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Full length article

Cadmium removal from aqueous solution by green synthesis zero valent silver nanoparticles with *Benjamina* leaves extract

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ABSTRACT

Cadmium (II) is an important element used in various industries, however, it is a poisonous element that affects the health of plants, animals and humans alike. It's very important to remove this element from contaminated waters. This study aims at synthesizing zero valent silver nanoparticles by environmentally ecofriendly method without using hazardous compounds (via green approach). In this work, silver nanoparticles were prepared using hot water for the *Ficus tree (Ficus Benjamina)* leaf extract (FBLE). The size of crystalline for AgNPs was measured by UV–vis spectroscopy and flourier transform infrared (FTIR). The properties of nano-silver particles (AgNPs) have been studied using scanning electron microscope (SEM). The capability of nanoparticles to remove Cd^{2+} from contaminated solution was then studied. Parameter like adsorbent dose, heavy metal concentration, pH, agitation speed and contact time were studied. Cadmium removal increased when the dosage of biosorbent increases, pH increased from 1 to 6, contact time from 5 to 40 and initial concentration of Cd decrease. Isotherm adsorption was also described by the Freundleich model with a constant correlation (R^2) higher than 0.973.

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Introduction

One of the most popular applications for the development and research in technical disciplines is nanotechnology. In the last decade, nanotechnology is one of the most important advances in technology and science. When preparing the nanoparticles, new and unique properties appear to have many benefits such as magnetization. Nanotechnology-derived products can also help reduce concentrations of toxic compounds at sub-levels to meet water quality standards and health reports (Savage and Mamadou, 2005). Nanoparticles are a good solution to address some pollution problems and clean up the contaminated environment. As the size of the material approaches the nanometer, its properties change and the proportion of atoms on the surface of the material becomes larger. According to (Zhang, 2003), nano scale iron particles have high surface reactivity and large surface areas (Zhang, 2003). Many researches have shown that the nanoparticles for iron are effective for the detoxification and transformation of a vast diversity of environmental pollutants, such as organo chlorine pesticides, harmful organic solvents and others contaminates. The characteristics of nanoparticle are characterized by X-ray Diffraction (XRD) and Scanning Electron Microscope (SEM). The average diameter of iron particles ranged from 10 to 30 nm (Ponder et al., 2000) while their size of is known to range from 10 to 100 nm (Sun et al., 2007).

The extraction of cadmium ions from polluted water is important benefit from both economic and environmental due to its high toxicity to animals, plants and humans. Different adsorbents have been used such as peels and wastes of different plants, activated carbons, biopolymers, clay and others. Long term Pollution of drinking water with Lead and Cadmium ions may cause many health problems as convulsions, nausea, coma, cancer, renal failure, and subtle effects on metabolism and intelligence (Deng et al., 2010). Several extraction methods have been tested for the extraction of heavy metal from solution, such as: coagulation, precipitation, ultrafiltration, adsorption, membrane separation and reverse osmosis (Sun et al., 2014; Yang et al., 2009). Recently, adsorption technique has been known as an effective and economic method for heavy metals extraction (Zhang et al., 2013; Cheng et al., 2015; Wang et al., 2011).

Last decade, the development of nanotechnology and Nano science has shown marked ability to address environmental pollution (Lee et al., 2010). Nanomaterials have shown high faster rates and efficiency in water treatment than traditional old materials. Nanoparticles commonly used include zero metals to uptake heavy metal ions from contaminated water (Xu et al., 2010;

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Wang et al., 2010; Xiong et al., 2007; Liu et al., 2010; Liu and Zhang, 2010; Lin et al., 2005).

The objective of this study is to evaluate the capacity of zero valent silver (Ag^0) nanoparticles (AgNPs) synthesized by Fresh *Ficus Benjamina* leaves to remove Cd (II) from aqueous solutions. Batch adsorption process has been used to evaluate the maximum adsorption capacity of zero valent silver (Ag^0) nanoparticles (AgNPs) synthesized by Fresh *Ficus Benjamina* leaves. The main parameters considered are pH, contact time, initial metal ions concentration, agitation speed and adsorbent dose.

Materials and methods

Preparation of AgNPs

Reagents were prepared using chemically pure grade (AnalaR). The fresh plant material was *Ficus tree (Ficus Benjamina*). This plant is from the *Moraceae* family and is one of the most abundant flowering plants in Australia and Asia Fig. 1. The species can also be found in the States of Florida and Arizona in the United States and in the West Indies. *Ficus benjamina* is a tree that in normal conditions can reach 30 meters.

A biomass of *Ficus Benjamina* was used as a biosorbent of Cd (II). Samples of the biomass were collected from the polluted area around second industrial area, Riyadh City, Saudi Arabia. The leaves were washed first with tap water, de-ionized water drying in oven at 50 °C then cut onto small pieces. To prepare the extract of *Ficus Benjamina* leaves, take 20 g of leaves in Erlenmeyer flask (250 mL) then add 100 mL of sterile distilled water, boil the previous solution for 5 min (60 °C). After boiling for 5 min the mixture was filtered and the filtrate will be stored at 4 °C until used as a reducing and stabilizer agent for the preparation of nanoparticles synthesis process.

For synthesis Ag^0 nanoparticles process, take 2 ml of *Ficus Benjamina* leaves extract (FBLE) and add to 25 mL of $AgNO_3$ (1 Mm). The solution changes its color to brown. Finally, the solution stirred for 40 min to form the silver nanoparticles. This experiment was conducted at room temperature (25 °C). The appearance of brown color shows the formation of Ag nanoparticles. Color changes to brown within 20 min shows that the AgNPs is formed. The characterizations of the formed Ag nanoparticles were studied.

The samples were taken at different times and analyzed by UV–Vis spectrophotometer on UV- 1800 (200–700 nm): JASCO V-530 Japan, measuring lead: Varian ICP – AES. FT-IR studies were performed to observe the evolution of nanoparticles of Ag^o. FT-IR spectra have been taken for dried nanoparticles, done by a Fourier-transform infrared spectrophotometer (JASCO FT/IR-410, Japan) from 400 to 4000 cm^{-1} by grinding the samples powder with potassium bromide for FTIR spectra tests. Scanning electron microscopy (SEM): A thin film of nanoparticles was formed on a copper slide covered with carbon, use blotting paper to remove the extraction solution and then dry the film under the mercury lamp for 5 min. Then the film was placed in the vessel and introduced into the microscope.

Preparation of Cd(II) stock solution

Doubly distilled water was throughout employed. Initial solutions with different concentration of Cd (II) were prepared using standard solution of cadmium 1000 mg L^{-1} (Merck, Ltd., UK). The pH adjustment of the solutions was made with aliquots of 1.0 mol l^{-1} of HNO_3 utilizing a pH/mV hand-held meter (Crison pH meter, PH 25).

Removal experiments

The biosorption experiments were carried out by mixing 0.05 g AgNPs in 30 mL solution of Cd (II) with different concentrations (30, 50, 70, 100, 120 and 150 mg/L). The solutions were well shacked onto bath shaker at different agitation speed (from 100 to 700 rpm). The effect of time on the adsorption process was carried out. Samples of 1 ml were collected from flasks at required time intervals 5–70 min and centrifuged for 5 min. The effect of pH on Cd (II) adsorption was carried out at 25 °C for 3 h equilibrium time. The initial pH values were adjusted in range of 1–8 with 0.1 M HCl or 0.1 M NaOH. The concentration of Cd(II) was estimated by Atomic Absorption Spectrophotometer (model: AAS 240 FS). The percentage of the removal of Cd (II) from polluted water was estimated using the formula:

$$\% \text{ removal} = \frac{Co - Ce}{Co} \times 100 \quad (1)$$

whereas, C_e: the final concentration of Cd(II) mg L^{-1} and C_o: the initial concentration of Cd(II) mg L^{-1}.

Assessment of adsorption performance

Adsorption isotherm is very important to understand and explain how the solutes are interacting with the adsorbent. Two models were used to explain the removal process. Freundlich (Eq. (2)), Langmuir (Eq. (3)) isotherm were organizing by using straight-line equation and all its parameter for cadmium (II) ion was calculated from their particular graphs. The straight-line



Fig. 1. Ficus tree (*Ficus benjamina*).

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equation for Freundlich (2) and Langmuir (3) models are written as follows:

$$\log\ qe = \log\ kf + \frac{1}{n}(\log\ Ce) \quad (2)$$

$$\frac{Ce}{qe} = \frac{1}{Qob} + \frac{Ce}{Qo} \quad (3)$$

where q_e (mg g^{-1}) is the adsorption capacity of Cd (II) at equilibrium, k_f (mg g^{-1}) is the Freundlich constant, "n" the Freundlich indicator, C (mg L^{-1}) is final concentration of Cd(II), b (L mg^{-1}) and Q_0 (mg g^{-1}) are Langmuir constants related to energy and capacity of adsorption .

Results and discussion

Characterization of nanoparticles

FT-IR spectroscopy is very necessary to prove that nanoparticles are formed. It helps in studying the chemical composition of prepared Silver nanoparticles (AgNPs). FT-IR spectrum of (AgNPs) formed by FBLE is shown in Fig. 2. The band at 3461 cm^{-1} indicates a hydroxyl group (O—H) stretching vibrations. There is also band at 1632 cm^{-1} indicating the presence of the amide group in the formed nanoparticles. It is possible that the amide band is formed because of N—H deformation vibrations and carbonyl stretch in the amide relation of proteins existing in it (Wu et al., 2009). The presence of these bands indicates the existence of proteins, polyphenols and flavonoids in FBLE which seem to reduce the formation of AgNPs. The FT-IR study assured that the FBLE has the capability to do double functions of stabilization and reduction of Ag nanoparticles.

The color change to brown in reaction mixture (metal ion solution + FBLE) indicates the formation of Ag^0 nanoparticles. The color change was on record out of visual observation. Ag^0 nanoparticles formed were characterized by UV–Visible spectrophotometer. The observed band positioned at 420 nm indicates the formation of Ag nanoparticles (Fig. 3). The UV–Vis spectra are accurate, sensitive to the size and shape of nanoparticles, medium refractive index and its interaction with the local. The wide range of UV–Vis spectra indicates the presence of nanoparticles with a wide size distribution (Suman et al., 2013).

Scanning electron microscope (SEM) is used to define the particles growth. It can also use to measure the particle size. After finishing the preparation of nanoparticles, the nanoparticles were placed on carbon coated copper. Fig. 4 shows the image of scanning electron microscopy (SEM) of the formed AgNPs particles. Fig. 4 shows that the particles formed are within the range of nanoparticles and the size of these particles range around 60–105 nm (Sosa et al., 2003).

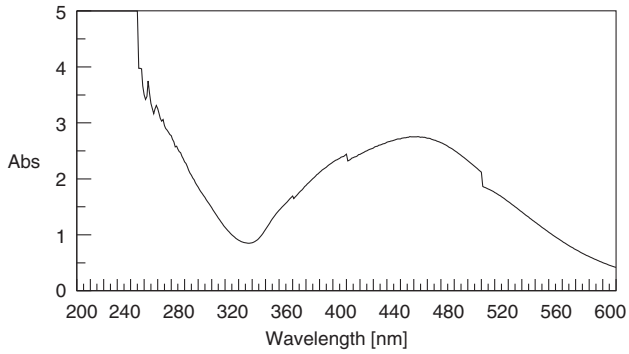


Fig. 3. UV–Visible Spectra of the synthesized AgNPs.

Influence of adsorption dose

Study on the effect of adsorbent dose for Cd(II) removal is important to get the trade-off between the adsorbent dose and the percentage removal of Cd(II) resulting in an optimum adsorbent amount. The influence of adsorbent amount, varying from (0.01 to 0.1 g) onto the Cd(II) adsorption at different initial Cd(II) concentration (50 and 100 mg.l^{-1}). The contact time was stated at 40 mins. The results, represented by Fig. 5, indicated that as the dosage of AgNPs increases, the Cd (II) percent removal increases till reach to stable level. The reason is because there are many sites of the surface of AgNPs to correlate with Cd(II) ions. The increase in the removal of cadmium from the solution by nanoparticles is attributable to the increased dose to increase surface area and absorption site.

Effect of contact time

Effect of contact time on Cd(II) removal were studied at different initial concentrations 50 and 100 mg.l^{-1} keeping pH at 5, adsorbent dose at 0.05 g, stirring speed at 150 rpm and different contact times (5–80 min), (Fig. 6). The results indicated that the Cd(II) removal percentage recorded the highest values at 40 min, which means that the equilibrium time of 40 min was necessary

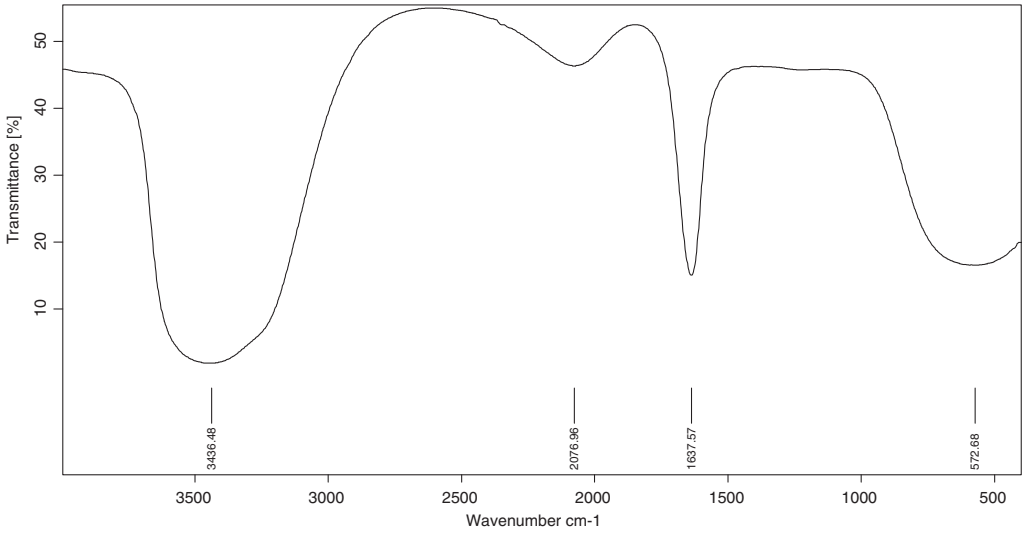


Fig. 2. IR Spectra of the synthesized AgNPs.

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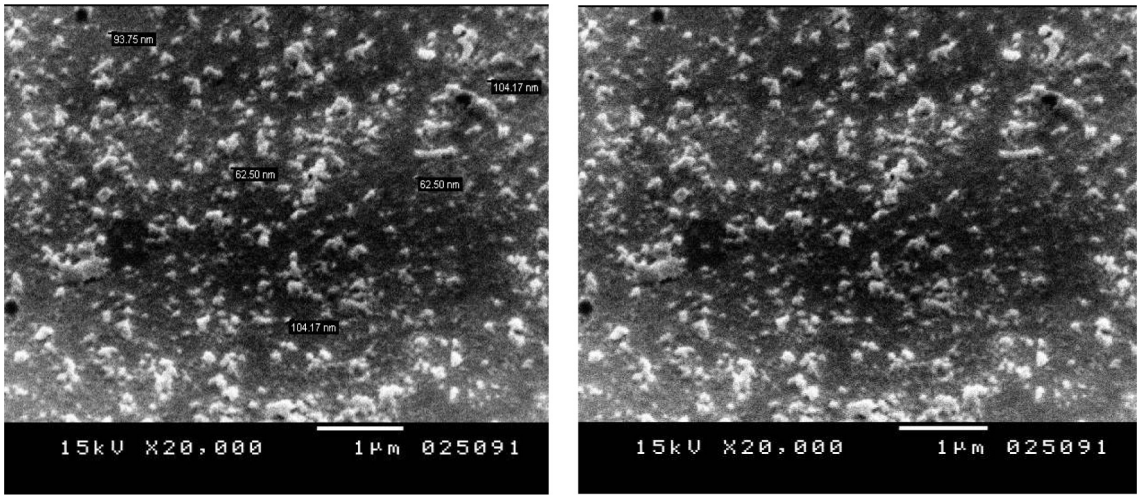


Fig. 4. SEM image of AgNPs using FBLE at 1 µm/20,000X.

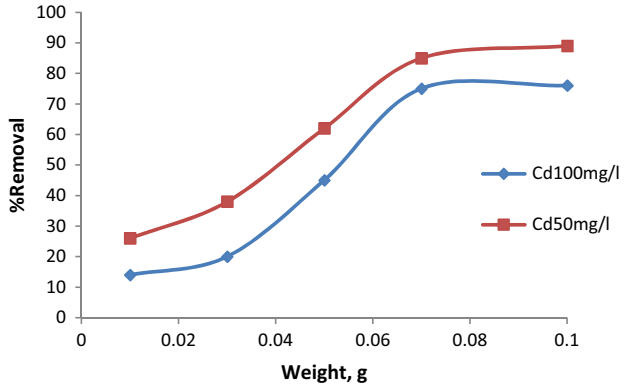


Fig. 5. The effect of dosage on Cd(II) removal by AgNPs.

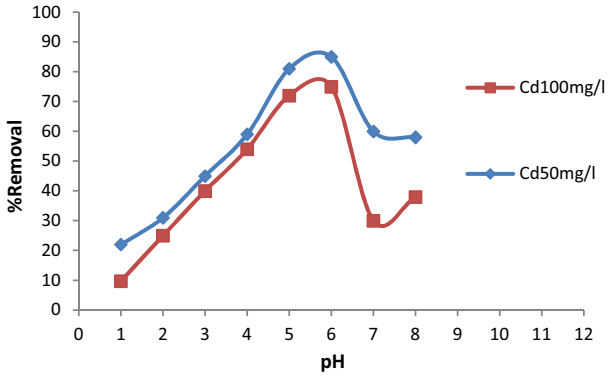


Fig. 7. The effect of pH on the Cd(II) removal by AgNPs.

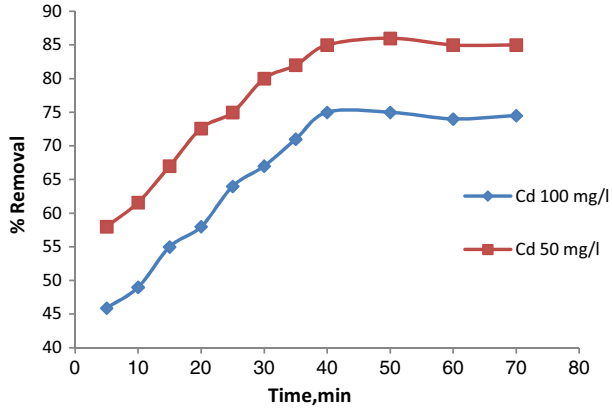


Fig. 6. The effect of time on the Cd(II) removal by AgNPs.

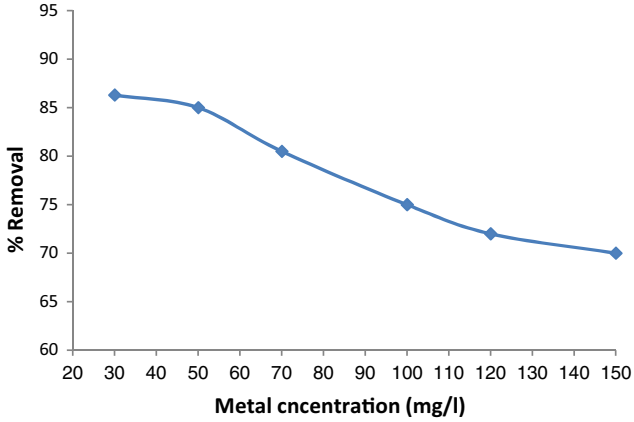


Fig. 8. The effect of metal concentration on the Cd(II) removal by AgNPs.

to reach the equilibrium for Cd(II) removal on AgNPs. Over time, there was no increase in Cd(II) removal from the solution. It is known that the rate of removal of metals is influenced by many factors, leading to the mass transfer from a mass solution to binding sites (Hanif et al., 2007).

Effect of solution initial pH

The effect of pH of the solution on Cd(II) removal onto AgNPs was studied at different values of pH ranging from (1–8) with no change of all parameter: contact time (40 min), dosage (0.05 g) and the initial metal concentration (50, 100 mg/L). The relation between the initial pH of the solution and the percentage removal of Cd (II) is shown in Fig. 7. It was found that the biosorption rate of Cd(II) is all parabolic, which indicates that there exists an optimum range of pH value. The percentage removal of Cd (II) is increased from 10.3 to 75.5% in case of 100 mg. L^{-1} and from 21.4 to 85% in case of 50 mg L^{-1} when the pH increased from 1 to 6. The sharpest increase in Cd (II) uptake was obtained at pH = 6.

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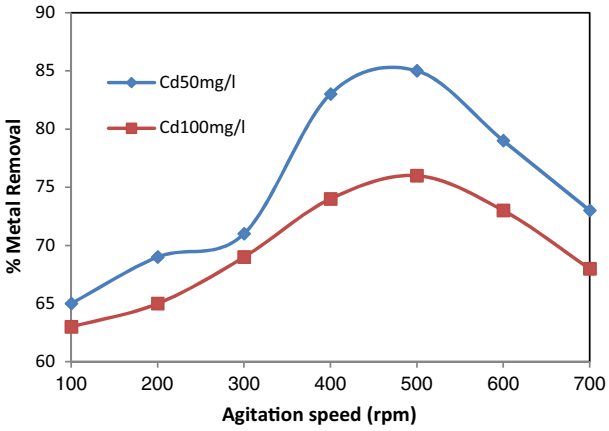


Fig. 9. The effect of agitation speed on Cd(II) removal by AgNPs.

Table 1
Kind of adsorption isotherm based on value of RL.

RL	Type of Isotherm
0 < RL < 1	Favourable
RL > 1	Unfavourable
RL = 0	Irreversible
RL = 1	Linear

Effect of initial concentration on Cd (II) removal

Effect of initial concentration on Cd (II) removal by AgNPs was studied at different initial concentrations ranging from 30 to 150 mg. l^{-1} keeping pH = 6, adsorbent dose (0.05 g), contact times (40 min). The removal percentage depends mainly on the initial metal concentration. As shown in Fig. 8, Cd(II) ions removal at various concentrations show that the removal percentage diminishes with increases in the metal concentrations, whereas the practical amount of adsorbed Cd(II) ions increases, which shows a significant relationship between the removal efficiency and initial metal concentration. In case of low metal concentrations, more vacant sites are available for adsorption which results in an increase in the concentration slope and rate of cadmium ions spread to adsorbent. In the case of high concentration of metal, the obtainable sites of nanoparticles decrease and therefore the removal percentage of Cd(II) ions decreases Sankar et al., 2013.

Effect of agitation speed on Cd (II) removal

The experiment was performed at agitated speed ranging from 100 to 700 rpm on a magnetic shaker. The results show that when the agitation speed increases from 100 to 500 rpm, Cd(II) removal percentage by AgNPs increases and then the removal percentage decreases after 500 rpm (Fig. 9). The reason can be attributed to the fact that increasing in the agitation speed reduces the resistance to move the mass around the adsorbent particle (Ramalingam et al., 2013).

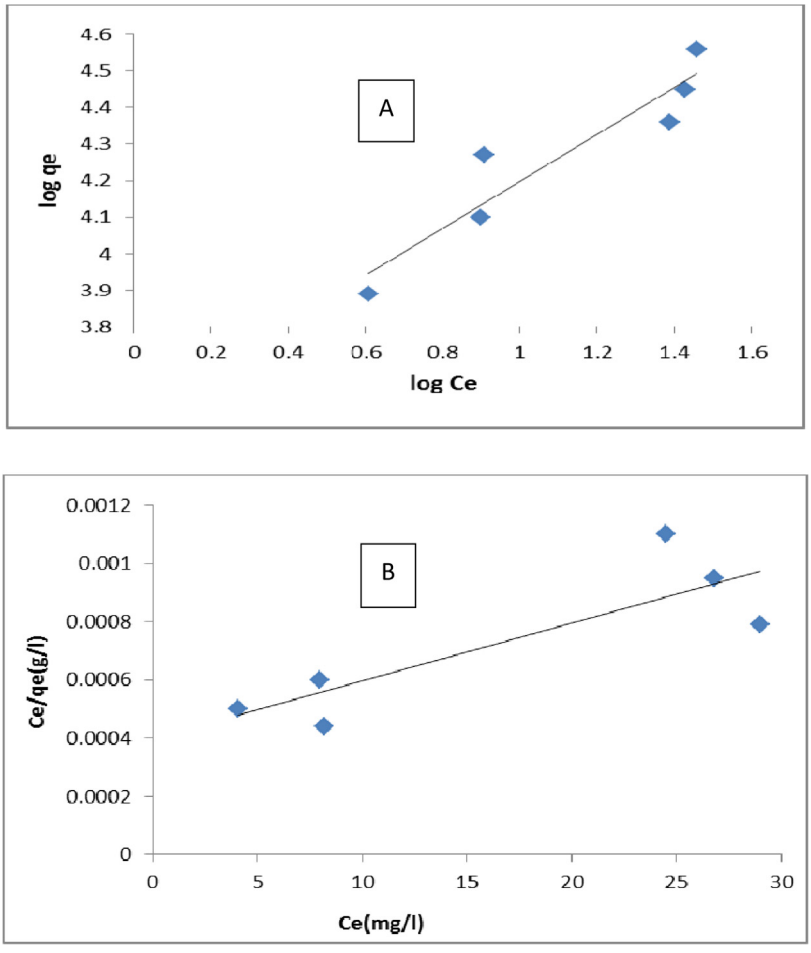


Fig. 10. Freundlich (A) and Langmuir (B) diagrams for Cd (II) removal from polluted water.

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Table 2
Adsorption isotherm parameters for Cd(II) removal from polluted water by AgNPs.

Langmuir				Freundlich			
Metal	Q_o(mg g^{-1})	b ($g\ l^{-1}$)	RL	R^2	n	K_f	R^2
Cd(II)	0.845	0.24	0.9	0.845	0.940	1.001	0.974

Adsorbent isotherms

Different adsorption isotherm models are used to study the surface property of AgNPs and its particle convergence for Cd (II) removal. Of the results obtained from Cadmium uptake by AgNPs using linear equation of both Freundlich and Langmuir isotherm models. In case of the Langmuir model, it occurs in cases where adsorption of homogeneous and monolayer surfaces. In case of the Freundlich adsorption model, it is supposed that adsorption occurs in heterogeneous surfaces.

The Langmuir isotherm dimensions constant separation parameter (R L) determines the nature of adsorption and gives important information on the type of adsorption. It was calculated using the following equation (Monika et al., 2010):

$$RL = \frac{1}{(1 + bQo)} \quad (5)$$

The feasibility of adsorption method is fixed in Table 1 with R L values that deduces the type of adsorption isotherm. Values of adsorption isotherm parameters are calculated and are shown in Fig. 10, and Table 2

The correlation coefficients, $R^2 > 0.96$, obtained from Freundlich model are comparable to that obtained from Langmuir model linear form (Fig. 10). This result indicates that the experimental data fitted well to Freundlich model but is not good as Langmuir model. The adsorption of Cd (II) on the surface of the AgNPs occurred by multiple layers of heterogeneous of the adsorbent surface. The high n value (0.940) indicates that Ag nanoparticles are better adsorbent for uptake of Cd (II) from aqueous solution. The RL values (0.9) range from (0 −1) indicated that the type of adsorption is favorable. All adsorption values obtained in this study are similar to the absorption values by Sheela (Sheela et al., 2012).

Conclusion

Nanoparticles were prepared for Ag in the laboratory using extract of *Ficus Benjamina* leaves and $AgNO_3$. The AgNPs were characterized with SEM, FTIR and UV. The results of SEM showed that the AgNPs prepared are in the nanoscale and dendritic structure. The results show the effectiveness of the method used in the preparation of nanoparticles and the stability of the prepared particles and their effectiveness in water purification and environmental remediation. The preparation of AgNPs in this way has several good properties such as high reactivity, easy preparation and low cost compared to other methods of preparation of metal nanoparticles. Behavior of Cd(II) removal onto AgNPs were inquired in the batch experiment in this study. Through the results, the removal of metal ion depends on adsorption dosage, concentration of the metal, interaction time, pH and agitation speed. Furthermore the equilibrium data of removal shown that Freundlich isotherm model for the cadmium removal was best when compared with Langmiur isotherm models. The data thus obtained may be helpful for designing and establishing a continuous treatment plant water and wastewater enriched in Cd(II).

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