Synthesis, Characterization and Application of Zero Valent Silver Nanoparticles for the Removal of Toxic Metal Pb (II) from Aqueous Solution

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Abstract: Lead is a heavy metal used in a variety of industrial applications which is highly toxic to humans, animals, plants and microorganisms. Therefore, it should be removed from contaminated waters. The main objective of this study was to synthesize silver nanoparticles via green approach (environmentally ecofriendly method) without using hazardous compounds. Here we present the distinctive properties of the silver nanoparticles (AgNPs) synthesized using hot water *Ficus tree (Ficus benjamina)* leaf extract (FBLE) a reducing and capping agent. The nature of AgNPs synthesized was analyzed by scanning electron microscope (SEM), flourier transform infrared (FTIR) and UV–vis spectroscopy (UV). We explored the ability of the AgNPs for removing Pb^{2+} from aqueous solution. The lead stock solution was treated with AgNPs by varying time and amount of nanoparticles in batch studies. Parameters like Heavy metal concentration, adsorbent dose, pH, contact time and agitation speed were studied. Langmuir and Freundlich isotherms were employed to describe adsorption equilibrium. The adsorption isotherms were well described by Freundlich isotherm model with correlating constant (R^2) higher than 0.950.

Key words: Adsorption Isotherms · Silver Nanoparticles · Green Synthesis · Pb(II) · SEM · UV-Vis Spectrophotometer · Aqueous Solution

INTRODUCTION

The field of nanotechnology is one of the most popular areas for current research and development in basically all technical disciplines. Nanotechnology is considered to be one of the most important advancements in science and technology in the past decade. At nano scale, materials exhibit unique properties that can be used for novel applications like magnetization. Additionally, nanotechnology derived products that reduce the concentrations of toxic compounds to sub-ppb levels can assist in the attainment of water quality standards and health advisories [1]. Nanoparticles represent a new generation of environmental remediation technologies that could provide cost-effective solutions to some of the most challenging environmental cleanup problems. The properties of materials change as their size approaches the nano scale and as the percentage of atoms at the surface of a material becomes significant. Nano scale iron particles have large surface areas and high surface reactivity [2]. Research has shown that nano scale iron particles are very effective for the

transformation and detoxification of a wide variety of common environmental contaminants, such as chlorinated organic solvents, organochlorine pesticides and PCBs (Polychlorinated biphenyls). Nanoparticle characterization is done by Scanning Electron Microscope (SEM), X-Ray Diffraction (XRD). The average iron particle diameter of nZVI (Nanoscale zero-valent iron) particles ranges from 10-30 nm [3]. The particle size of iron nanoparticles ranges from tens to hundreds of nanometers [4].

Lead in the natural environment arises from both natural and anthropogenic sources and is detrimental to human health and all living things [5]. All lead compounds are considered cumulative poisons. Recently, increasing interest has been focused on removing Pb (II) ions from drinking water due to its supreme toxicity to our health [6]. Drinking water contaminated with Pb (II) ions for long term, even if in a very low concentration, could lead to a wide spectrum of health problems, such as nausea, convulsions, coma, renal failure, cancer and subtle effects on metabolism and intelligence [7, 8]. A number of methods have been examined for the removal of lead from water, such as: precipitation, coagulation, adsorption,

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ultrafiltration, reverse osmosis and membrane separation [9, 10]. Among them adsorption is recognized as an effective and economic method for heavy metals removal [11-13].

In recent years, the development of nanoscience and nanotechnology has shown remarkable potential for the remediation of environmental problems [14]. Compared with traditional materials, nanostructure adsorbents have exhibited much higher efficiency and faster rates in water treatment. The generally used nanoparticles include zero-valent metals [15-21] to remove heavy metal ions from aqueous solution.

In the present study, we have synthesized these FBLE capped AgNPs and applied these AgNPs for adsorbing heavy metals from aqueous solution. Mean while the influence of experimental parameters such as pH, adsorbent dose, contact time, initial concentration, agitation speed and the adsorption isotherm were revealed.

MATERIALS AND METHODS

Synthesis and Characterization of FBLE capped AgNPs:
Preparation of Materials: The raw plant material used in the present study was *Ficus tree (Ficus benjamina)*. This is a species of flowering plant in the family *Moraceae*, native to Asia and Australia (Fig. 1). AR grade SD fine silver nitrate (AgNO$_3$) was purchased and its 0.1 M solution was prepared in stock and diluted to 1 mM solution. The other chemicals and reagents were of chemically pure grade (Analar).

Preparation of FBLE: Fresh *Ficus Benjamina* leaves were collected and washed with sterile distilled water and dried. After drying cut in to small pieces. The extract was prepared by taking 20 g of thoroughly washed finely cut *Ficus Benjamina* leaves in a 250 mL Erlenmeyer flask with 100 mL of deionized water and then boiling the mixture at 60°C for 5 min. After boiling, the solution was decanted and filtered through nylon mesh (Spectrum). The filtrate is used as reducing agent and stabilizer, was stored at 4°C for further nanoparticles synthesis process.

Green Synthesis of AgNPs: The chemicals used in this study such as AgNO$_3$ was of analytical grade. For synthesis process, the FBLE of 2 mL was added to 25 mL of 1 mM AgNO3 aqueous solution and the resulting solution became brown in color. Then the mixture was stirred for 30 min to obtain the AgNPs. Here the formation of AgNPs was identified by change in

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

the color of the stock solution to brown within 20 min [22]. Further the extract and aqueous solution ratio was varied. These biologically-reduced aqueous solutions of Ag nanoparticles were used for further characterizations. This process was carried out at room temperature.

Spectral Analysis: The synthesized AgNPs were confirmed by sampling the aqueous component of different timeintervals and the absorption maxima was scanned by UV-Vis spectrophotometer at the wavelength of 300-800 nm on UV-1800: JASCO V-530 Japan, measuring lead : Varian ICP - AES. The infrared spectra were achieved in a Shimadzu FT-IR spectrophotometer IRAffinity^{-1} by employing KBr pellets and registering amplitude waves ranging from 400 to 4000 cm^{-1}.

Morphological Analysis: Morphological analysis was done using Philips model CM 200 SEM machine. Thin films of the sample were prepared on a carbon coated copper grid by just dropping a very small amount of the grid, extra solution was removed using a blotting paper and then the films on the SEM grid were allowed to dry by putting it under mercury lamp for 5 min.

Treatment of Water Samples Containing Pb (II):
Preparation of Pb (II) Stock Solution: All the chemicals used were of analytical reagent (AR) grade purchased from Merck, Germany. Stock solutions of 1000 mg/L of lead (II) was prepared from lead nitrate [Pb (NO$_3$)$_2$] using double distilled water. Desired test solutions of Pb (II) ions were prepared using appropriate subsequent dilutions of the stock solution. The range of concentrations of Pb (II) ions prepared from standard solution varied between 10 and 100 mg/L. Before mixing the adsorbent, the pH of each test solution was adjusted to the required value with 0.1 M NaOH or 0.1 M HCl.

Batch Adsorption Studies: Adsorption studies were carried out in a batch mode by shaking 0.1 g AgNPs in 30 mL solution of Pb (II) with concentration range from 30 to 150 ppm onto the water bath shaker at 100 rpm stirring speed. Furthermore the adsorption studies were also carried out by varying time interval (5–70 min.) at 50,100 ppm concentration of Pb (II) to optimize the time required for the removal of Pb (II) from aqueous solution. The equilibrium adsorption capacity of AgNPs was estimated with the help of following equation (1):

$$qe = \frac{Co - Ce}{W} X\ V \qquad (1)$$

where q_e is the equilibrium adsorption capacity (mg g^{-1}), C_o is the metal ion initial concentration (mg L^{-1}), Ce is the metal ion concentration (mg L^{-1}) at equilibrium, V is the volume of solution (L) and W is the weight (g) of adsorbent.

Study of Adsorption Isotherm: Varying metal ion solutions with concentration 30, 50, 70, 100, 120 and 150 gmL^{-1} were made by proper dilution of stock solution of Lead.

pH was adjusted to (1-8). 0.1 g of biosorbent was added to 30 mL of metal solution and was agitated for half an hour. At the end it was filtered and the filtrate with added suitable reagent was analyzed for metal ion by UV-visible spectrophotometer.

Assessment of Sorption Performance: Freundlich (eq.2), Langmuir (eq.3) isotherms were plotted by using standard straight line equation and corresponding parameter for lead (II) ion was calculated from their respective graphs.The linear equation for Freundlich and Langmuir [23,24] isotherm models are expressed as follows:

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

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

where kf (mg g^{-1}) is the Freundlich constant and "n" the Freundlich exponent. Where qe (mg g^{-1}) is the adsorbed amount of Pb (II) at equilibrium, Ce (mg L^{-1}) is the equilibrium concentration of Pb (II), Q_0 (mg g^{-1}) and b (L mg^{-1}) are Langmuir constants related to adsorption capacity and energy of adsorption.

RESULTS AND DISCUSSION

Characterization: FT-IR spectroscopy is used to probe the chemical composition of the surface of the AgNPs and the local molecular environment of the capping agents on the nanoparticles. The FT-IR spectrum of FBLE mediated AgNPs is shown in Fig. 2. The band at 3436 cm^{-1} corresponds to intermolecular O-H stretching vibrations. The medium absorption peak located at 1637 cm^{-1} is identified as the amide group. This amide band occurs due to carbonyl stretch and N–H deformation vibrations in the amidelinkage of proteins present in it [25]. All these bands clearly confine the presence of polyphenols, proteins, tannins and flavonoids in FBLE which act as reducing agents for the synthesis of silver nanoparticles. Thus, the IR spectroscopic study confirmed that the FBLE has the ability to perform dual functions of reduction and stabilization of AgNPs.

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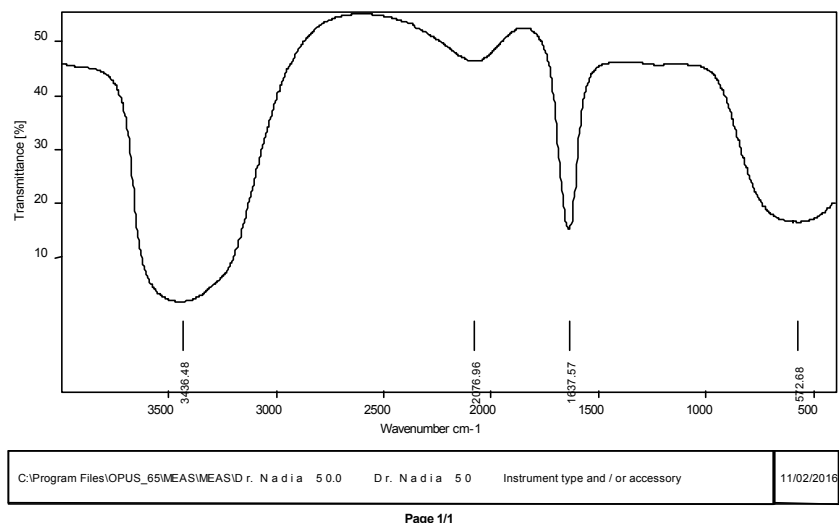
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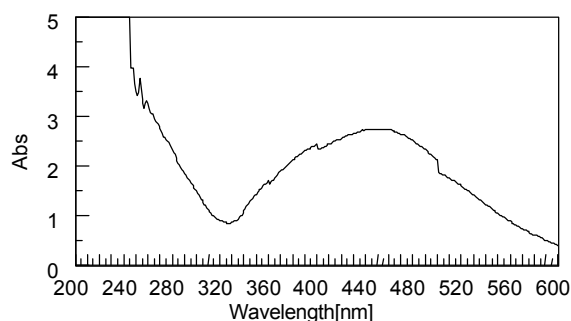Fig. 2: The FT-IR spectrum of FBLE mediated AgNPs.

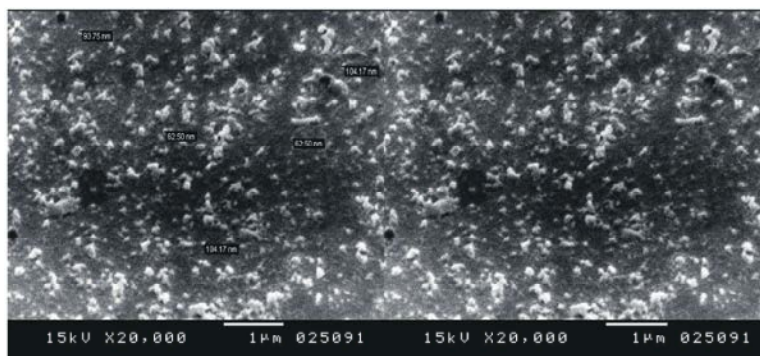Fig. 3: UV-Visible Spectra of the synthesized AgNPs.

Fig. 4: SEM image of AgNPs using FBLE leaf extract

The color change in reaction mixture (Metal ion solution + FBLE) was recorded through visual observation. The color change showed the presence of silver nanoparticles in the leaf extract and it was characterized by UV-Visible spectrophotometer. The strong surface plasmon resonance band positioned at 420 nm was observed for AgNPs (Fig. 3). The position of SPR band in UV–Vis spectra is sensitive to particle shape, size, its interaction with the medium, local refractive index and the extent of charge transfer between medium and the particles. The broad spectra indicate the presence of particles with a broad size distribution [26].

The synthesized nanoparticle morphology was characterized by scanning electron microscope (SEM). After the completion of reaction, the nanoparticles placed on carbon coated copper grid, it exhibited spherical in shape (Fig. 4). Further, from all the SEM images it was evident that the morphology of AgNPs is nearly spherical which is in good agreement with the shape of SPR band in the UV–Vis spectra. The size of the AgNPs ranges around 60 -105nm [27].

Batch Studies:
Effect of Adsorbent Dose: Effect of adsorbent dosage on adsorption of Pb (II) was studied by changing the biosorbent dosage from 0.001 to 0.2 g and there was no
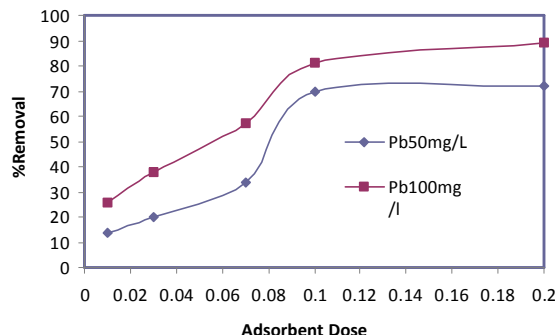

Fig. 5: Effect of dosage on the adsorption of Pb (II) by AgNPs.

change in other parameter like pH and initial concentration (50 and 100 mg/L). The contact time was 35 mins for Pb as stated earlier. The graph has shown an increase in the biosorption percentage as dosage of biosorbent increases to certain level and then it was stable (Fig. 5). This is because of the availability of more binding sites in the surface of the biosorbent for complecation of Pb (II) ions.

Effect of Contact Time: Contact time profile for the biosorption of Pb (II) for a solution of 50 mg/L and 100 mg/L is shown in the data obtained from the biosorption of Lead ions on AgNPs showed that a contact time 35 min
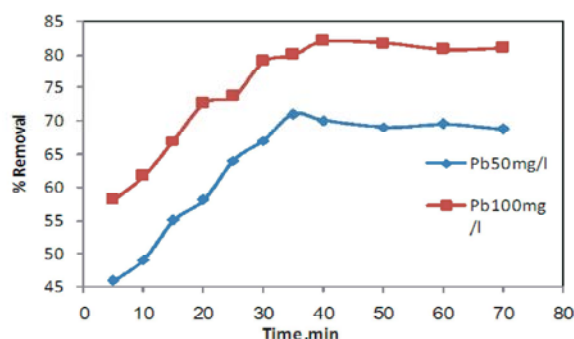
Fig. 6: Effect of time on the adsorption of Pb (II) by AgNPs.
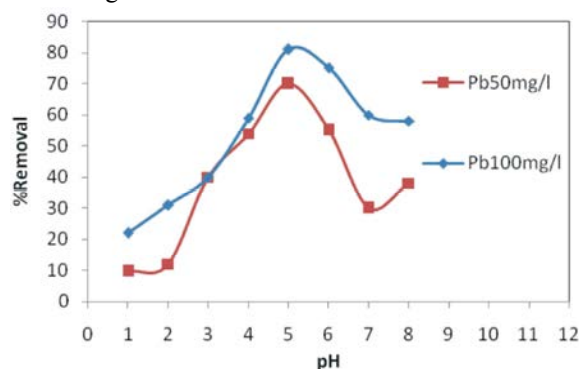

Fig. 7: Effect of pH solution on the removal of Pb(II) from aqueous solution by AgNPs.
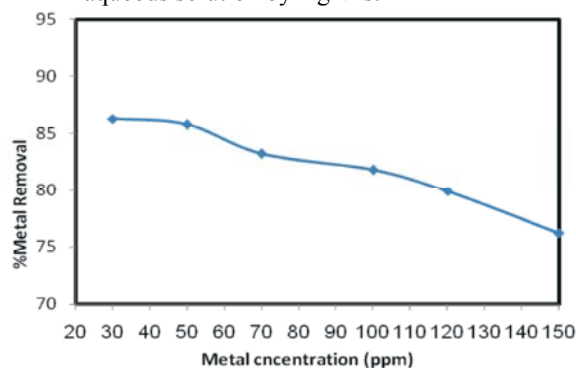

Fig. 8: Effect of initial metal concentration on the removal of Pb(II) from aqueous solution by AgNPs (pH = 5, contact time 35 min, adsorbent 0.1 g, temperature 25±1 °C).

is needed to achieve equilibrium and the biosorption decreased significantly with further increase in contact time (Fig. 6).

Effect of pH: The effect of the solution pH on the adsorption of Pb (II) ions onto AgNPs was assessed at different values, ranging from (1 -8), with a stirring time of 35 min. In these experiments, the initial metal
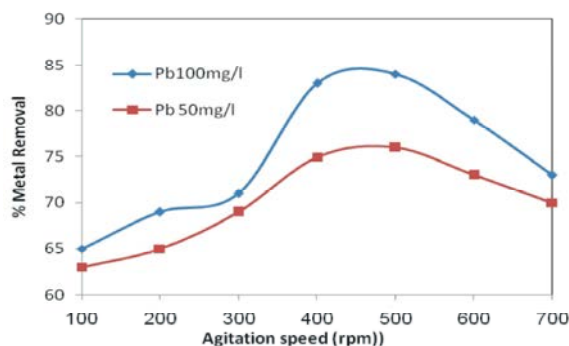

Fig. 9: Effect of agitation speed on the adsorption of Pb(II) by AgNPs.

concentration and the amount of adsorbent were set at 50, 100 mg L^{-1} and 0.1 g, respectively, for all batch tests in this experiment. Fig.7, represents the results of the effect of the solution pH on the removal efficiency of Pb (II) ions and as it shows, removal of Pb (II) increased from 10.3 to 70.5% in case of 50 mg L^{-1} and from 21.4 to 85 % in case of 100 mg L^{-1} when the pH increased from 1 to 5.

Effect of Initial Concentration: The removal percentage for adsorption of Pb (II) ions using the AgNPs was studied by varying lead ion concentrations from (30, 50, 70, 100, 120 and 150 mgL^{-1}) while keeping adsorbent quantity, pH, temperature and contact time constant. The adsorption capacity is dependent on the initial metal ion concentration. As seen in Fig. 8, the Pb (II) ions adsorption at various concentrations reveals that the removal percentage diminishes with increases in the initial metal concentrations, while the actual amount of adsorbed Pb(II) ions per unit mass of adsorbent increases, which shows a significant relationship between the adsorption efficiency and initial metal concentration. At lower metal concentrations, more vacant sites are available for adsorption which results in an increase in the concentration gradient and rate of Pb (II) ions diffusion to adsorbent. At high concentration of metal, the available sites of adsorbent become fewer and hence the removal percentage of these Pb (II) ions decreases [28].

Effect of Agitation Speed: The agitated speed varied from 100 to 700 rpm was carried out with a magnetic shaker. As agitation rate on adsorption increased from 100 to 400 rpm, adsorption capacity of the AgNPs increased and then decreased (Fig. 9). The effect of increasing the agitation rate was to decrease the filter resistance to mass transfer surrounding the adsorbent particle.
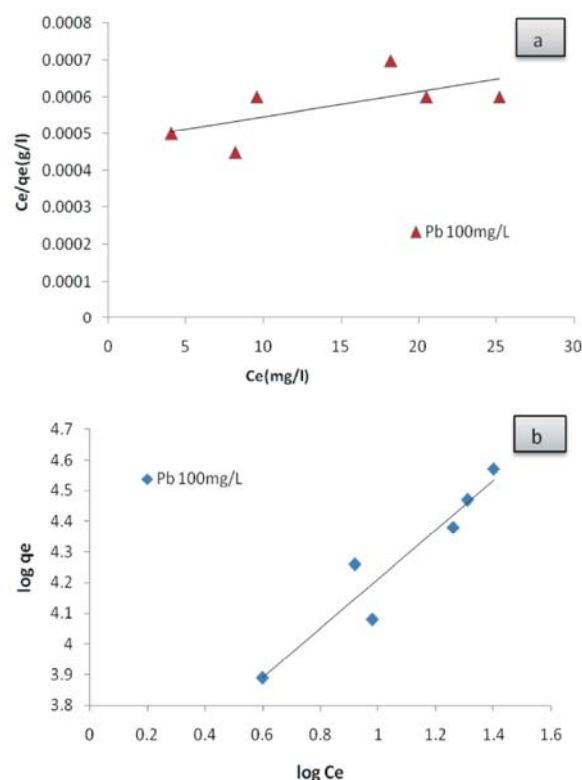
Fig. 9a&b: Langmuir and Freundlich plots for the removal of Pb (II) from aqueous solution.

Table 1: Nature of adsorption isotherm and the feasibility of adsorption process

RL	Adsorption process
RL > 1	Unfavorable
RL = 1	Linear
0< RL< 1	Favorable
RL = 1	Irreversible

Table 2: Adsorption isotherm parameters for the adsorption of Pb (II) from aqueous solution onto the AgNPs.

	Langmuir			Freundlich			
Metal ion	Qo(mg/g)	b g/l	RL	R^2	n	Kf	R^2
Pb(II)	0.885	0.25	0.8	0.645	0.995	1.001	0.954

Adsorbent Isotherms: The surface property and affinity of AgNPs for Pb (II) removal can be determined using the different adsorption isotherm models. The obtained equilibrium data from the adsorption of Pb (II) onto the AgNPs fitted to the linear equation of Freundlich, Langmuir isotherm models. The Freundlich adsorption model assumes that adsorption takes place on heterogeneous surfaces.The Langmuir model is applied in cases where monolayer adsorption occurs on homogeneous surfaces.

The essential characteristics of the Langmuir isotherm can be described in terms of a dimensionless constant *viz.,* separation factor or equilibrium parameter, RL, which is defined by the equation [29]:

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

The value of RL infers the nature of adsorption isotherm and the feasibility of adsorption process are indicated in Table 1. The all values of applied adsorption isotherm parameters were calculated from their respected plots, which are included here in Fig. 9 a and b, reported in Table 2. Based on the higher values of correlation coefficient R^2 for Freundlich model compared to the Langmuir model, adsorption data was better described by Freundlich model. This implies that the adsorption of Pb(II) on the surface of the AgNPs occurred by multiple layers of heterogeneous of the adsorbent surface. The n value was found to be o.995 for the Pb(II) adsorption onto the AgNPs from aqueous solution. These values are suggesting that AgNPs is better adsorbent for the separation and removal of Pb (II) from aqueous solution.

The values of RL are observed to be fraction, in the range of 0 to 1 (0.8) which indicate that the adsorption process is favorable for all these adsorbents. The obtained results for the Pb adsorption are similar to the reported adsorbent by Sheela *et al*. [30].

CONCLUSIONS

AgNPs were successfully synthesized in the laboratory using AgNO₃. The particles were characterized with SEM, FTIR and UV. SEM results indicated that the synthesized AgNPs particles show dendritic structure and most of the AgNPs particles are in nano scale. The characterization results clearly showed that the present synthesis method would be useful to synthesize and could solve long pending stability issue of AgNPs for its versatile applications in environmental remediation and water purification. The synthesized nanozero valent Ag material showed dendritic texture of crystal growth. AgNPs has several advantages such as low cost, easy preparation and high reactivity compared to other metal nanoparticles. In this study the adsorption behavior of Pb (II) onto AgNPs was investigated in the batch experiment. The adsorption was found to be drastically depending on initial metal ion concentration, adsorption dosage, contact time, pH and agitation speed. Furthermore the equilibrium data of adsorption indicated that Freundlich isotherm

model for the Pb (II) adsorption was best fitted in comparison with Langmuir isotherm models. This adsorption can be a good choice for adsorption of not only Pb (II) ions but also other heavy metal ion in waste water stream.

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