Asian Journal of Physical and Chemical Sciences

2(3): 1-11, 2017; Article no.AJOPACS.34356
ISSN: 2456-7779

Adsorption of Cadmium (II) Ions from Aqueous Solution onto Mango Leaves

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Authors' contributions

This work was carried out in collaboration between all authors. Author AEAP designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Author MAEA managed the analyses of the study and wrote the protocol. Author AA gave her experience during the analysis of the data and critical revision. Authors MEAEM and HTAE assistant in the Lab work. Author KMEM managed the literature searches and revision. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/AJOPACS/2017/34356
Editor(s):
(1) Parveez Gull, Department of Chemistry, School of Chemical Sciences & Pharmacy, Central University of Rajasthan, India.
Reviewers:
(1) Itodo Udoji Adams, Federal University of Agriculture, Makurdi, Nigeria.
(2) Farid I. El-Dossoki, Port Said University, Egypt.
Complete Peer review History: http://www.sciencedomain.org/review-history/19981

Original Research Article

Received 25th May 2017
Accepted 6th June 2017
Published 10th July 2017

ABSTRACT

The objective of this study was to evaluate the use of mango leaves for their ability to remove Cd^{2+} ions from aqueous solution by batch experiments. The adsorption of Cd^{2+} ions onto mango leaves were characterized by using various analytical techniques: Fourier transform infrared spectroscopy and Scanning electron microscopy. The effects of various parameters, such as solution pH, adsorbent dosage, initial concentration of cadmium ions, temperature and presence of chelating agent EDTA suppressed the uptake of Cd^{2+} ions were examined. The mango leaves investigated in this study showed good potential for the removal of cadmium from real industrial wastewater. The desorption test revealed that HCl was the best for the elution of metals from the tested mango leaves. The equilibrium data were well fitted with the Langmuir isotherms. In conclusion, the mango leaves was the favourable alternative of cadmium removal from water.

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Keywords: Mango leaves; cadmium; wastewater; desorption.

1. INTRODUCTION

Wastewater treatment is becoming more critical because diminishing water resources, increasing wastewater disposal costs and stricter discharge regulations that have lowered permissible contaminant levels in waste streams. The heavy metals most commonly found in contaminated waters as Pb^{2+}, Cr^{2+}, Zn^{2+}, Cd^{2+}, Cu^{2+} and Hg^{2+}. So, their removal from contaminated waters has become a major topic of research in recent years, due to the toxicological problems caused by the metal ions to the environment as well as to human health [1,2]. Several methods have been applied during recent years for the elimination of these metal ions present in industrial wastewaters. The commonly traditional methods used for removal of heavy metal ions from aqueous solution include precipitation, filtration, ion exchange, evaporation, reverse osmosis, solvent extraction, electrochemical treatment and membrane technologies. However, these traditional methods are either inefficient and expensive when heavy metals exist in lower concentrations [3,4]. Consequently, it is essential to find new methods for effective removal of heavy metals from water and wastewater. Compared with other traditional methods, adsorption is quite popular due to its simplicity and high efficiency, and the availability of a wide range of adsorbents. The utilization of mango leaves as an adsorbent to remove heavy metals from industrial effluent can greatly reduce the amount of waste produced. Leaves of different trees are very versatile natured chemical species as these contain a variety of organic and inorganic compounds. Cellulose, hemicellulose, pectins and lignin present in the cell wall are the most important sorption sites [5]. The important feature of these compounds is that they contain hydroxyl, carboxylic, carbonyl, amino and nitro groups which are important sites for metal sorption [6]. Chelating agents are widely used in industrial applications hence, wastewater generated from these industries contain significant amount of chelating agents. Chelating agents most widely used in industrial application as ethylene diamine tetra acetic acid. Therefore, the main objective of this study was to evaluate the adsorptive performance of local mango leaves for the removal of cadmium ions from aqueous solutions, as well as study the effect of pH, biomass dosage, initial metal concentration and temperature on the treatment process. In addition to study the efficiency of different elutants to desorp the cadmium from the mango leaves.

2. MATERIALS AND METHODS

2.1 Materials

2.1.1 Preparation of adsorbate

The analytical grade salt $Cd(NO_3)_2$ was used to prepare stock solution (1000 mg/L) of Cd^{2+}. The desired concentrations were prepared by dilution of stock solution with deionised water. The initial pH was adjusted with dilute acid or base. The initial and equilibrium phase metal concentrations (C_o and C_e) was measured using a flame atomic absorption spectroscopy (Perkin Elmer AAnalyst 100).

2.1.2 Preparation of adsorbent

Mango leaves was collected from agricultural areas, and the adsorbent material was washed, cut into small pieces then oven dried at 80°C and finally was grinded to suitable size. The untreated material were used as adsorbent in the bench-scale studies [5].

2.2 Methods

2.2.1 Effect of pH

During the batch experiment of pH effect, the parameters of temperature, solution volume, adsorbent amount, initial metal ion concentration, and shaking time were fixed at 30°C, 10 mL, 10 mg/L,0.2 g and 120 min, respectively. Effects of pH were tested at pH 3, 4, 5, 6, 7 and 8, at agitation rate of 130 rpm [7].

2.2.2 Effect of adsorbent amount

This part of the batch experiment was performed to verify the effect of adsorbent weight on sorption process. Different weights of adsorbents (0.1, 0.2, 0.3, 0.4 and 0.5 g) were mixed and shaked with 10 mL solution of 10 mg Cd^{2+}/L at 30°C, pH 6 for 120 min and agitation speed was maintained at 130 rpm [8].

2.2.3 Initial cadmium concentration

In order to assess different Cd^{2+} concentrations of 10, 20, 30, 40, 50, 60, 70 and 80 mg/L were examined at constant parameters, pH 6 with 0.2 g of adsorbent added into 10 ml solutions at

30°C with agitation speed was maintained at 130 rpm [9].

2.2.4 Effect of temperature

Adsorption process was carried out at different values of temperature (25, 30, 35, 40, 45 and 50°C) by batch experiments, at constant pH 6, 0.2 g adsorbent weight, volume of 10 ml of 10 mg Cd^{2+} /L, and agitation rate was maintained at 130 rpm for 120 min [10].

2.2.5 Effect of chelating agent

The chelating agents used in this study is sodium salt of ethylenediamine tetraacetic acid (EDTA) as showed in Fig. 1. 10 mg/L of EDTA was added to a mixture of 0.2 g of mango leaves powder and 10 mL of Cd^{2+} solution and the mixture was agitated at 130 rpm over time periods of 5, 10, 15, 30, 60 and 120 minutes. The above step was repeated by using 20 mg/L of EDTA [5].

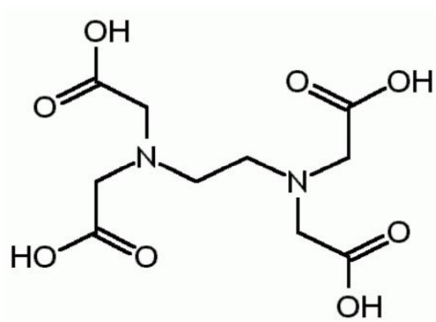

Fig. 1. Structure of EDTA

2.3 Application to Waste and Seawater

The industrial wastewater and seawater samples were collected from chemical and plastics company for paints and Mediterranean Sea at Baltium, Egypt, respectively. Owing to the low concentrations of cadmium, quantity of metal ions was added as metal solution to obtain 10 mg/L of cadmium. The metal solution was adjusted to pH 6 and filtered. The adsorption process were carried out under optimization conditions [11].

2.4 Metal Removal Efficiency

Adsorption capacity (q_e), the amount of metal adsorbed per gram of biosorbent [12], was calculated at equilibrium in (mg/g) as follows:

$$q_e = (C_0 - C_e)\, V/m \quad (1)$$

Where C_0 is the initial concentration of metal ions in the solution (mg/L), C_e is the equilibrium concentration of metal ions in the solution (mg/L), V is the volume of solution (in liters) and m is the mass of adsorbent applied (in grams). Metal uptake was also be displayed as the percentage of metal removal given by the equation:

$$\text{Metal removal (\%)} = 100(C_0 - C_e)/C_0 \quad (2)$$

2.5 Characterization of Adsorbent

2.5.1 Scanning Electron Microscopy (SEM)

Scanning electron microscope (Model S 3400N, Hitachi, Japan) analysis was also carried out for the dry mango leaves particles and different sections in the samples were examined (before and after metals adsorption) for investigation of the surface morphology.

2.5.2 Fourier-transform infrared analysis (FTIR)

Dry mango leaves samples (before and after cadmium adsorption) were examined with a Model Tensor – 27 Bruker FTIR within the wave number 400–5000 cm^{-1} under ambient conditions. This technique was used to elucidate the chemical characteristics relevant to metallic ion sorption by the mango leaves.

2.6 Desorption Experiment

Desorption studies were performed as follows: 0.1 g of adsorbent was shaken with 10 mL of 10 mg/L cadmium ions solution for 120 minutes. After shaking and filtration steps, adsorbed metals were desorbed in separate experiments with 1M of (HCl, H_2SO_4, HNO_3, NaOH and distilled water). The eluted metal was determined and the elution efficiency was defined as follows:

$$\text{Elution efficiency (\%)} = 100(C_s V_s) / (q_e\, m) \quad (3)$$

Where C_s is the concentration of metal ions in the desorbed solution (mg /L), V_s is the volume of solution in the desorption (L), m is the mass of biosorbent used in desorption studies (g) and q_e is defined in equation (1) [11].

2.7 Adsorption Isotherm Models

The sorption isotherms are the mathematical model which provides an explanation about the behaviour of adsorbate species between solid and liquid phases. Langmuir and Freundlich isotherm models [13,14] were studied for the investigation of adsorption of Cd^{2+} ions by mango leaves. The Langmuir isotherm assumes monolayer coverage of metal ions over a homogeneous sorbent surface [15].

The isotherm is presented by the following equation:

$$q_e = q_{max}\, bC_e / (1 + bC_e) \quad (4)$$

Where: q_e (mg/g) is the observed biosorption capacity at equilibrium, q_{max} (mg/g) is the maximum biosorption capacity corresponding to the saturation capacity (representing total binding sites of biomass), C_e (mg/L) is the equilibrium concentration and b (L/mg) is a coefficient related to the affinity between the sorbent and sorbate (b is the energy of adsorption).

The linear relationship can be obtained by plotting ($1/q_e$) vs. ($1/C_e$):

$$1/q_e = 1/(bq_{max}C_e) + 1/q_{max} \quad (5)$$

In which b and q_{max} are determined from slope and intercept, respectively.

Freundlich isotherm is used for modeling the adsorption on heterogeneous surfaces. This isotherm can be described as follows:

$$q_e = K_f\, C_e^{1/n} \quad (6)$$

The Freundlich model can be easily linearised by plotting it in a logarithmic form:

$$\mathrm{Log}q_e = \log K_f + 1/n \log C_e \quad (7)$$

By plotting log q_e vs. log C_e, the constant n and K_f can be determined from the slope and intercept, respectively. Where K_f and n are the Freundlich constants related to the adsorption capacity and intensity of the sorbent, respectively [14].

3. RESULTS AND DISCUSSION

3.1 Parameters Affecting Adsorption of Cd^{2+}

3.1.1 Effect of pH

pH of aqueous solution is an important parameter governing the adsorption process which affects the surface charge of adsorbents. The effect of solution pH on the adsorption of Cd^{2+} ions onto mango leaves was evaluated in the pH range of 2 to 8 and the result was showed in Fig. 2. Sorption experiment at pH 6 was able to give cadmium removal efficiency with 93.49%. Protonated adsorption sites were incapable of binding Cd^{2+}ions due to electrostatic repulsion between positively charged Cd^{2+} ions and positive charged sites. Hence, only low percentage of Cd^{2+} ions were adsorbed. As the pH increased, there were fewer H^+ ions present in the solution and consequently more negatively charged sites were made available and this facilitated greater Cd^{2+} ions uptake by electrostatic attraction. At pH values higher than 8 for cadmium, several hydroxyl low-soluble species were probably formed i.e. $Cd(OH)_2$ or $Cd(OH)_3$ [16].

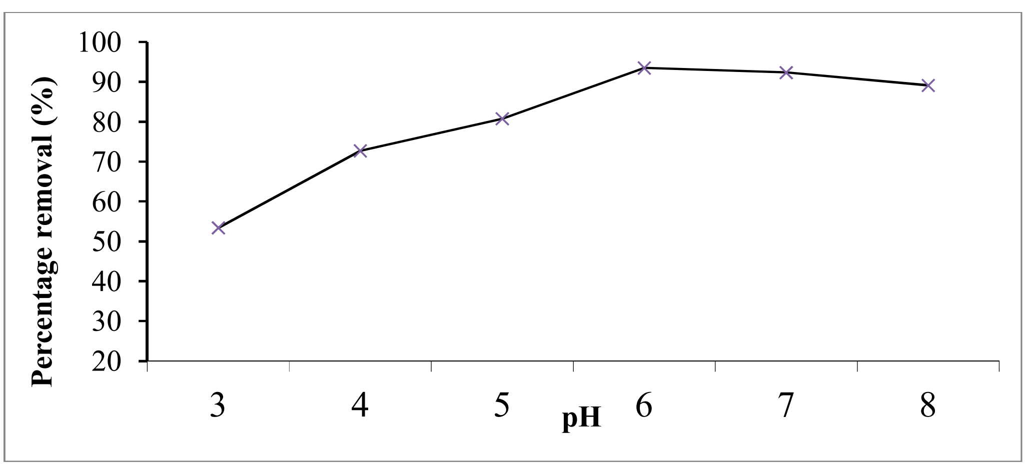

Fig. 2. Effect of pH on the adsorption of Cd^{2+} ions

3.1.2 Effect of adsorbent dosage

The effect of the adsorbent dose was studied by varying the sorbent amounts and the results were illustrated in Fig. 3. The percentage removal increase in uptake was observed when the dose was increased from 0.1 to 0.5 g. The maximum removal of metal ions was obtained at the adsorbent dose of 0.5 g due to the availability of more and more adsorbent surfaces for the solutes to adsorb. At a large amount of metal ion is biosorbed if the distance between the cells is greater, other words lower amount of biosorbent is used which is enough for the sorption takes place [17].

3.1.3 Effect of initial heavy metal concentration

The extent of removal of heavy metals from aqueous solution depends strongly on the initial metal ion concentration. By using mango leaves, the maximum uptake was found at the concentration of 10 mg/L for Cd^{2+} ions as illustrated in Fig. 4, after that, the metal removal decreased with the increase in initial concentration of metal ions until the end of experiments.

The fast initial uptake occurred in the early stage of adsorption was due to the fact that most of the binding sites on mango leaves were free which allowed quick binding of Cd^{2+}ions on the biomass. As the binding sites became exhausted, the uptake rate slowed down due to competition for decreasing availability of actives sites by metal ions. Kumar and Gayathri [18] reported that, the uptake rate is controlled by the rate at which the adsorbate is transported from the exterior to the interior sites of the adsorbent particles.

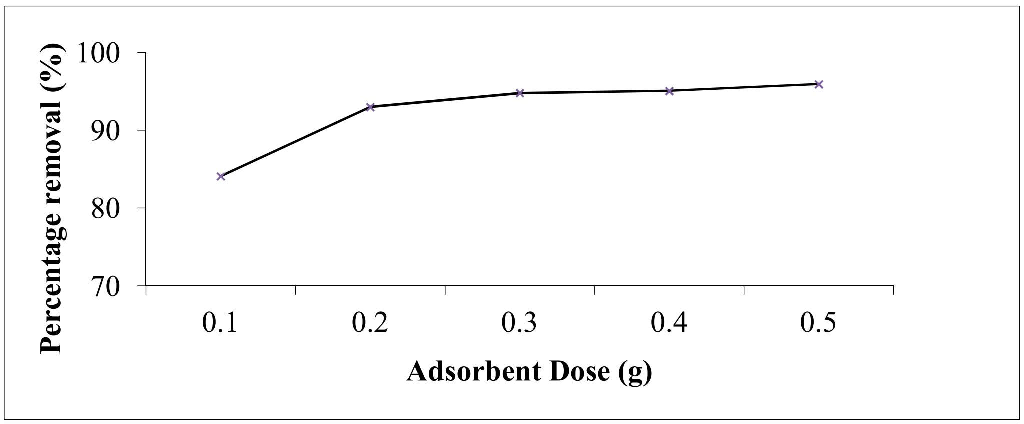

Fig. 3. Effect of adsorbent dose on adsorption of Cd^{2+} ions

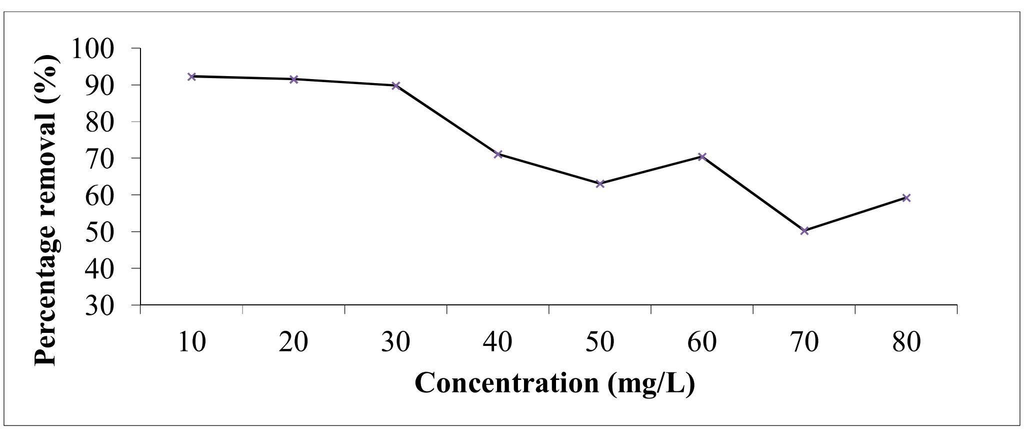

Fig. 4. Effect of initial metal ion concentration on adsorption of Cd^{2+} ions

3.1.4 Effect of temperature

Experiments were performed at temperatures of 20 to 50°C. It can be concluded that a maximum percentage removal of metals has been obtained at 30°C and reduced with the rise in temperature from 30 to 50°C for Cd^{2+} ions as shown in Fig. 5. With an increase in temperature above 20°C to 30 or 35°C, increasing temperature is known to increase the diffusion rate of adsorbate molecules within pores as a result of decreasing solution viscosity and will also modify the equilibrium capacity of the adsorbent for a particular adsorbate. Further increase in temperature above 30 or 35°C leads to a decrease in the percentage removal. This is mainly due to the decreased surface activity, suggesting that adsorption between metals and adsorbents [19].

3.1.5 Effect of chelating agent

EDTA was selected in this study to test how the presence of chelating agent affect Cd^{2+} ions uptake by mango leaves. Fig. 6 shows the uptake of Cd^{2+} ions in the presence of different concentration. It was evident that the percentage uptake was significantly reduced in the presence of EDTA. The uptake of Cd^{2+} ions reduced from 72.46% to 68.29% when 10 mg/L of EDTA was added and further decreased to 56.71% when the concentration of EDTA added was increased to 20 mg/L after 120 min, when compared with the same time without EDTA as shown in Fig. 6.

The reduction in uptake of Cd^{2+} ions in the presence of chelators occurred because Cd^{2+} ions combined strongly with EDTA rather than mango leaves, forming a stable complex and hence, inhibited the uptake of Cd^{2+} ions by mango leaves. The sorbent adsorbed Cd^{2+} via free cations form and was not able to bind EDTA-Cd^{2+} complexes, thus leaving behind unadsorbed Cd^{2+} in the solution.

3.2 Application to Waste and Seawater

Due to the complexity of industrial wastewater environment, the evolution of adsorption capabilities of biomass for heavy metals is fundamental to develop bioprocess to remove and recover metals from industrial wastewater. So, the adsorption behavior of the mango leaves in treating industrial wastewater effluent and seawater samples were studied and the obtained results are illustrated in Fig. 7.

Industrial wastewater showed pronounced metal effect on the adsorption process with 53.8%, while in seawater the removal percentage was high with 55.04%, this may be due to the existence of organic materials in wastewater, which could also be adsorbed onto adsorbents and complexication of organic materials and metal ion could also reduce the adsorption of metals on adsorbents. In the present investigation, it is appeared that seawater has a high concentration of cation metals (Na^{+}, Mg^{2+} and Ca^{2+}) than that of wastewater and deionized water because of decreasing in metal ions percentage removal. Moawad [20] reported that cadmium tends to be more affected by interfering ions present in seawater than wastewater and the increases in magnitude of cation metal ions in seawater that retard cadmium adsorption.

3.3 Characterization of Adsorbents

3.3.1 Scanning electron microscope (SEM)

Scanning electron microscopy was used to investigate samples of mango leaves before and after metal binding. Fig. 8 shows mango leaves before and after exposure to metal ions solution. Its particles were organized, fine and well-shaped before its exposure to metal ions solution but after its exposure to these ions, they distorted and exhibited uneven surface texture along with lot of irregular surface the heavy metal ions occupied the available free binding sites.

3.3.2 Fourier transform infrared spectroscopy

Fig. 9 shows the FTIR spectrum of mango leaves under investigation before and after adsorption of metal ions. The analysis of the FTIR spectra indicates broad band at 3418.21 and 3423.06 cm^{-1} (ranging from 3200 to 3600 cm^{-1}) before and after adsorption respectively, representing OH^{-} group due to inter and intra molecular hydrogen bonding of polymeric compounds such as alcohols or phenols as in pectin, hemicelluloses, cellulose and lignin [21] and -NH groups [15]. The region between 3000-2800 cm^{-1} showed the C-H stretching vibrations of sp3 hybridized C in CH_3 and CH_2 functional groups [17]. The peak registered at 1730.30 cm^{-1} before and after adsorption this band was shifted to 1729.08 cm^{-1} represents the stretching vibration of C=O bonds, which originates from non-ionic carboxyl groups (–COOH, –$COOCH_3$) and may be denoted by carboxylic acids or corresponding esters [22]. The peaks around from 1624.71 to 1624.05 cm^{-1} before and after

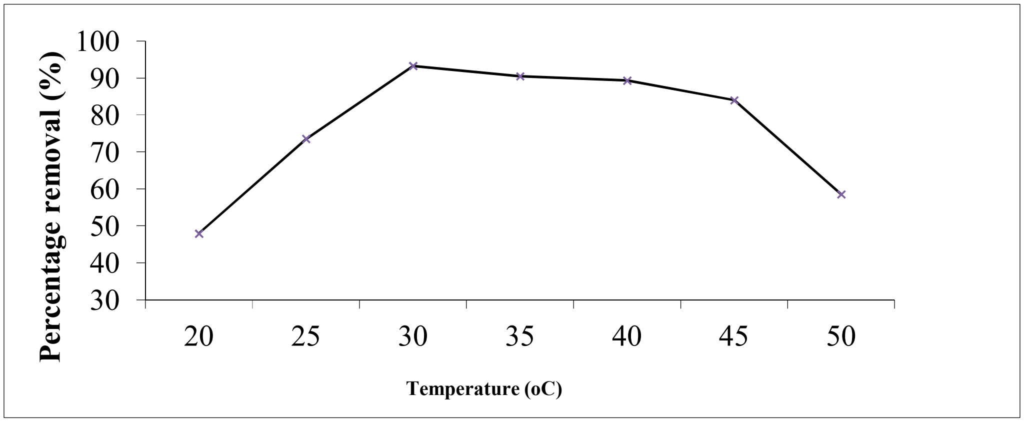

Fig. 5. Effect of temperature on adsorption percentage of Cd^{2+} ions

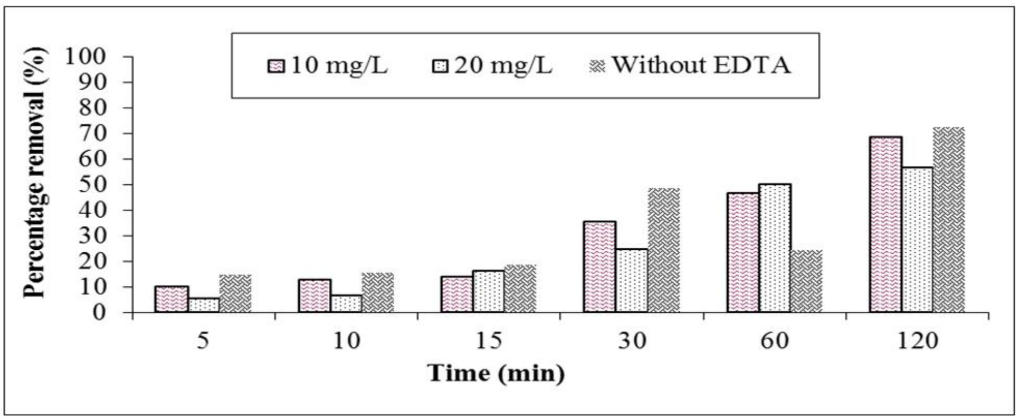

Fig. 6. Effect of chelating agent (EDTA) on Cd^{2+} ions removal

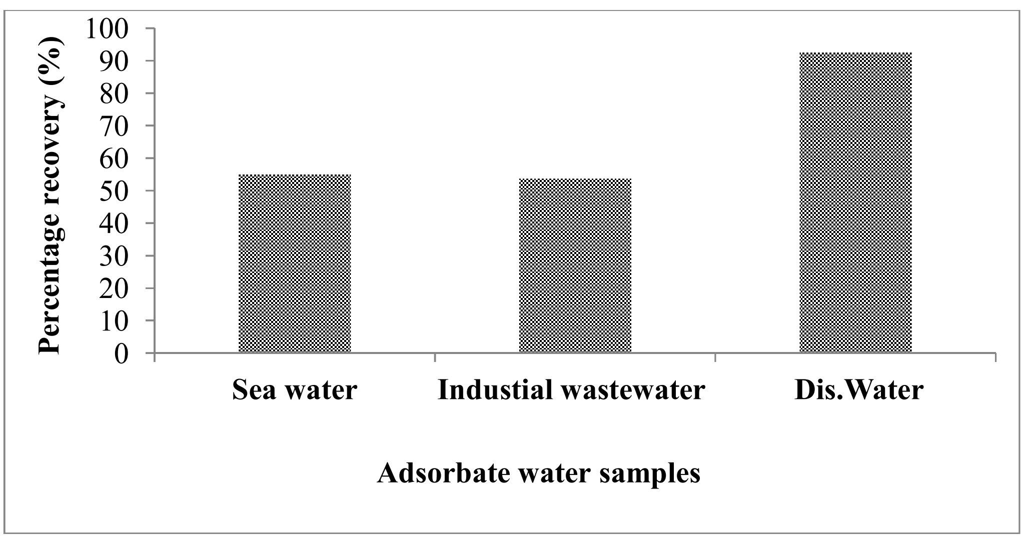

Fig. 7. Metals removal percentage of Cd^{2+} ions in different water sample

adsorption represents C=O stretching [23]. The additional peaks at 603–893 cm^{-1} can be assigned to bending modes of aromatic compounds. From FTIR study, the formation of new absorption bands, the change in absorption intensity and the shift in wavenumber of functional groups in the raw sample and small differences in the frequency bands could be due to interaction of metal ions with active sites of adsorbents.

3.4 Desorption of Cadmium Ions

Desorption of metals or regeneration of adsorbents from used-adsorbents are crucial to repeat the use of adsorbent, recover of precious metals and reduce the cost of operation in any water treatment system. Desorption of Cd^{2+} ions by mango leaves biomass was studied by using 1 M of NaOH, HCl, H_2SO_4 and HNO_3 as well as distilled water. The results of desorption are shown in Fig. 10, which clearly indicates effective desorption with mineral acids (HCl and H_2SO_4). The highest amount of Cd^{2+} ions recovery was 66% using of 1 M HCl. The rapid desorption elevated the applicability of the adsorption process. At acidic conditions, H^+ ions protonate the adsorbent surface by replacing the adsorbed metal ions on the adsorbent surface leading to desorption of the positively charged metal ion [24].

3.5 Adsorption Isotherm Models

The plots of non-linearized Langmuir and Freundlich adsorption isotherms were shown in

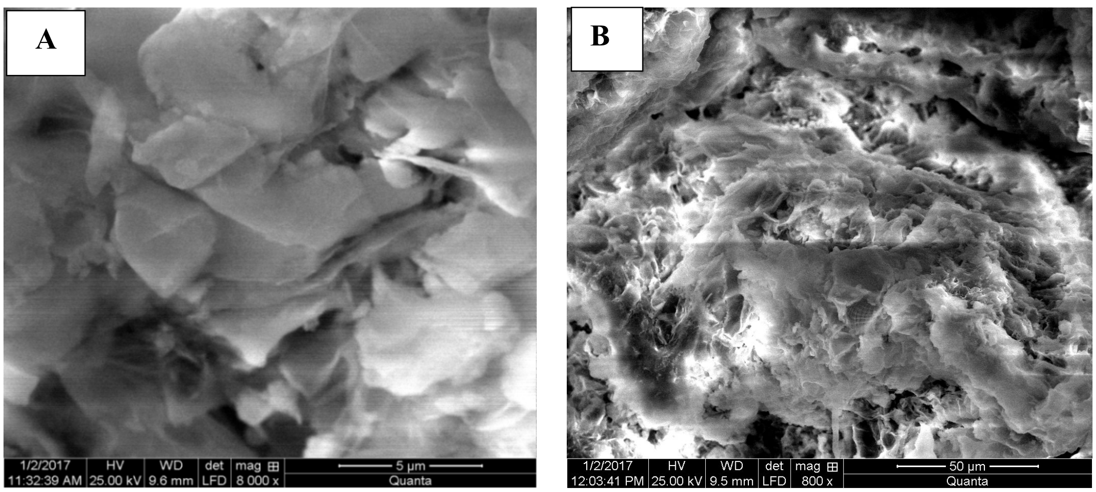

Fig. 8. SEM micrograph mango leaves cell wall before (A) and after (B) metal exposure

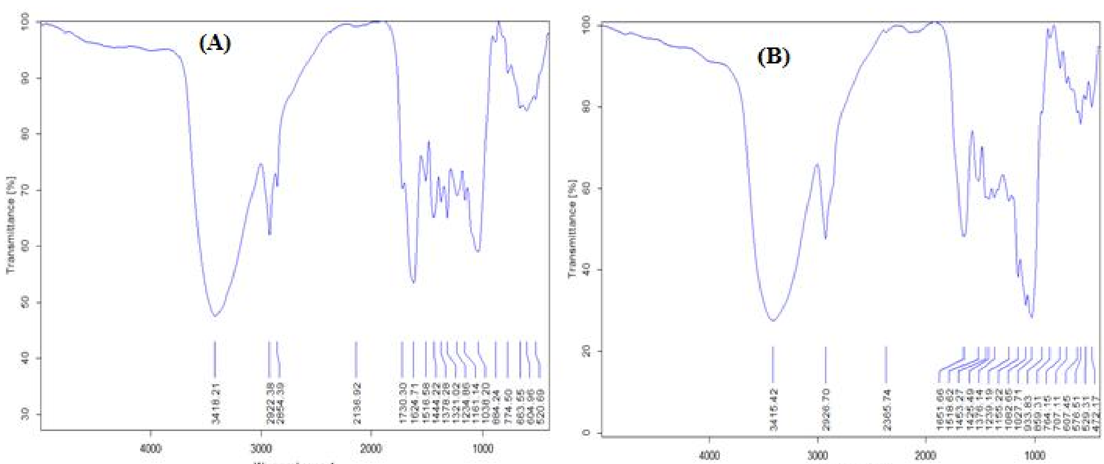

Fig. 9. FTIR spectrum of before (A) and after (B) adsorption

Figs. (11 and 12). The adsorption isotherm constants were determined by using non-linear regression. The Langmuir adsorption constants evaluated from the isotherms with the correlation coefficients were also presented in Table (1). The best-fit equilibrium model was determined based on the linear regression correlation coefficient R^2. The fitting of experimental data with Langmuir model was emphasized by high R^2 values. The linear correlation regression coefficient (0.96) shows that the adsorption data is best fitted with the Langmuir equation. The Langmuir fit is consistent with strong monolayer sorption on to specific sites.

Table 1. Langmuir and Frendlich isotherm parameters for cadmium

Model	Parameter			
Langmuir	q_{max}	b	R^2	R_L
	4.08	0.97	0.961	0.25
Freundlich	n	k_f	R^2	
	2.538	1.577	0.889	

Accordingly, the essential characteristics of the Langmuir isotherm parameter can be expressed in terms of separation factor or dimensionless equilibrium parameter, R_L according to the equation as follows:

$$R_L = 1 / (1 + bC_o) \quad (8)$$

Where b is the Langmuir constant and C_o is the initial concentration of Cd^{+2} ions. The value of separation parameter R_L provides important information about the nature of adsorption. The value of R_L indicated the type of Langmuir isotherm to be irreversible (R_L = 0), favorable (0 < R_L < 1), linear (R_L = 1) or unfavorable (R_L > 1). R_L values between 0 and 1 indicate favourable absorption [25]. From this study, The R_L was found to be 0.25 (Table 1).

The experimental data obeyed also the Freundlich model, as confirmed by the high determination coefficient (R^2 = 0.889) (Table 1). As can be seen from the results, the *n* values

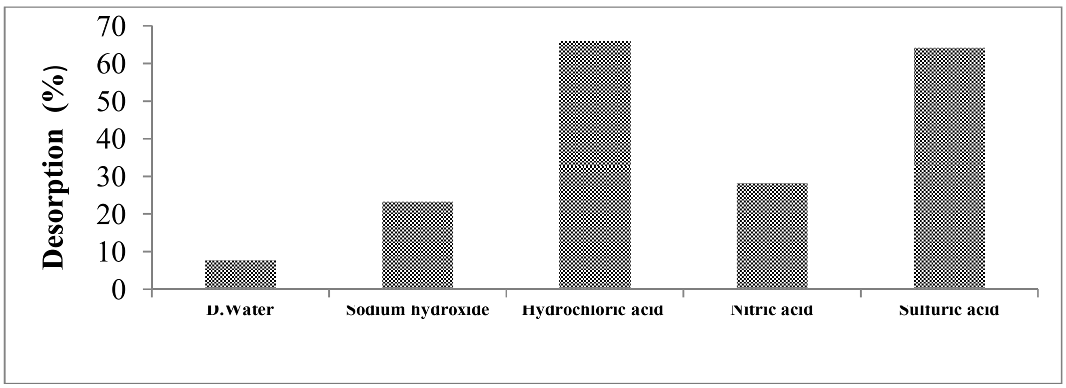

Fig. 10. Effect of various agents on desorption of cadmium

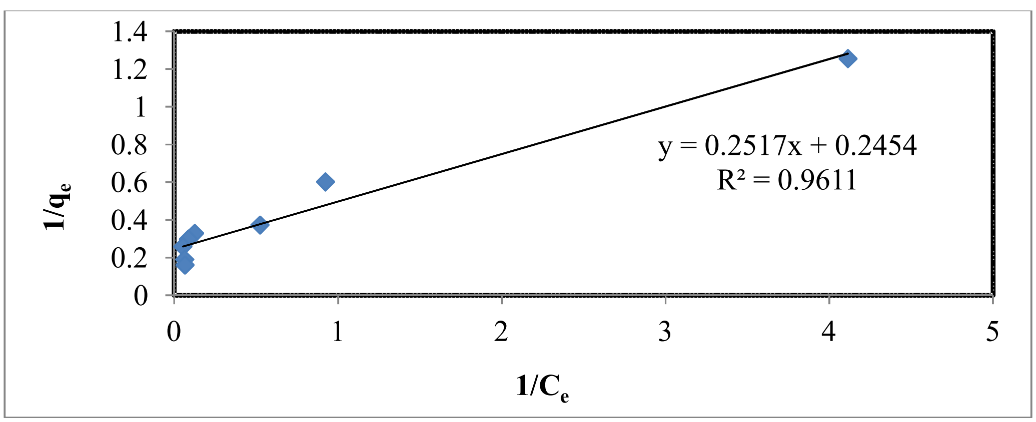

Fig. 11. Langmuir isotherm plot for Cd^{2+} ions

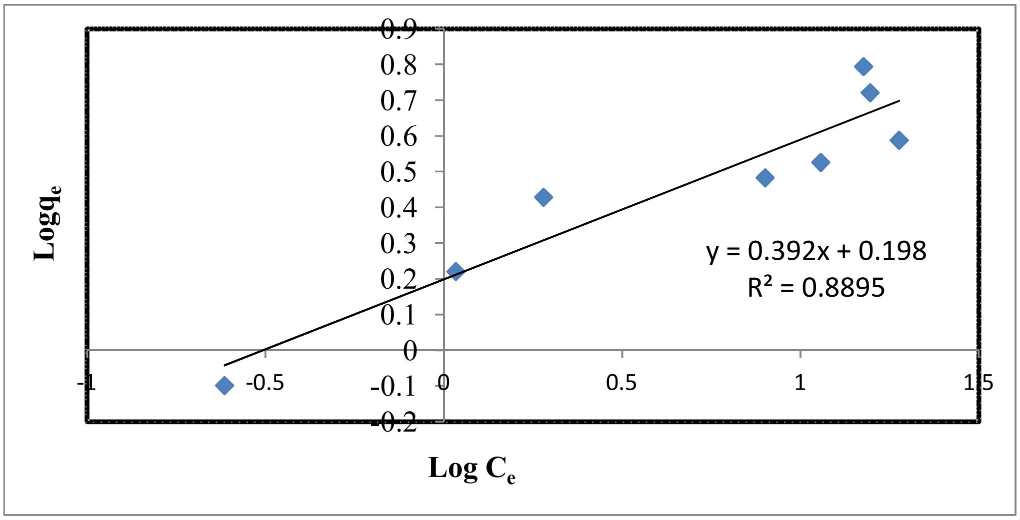

Fig. 12. Freundlich isotherm plot for Cd^{2+} ions

were found to be 2.538. When, *n* values between 1 and 10 represent beneficial biosorption [26]. The 'n' value of Freundlich equation could give an indication on the favourability of sorption. K_f can serve as indicators for the maximal metal cation uptake capacity of the biomass.

4. CONCLUSION

The batch experiment conducted in this study focused on the adsorption of Cd^{2+} ions onto mango leaves from aqueous solution. Adsorption tests have been carried out in order to investigate the effect of pH, adsorbent amount, initial heavy metal concentration, temperature and chelating agent. The obtained results showed that the adsorption equilibrium data is best fitted with the Langmuir model. The characterization studies were performed using scanning electron microscope and fourier transform infrared spectrometer. The desorption experiments suggested that the regeneration of the adsorbents was possible for repeated use especially with regard to Cd^{2+}. Finally, it was concluded that dry mango leaves could be used as an efficient adsorbent material for the removal of cadmium ions from aqueous solutions.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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