of dissolved iron from acidic effluents

The feasibility of the *Calotropis procera* as a possible sorbent for removing iron from various industrial and mining effluents was demonstrated using a simulated multi-metal ion solution for the adsorption of Fe^{2+}. Under similar experimental conditions, the biomass showed 62 % removal of 50 mg L^{-1} Fe^{2+} at pH 4.5 in the presence of Cl^-, SO_4^{2-}, F^-, Fe^{3+}, Ni^{2+}, Cr^{6+}, Cd^{2+}, and Zn^{2+} (Table 4). It is evident from the result that in the presence of multi-metal ions present in several industrial and mining effluents, the percentage of Fe(II) adsorption reduces as compared to the presence of parent metal ions alone.

4. Conclusion

Calotropis procera was found to be a good Fe(II) adsorbent, indicating its potential to sequester heavy metals from wastewater present in low concentrations. Infrared analysis of the biomass indicated the presence of functional groups like -OH, $-NH_2$, and $-NO_2$ responsible for the binding mechanism. A significant amount of Fe^{2+} (1000 mg L^{-1}) could be treated successfully at the acidic pH of 4.5 with the leached biomass dosage of 5.0 g L^{-1}. The equimolar concentration of Cl^-, SO_4^{2-} and lower concentrations of Ni^{2+}, Zn^{2+}present along with Fe^{2+}reduced the adsorption efficiency of the biomass. A higher R^2 value of Freundlich isotherm indicated a multilayered sorption process in the biomass. The biomass can be used for multiple adsorption cycles before disposing of in soil due to its regenerative nature. Thus *Calotropis procera,* a cost-effective, efficient, regenerative biosorbent, can be successfully applied to remove Fe(II) from industrial and mining effluents.

Table 4. Removal of $Fe^{2+}$from simulated industrial and mining effluents

	Fe(II) (50 mg L^{-1})	**Simulated effluent**
Biomass	*Calotropis procera* (LBM)	*Calotropis procera* (LBM)
pH	4.5	4.5
%Fe removal	98	62

5. Acknowledgment

I wish to express my deepest gratitude to Bhilai Institute of Technology, Durg, C.G.for providing a good research lab and rich library to complete my research work.

6. Conflict of Interest

The authors declare that they have no conflict of interest.

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