



RESEARCH ARTICLE

REMOVAL OF LEAD IONS BY GREEN SYNTHESIZED SILVER NANOPARTICLES USING AQUEOUS EXTRACTS OF *PROSOPIS JULIFLORA* AND *MENTHA PIPERITA* L. LEAVESElham Abdalrahem Bin Selim¹, Rokhsana Mohammed Ismail^{2,*}, Suhair Ruban³¹Dept. of Chemical, Faculty of Science, University of Hadramout, Hadramout, Yemen.²Dept. of Chemical, Faculty of Science, University of Aden, Aden, Yemen.³Dept. of Chemical, Faculty of Education-Aden, University of Aden, Aden, Yemen.

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Received: 2 June 2024 / Accepted: 14 June 2024 / Published online: 30 June 2024

Abstract

Heavy metal compounds are commonly used as required industrial materials. Heavy metal ions are known to possess poisonous properties. Lead (II) ion is one of them; it is more toxic and carcinogenic. The contamination of water with Pb (II) may lead to kill people who drink it, in addition to harmful effects on plants and animals. Therefore, Pb (II) ions must be removed from the polluted water. In this work, an eco-friendly method was employed to synthesize silver nanoparticles (AgNPs) from silver nitrate and two separate plant extracts; leaves of *Prosopis Juliflora* and *Mentha Piperita* L. AgNPs were used to remove Pb (II) ions from aqueous solutions through the adsorption process. The synthesized AgNPs were identified by ultraviolet- visible (UV-Vis), Fourier-transform infrared (FTIR), transmission electron microscope (TEM), scanning electron microscope (SEM), and energy-dispersive X-ray spectroscopy (EDS) analysis. The diameters of formation nanoparticles were within the range of 8.06–11.5 and 8.02- 11.5 nm of AgNPs from *Prosopis Juliflora*, and AgNPs from *Mentha Piperita* L. extracts, respectively. During the adsorption process, the influence of adsorbent dose, Pb (II) ion concentration, and contact time in removing were studied at constant temperature and constant pH.

Keywords: Silver nanoparticles (AgNPs), (Pb(II)), *Prosopis Juliflora*, *Mentha Piperita* L., Ggreen synthesis.

Introduction

Heavy metals have toxic properties, and as ions they have a strong accumulation ability in the environment, which leads to global environmental risks. Pb, Hg, and Cd are the heaviest metals toxic and affect human health. On the other hand, water is essential for life, and air pollution treatment is one of the most significant problems that must be solved [1]. Contamination of drinking water with Pb and Cd ions effects on human health and may lead to cancer, convulsions, coma, renal failure, nausea, and subtle effects on metabolism and intelligence [2]. There are technology methods that have high operational costs that have been used lately to treat the contamination of water such as ion exchange [3], nanofiltration [4], and ultra-filtration [5].

However, nanotechnology is an important in all areas of science such as biomedicine, sensors, catalysis, and

energy storage [6]. There are nanotechnology-derived products that may help reduce the toxicity of compounds which is useful in water quality standards [7]. Nanotechnology is developed as a new application used to overcome the problems of pollution and contamination in the environment [8]. Recently, there were many types of research showing that nanoparticles which synthesized from plant extract were beneficial and effective for removing some heavy metal ions. Nanoparticles synthesized from metals and plant extracts are one recent application that is environmentally friendly. Green chemistry is the best approach to the synthesis of nanoparticles because it uses plants and microorganisms which not affect human health and the environment compared to the chemical methods such as reduction, sol-gel method, laser pyrolysis, and precipitation, etc. which are hazardous [9,10]. Furthermore, the metal nanoparticles have more attention due to their significant morphological properties, sizes,

and shapes compared to the semi-conductor nanoparticles, carbon-based nanoparticles, polymeric, and lipid-based nanoparticles, and ceramic nanoparticles [11]. Several studies showed that silver nanoparticles were affected and removed some heavy metals from polluted water and other contaminants. The adsorption method by plant extracts of heavy metal is also economical [12, 13]. Many researchers have focused on the removal of heavy ions by green synthesis of silver nanoparticles from aqueous solutions. Some significant heavy metals were extracted by this eco-friendly adsorption technique involving Cd^{+2} , Pb^{+2} , Ni^{+2} , Fe^{+2} , Mn^{+2} , Hg^{+2} , Cu^{+2} , and Co^{+2} [15-18]. Therefore, the aim of this study was the synthesis of silver nanoparticles (AgNPs) which obtained separately from two sources of plant leaves; *Prosopis Juliflora* and *Mentha Piperita* L.; and evaluate their potential and efficiency to remove Pb (II) ions from aqueous solutions by using adsorption process. Additionally, the influence of an initial nanometal concentration, Pb (II) concentration, and contact time were studied at constant pH and room temperature.

Materials and methods

Preparation of AgNPs

The plant leaves of *Prosopis Juliflora* and *Mentha Piperita* L. were collected from the city of Abyan Governorate, Yemen. The species of the plants were taxonomically identified at the Department of Biology, Aden University, Yemen. The small pieces of leaves were washed with distilled water, air dried under the shade, ground into powder form, and kept at room temperature for use in the synthesis of AgNPs. To prepare aqueous plant extractions, add 40 g of leaf powder to 400 mL distilled water in an Erlenmeyer flask (1000 mL), heat the solution for 25 min to 40-45 °C, then filtrated with a thick layer of cotton, and the filtrate was kept in the fridge for future use. To obtain AgNPs the plant extract (30 mL) was added gradually drop by drop from a burette to 100 mL of silver nitrate solution (0.003M) at room temperature, and then the temperature was raised to 80 °C. After that the solution was neutralized by NaOH (1M) and the brown precipitate was observed. The mixture left 72 hours, then centrifuged for 10 min at 2800 rpm. Filtrated and the formed silver nanoparticles were collected, washed three times with distilled water, and dried in an oven at 60 °C for two hours. The Ag nanoparticles were characterized by taking the samples at different times and analyzed them by UV-Vis spectrophotometer on Perkin Elmer Lambda 365 (200–800 nm). The evolution of nanoparticles ago was performed by FTIR spectra studies by using Perkin Elmer FTIR Spectra Clarus 600 MODEL in the frequency range 4000–450 cm^{-1} in KBr pellets. Scanning electron microscopy (SEM and TEM): Thermo Fisher (USA) Quattro S Field Emission Gun,

Environmental SEM (FEG ESEM), equipped with some detectors including; 1- Scanning Transmission Electron Microscopy (multiple mode detector; Bright Field, Dark FIELD, HADF, custom) (up to 6-samples per run. Flat or tilted), 2- EDX (Elemental Analysis & Elemental Mapping).

Preparation of Pb (II) stock solution

Standard solution of Lead chloride was prepared at 1000 mg/L, then a series different of concentrations (15, 20, 25, 30, 35, and 40 mg/L) were prepared from it.

Removal experiments

To study the effect of the concentration of Pb (II) ions on the adsorption process, 0.1 g of AgNPs was added to 50 mL solution of Pb (II) with different concentrations (15, 20, 25,30,35, and40mg/L) in volumetric flasks at 25 °C and pH 6. The flasks were shaken onto the bath shaker for 40 min. Samples of 1 ml were collected from flasks at the required time (40 min) and centrifuged for 5 min. Then filtrated by using Whatman no. 1. The concentration of Pb(II) was determined by Atomic Absorption Spectrophotometer (PerkinElmer, PinAAcle 900F). The percentage of the removal of Pb (II) from polluted water was estimated using the formula:

$$\% \text{ removal} = \frac{C_0 - C_e}{C_0} \times 100 \quad (1)$$

whereas, C_e : is the final concentration of Pb(II) from mg/L and C_0 : the initial concentration of Pb(II) mg/L

To obtain the effect of the dose of AgNPs on the on Pb(II) removal, all previous steps were repeated with 20 mg/L of Pb(II) concentration, once by different amounts of AgNPs (0.025, 0.0375, 0.05, 0.075, 0.10 g).

After that all previous steps repeat again to study the effect of contact time on the adsorption process at different times (5, 10, 15, 20, 40, 60, 80 min) at constant AgNPs (0.1 g), and constant Pb(II) concentration (20 mg/L).

Results and Discussion

Characterization of AgNPs

The formation of AgNPs was observed by the color changing from bright yellow to dark of the solution during the addition of plant extract to the silver nitrate solution within an hour. The appearance of maximum peak absorption in the UV-Vis spectra around 420 nm indicated the characteristic position of the silver nanoparticles, as showed in figures 1 and 2 [19-21].

FTIR spectra, figures 3 and 4, showed the broad band around 3300 cm^{-1} corresponding to stretching vibrations of (O-H) functional group and another band at 1700 cm^{-1} for (C=O) functional group. However, the two bands may be indicated the characteristic groups of

polysaccharides, fatty acids esters, and lipids. The FTIR spectra also contained absorptions between 3000-2700 cm^{-1} are due to the vibration frequencies of (C-H) of the sp^3 carbon hybridization. While the vibrational peaks between 1600 - 1400 cm^{-1} , and at around 1200 cm^{-1} , were corresponding to the stretching vibrations of (C=C), aliphatic amines (C-N), and (C-O-C) [22-24].

Figures 5-8 show the scanning electron micrograph of the growth silver nanoparticles (TEM and SEM). The formed AgNPs are within the range of nanoparticles, and they have spherical shapes. Their diameters are placed at 8.06–11.5 and 8.02- 11.5 nm of AgNPs of *Prosopis Juliflora*, and AgNPs of *Mentha Piperita L.* extracts, respectively. On the other hand, figures 9 and 10 exhibited the EDS spectra of the AgNPs from the two sources of plant extracts, and both of them contained a peak around 3 keV that characteristic of the surface plasma resonance of silver nanoparticles [25].

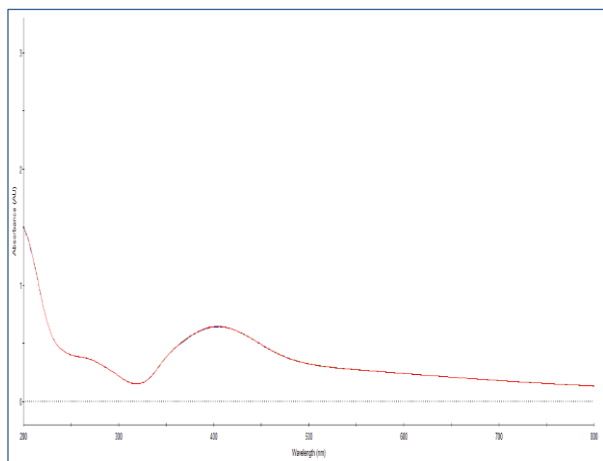


Fig. 1: UV-Vis spectrum of the synthesized AgNPs of *Prosopis Juliflora*

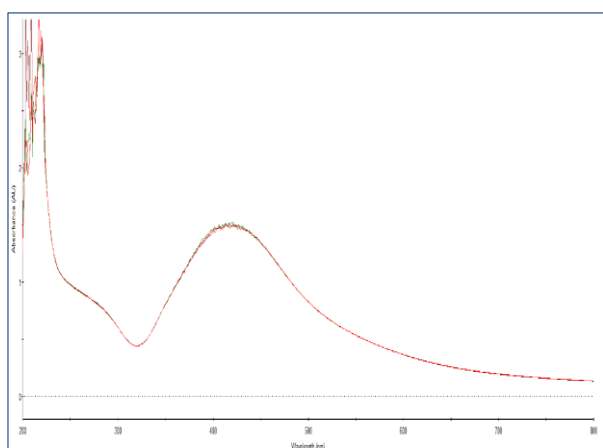


Fig. 2: UV-Vis spectrum of the synthesized AgNPs of *Mentha Piperita L.*

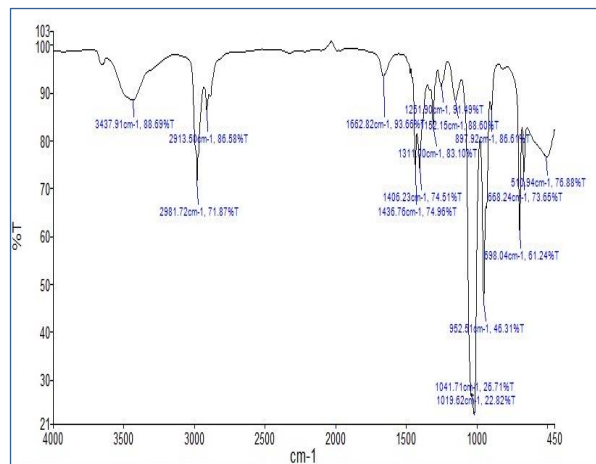


Fig. 3: FTIR spectrum of the synthesized AgNPs of *Prosopis Juliflora*

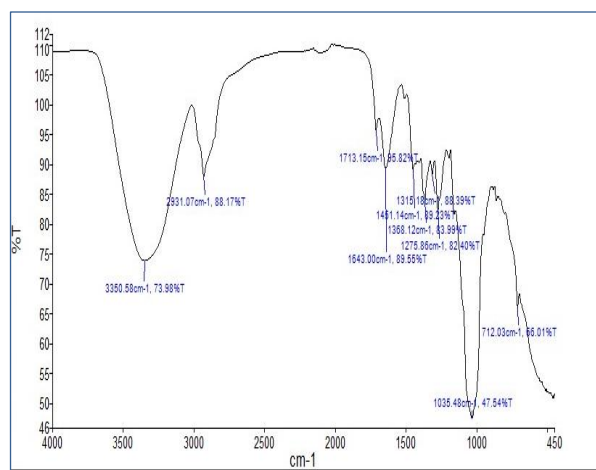


Fig. 4: FTIR spectrum of the synthesized AgNPs of *Mentha Piperita L.*

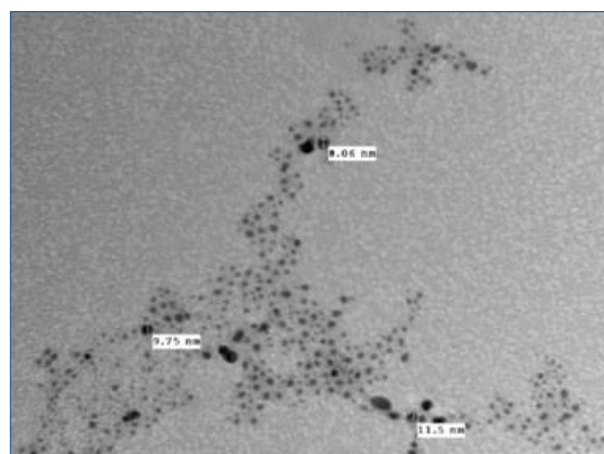


Fig. 5: TEM image of synthesized AgNPs of *Prosopis Juliflora*

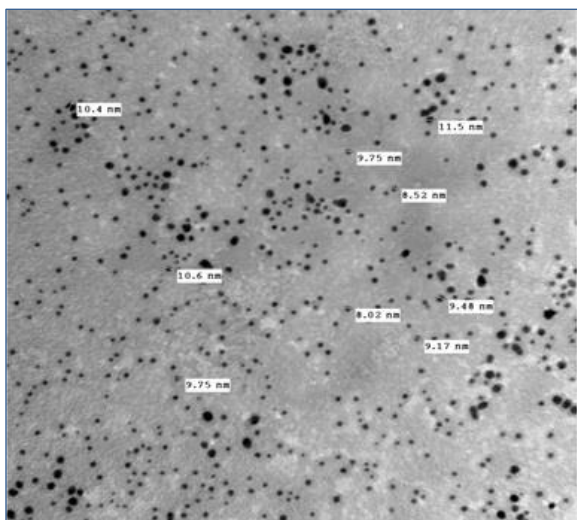


Fig. 6: TEM of image of synthesized AgNPs of *Mentha Piperita L.*

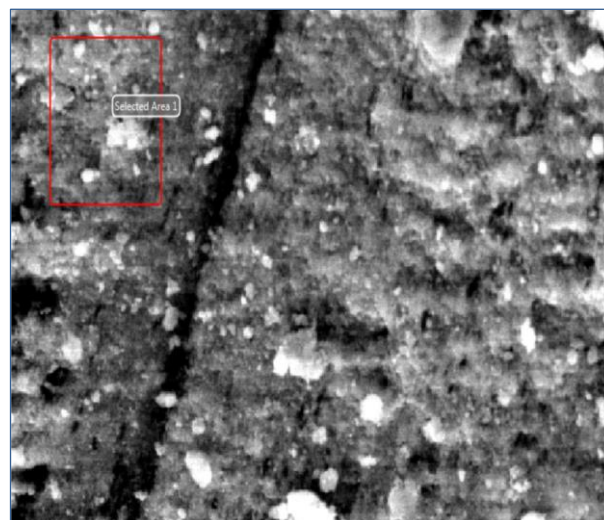


Fig. 9: EDS spectrum of synthesized AgNPs of *Prosopis Juliflora*

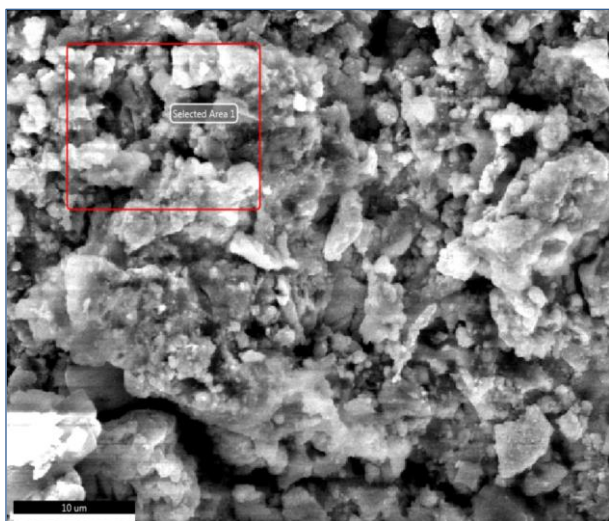


Fig. 7: SEM image of synthesized AgNPs of *Prosopis Juliflora*

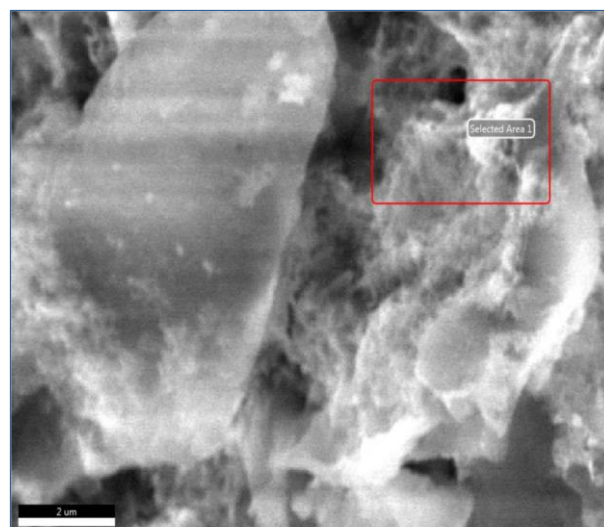


Fig. 10: EDS spectrum of synthesized AgNPs of *Mentha Piperita L.*

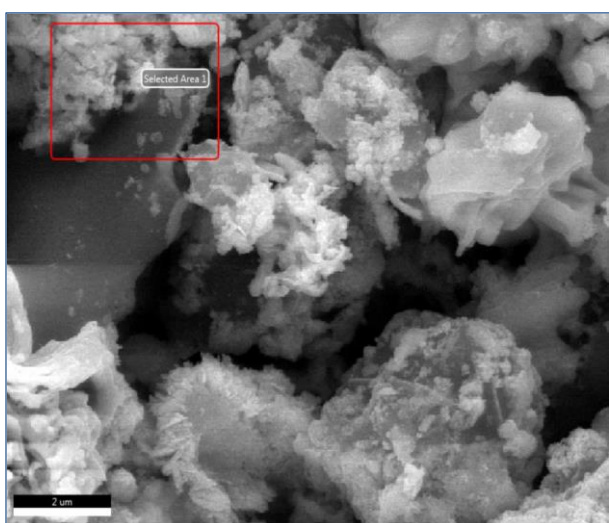


Fig 8: SEM image of synthesized AgNPs of *Mentha Piperita L.*

Influence of adsorbent dose (amount of AgNPs) on removal of Pb (II) on AgNPs

To obtain the relation between the adsorbent does and the percentage removal of Pb(II). The amount of AgNPs varied from 0.025 to 0.1 g and the temperature, pH, contact time, and initial Pb (II) concentration were stated at 25°C, 6, and 40 min, and 20 mg/L, respectively. The obtained results are shown in Fig.11 (a and b). The figures showed that when the amount of AgNPs increased, the percentage removal of Pb (II) increased until it almost reached to stable level. This can be explained by the increasing of the surface area of AgNPs that are needed for the absorption of Pb (II).

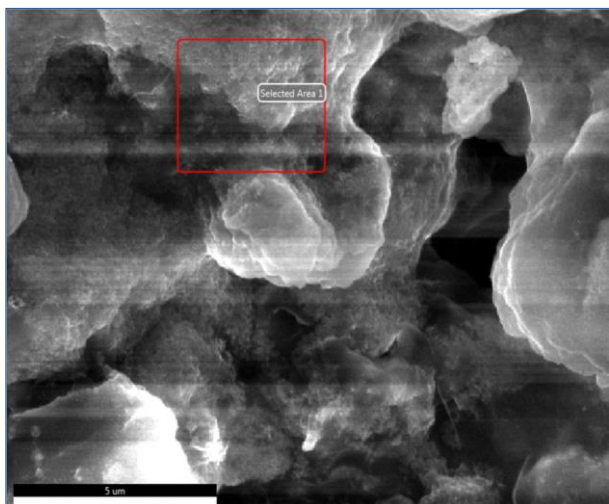


Fig. 11: The effect of dosage of AgNPs on the Pb(II) removal for (a) *P. Juliflora* and (b) *Mentha Piperita* L.

Effect of contact time removal of Pb (II) on AgNPs

Study the effect of interaction time between the AgNPs and initial concentration of Pb (II) on the Pb (II) percentage removal at various times from 5 to 80 min, keeping the temperature at 25 °C, pH 6, adsorbent dose 0.10 g, and initial concentration of Pb (II) solutions 20 mg/L were shown in figure 12 (a and b). The figures exhibited that the Pb(II) removal was increasing by increasing the time till reached 40 min which indicated the highest optimum contact time that must be reached to obtain the equilibrium for Pb (II) on silver nanoparticles. This corresponds to the Pb (II) ions transferred from a solution to the binding sites of AgNPs [26]

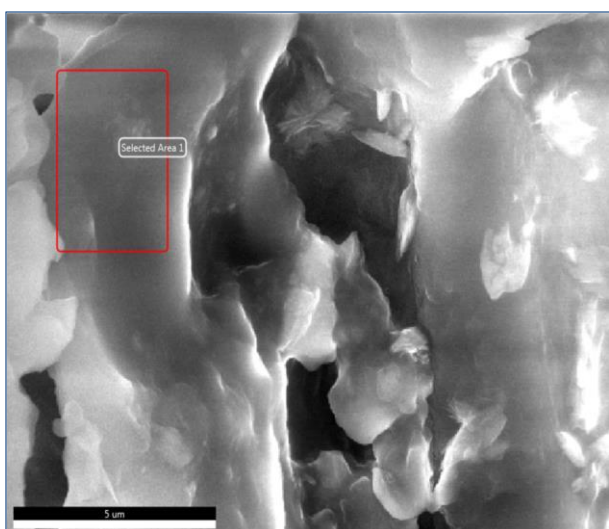


Fig. 12: The effect of contact time on the Pb(II) removal for (a) *P. Juliflora* and (b) *Mentha Piperita* L.

Effect of initial concentration of Pb (II) on its removal on AgNPs

To study the influence of initial concentrations of Pb (II) ions on their removal on the surface of adsorbent

(AgNPs), different Pb (II) concentrations were used (15, 20, 25, 30, 35, and 40 mg/L), with maintained the temperature at 25 °C, pH = 6, adsorbent dose (0.1 g), and contact times 40 min. The maximum removal of Pb (II) ions were 39.49 and 39.40 mg/L in the case of AgNPs from *Prosopis Juliflora* and *Mentha Piperita* L., respectively at 40 mg/L initial metal concentration Fig.13, a and b. It was observed that the adsorption process increased as the concentration of Pb(II) ions increased from 15 to 40 mg/L. However, it is believed that at high concentrations than 40 mg/L, the possibility of adsorption will decrease due to penetration of all active binding sites in the AgNPs surface. [27].

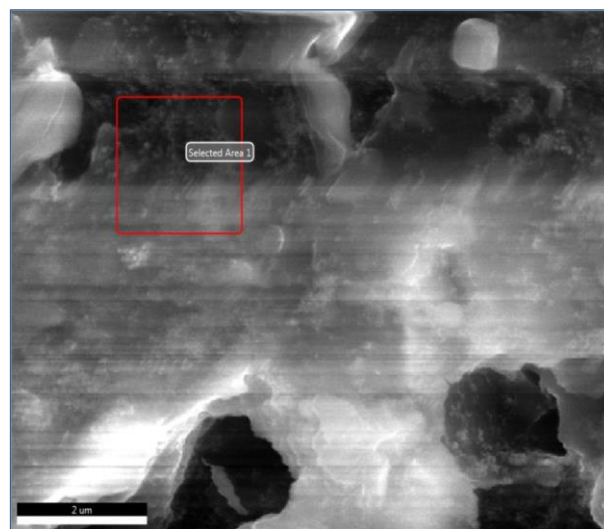


Fig. 13: Effect of adsorbate dose on adsorption of Pb (II) by AgNPs for (a) *P. Juliflora* and (b) *Mentha Piperita* L.

Conclusion

Silver nanoparticles (AgNPs) were synthesized by an eco-friendly approach using silver nitrate and two separate plant aqueous extracts. The plants are characterized as *Prosopis Juliflora* and *Mentha Piperita* L. The AgNPs formation was confirmed by observed the changing of color from bright yellow to dark brown, the maximum peak around 420 nm in the UV-Vis spectra, the peak at 3 keV in the EDS spectra which characterized of the surface plasma resonance of AgNPs. The results of TEM and SEM showed that the synthesized particles were spherical within the nanoscale sizes which were 8.06–11.5 and 8.02- 11.5 nm of AgNPs from *Prosopis Juliflora* and *Mentha Piperita* L., respectively. The removal of Pb (II) ion from the aqueous solutions as a more poisoned and carcinogenic heavy metal ion was evaluated by adsorption process on AgNPs. The results showed that the removal of heavy metal depended on the amount of AgNPs, initial concentration on Pb (II) removal, and interaction time in the case of room temperature and constant pH.

In conclusion, the silver nanoparticles provides an interesting alternative method than traditional tools for removal heavy metals such as Pb(II) from water, and it is a promising tool for use in a number biological science applications

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إزالة أيونات الرصاص باستخدام جسيمات الفضة النانوية المصنعة بالطريقة الخضراء من المستخلصات المائية لأوراق السيسبان والنعناع

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استلم في: 2 يونيو 2024 / قبل في: 14 يونيو 2024 / نشر في 30 يونيو 2024

المُلخَص

معروف أن مركبات المعادن الثقيلة شائعة الاستخدام كمواد متفاعلة في العديد من الصناعات، كما أن أيونات المعادن الثقيلة لها خصائص سمية ولعل من أهمها أيون الرصاص الثنائي التكافؤ (Pb(II)) الذي يتمتع بخواص سمية ومسرطنة كذلك. أن تلوث مياه الشرب للإنسان بأيونات الرصاص قد يؤدي على المدى الطويل إلى الموت، بالإضافة إلى آثاره الضارة على كل من النبات أو الحيوان. من هذا المنطلق كان الهدف من هذا البحث هو استخدام طريقة صديقة للبيئة لاصطناع جسيمات الفضة النانوية (AgNPs) من مصدرين. أحدهما تفاعل نترات الفضة مع مستخلص نبات أوراق السيسبان (*Prosopis Juliflora*)، والمصدر الآخر تفاعل نترات الفضة مع مستخلص أوراق النعناع (*Mentha Piperita L.*). بعد ذلك تم استخدام جسيمات الفضة النانوية المتحصل عليها في إزالة أيونات الرصاص الثنائية التكافؤ من محاليلها المائية من خلال عملية الامتزاز على سطوح جسيمات الفضة النانوية المصنعة. وقد تم اثبات تكوين الجسيمات النانوية من خلال تقنيات الأشعة فوق البنفسجية-المرئية (UV-Vis)، الأشعة تحت الحمراء (FTIR)، المجهر الإلكتروني النافذ (TEM)، المجهر الإلكتروني الماسح (SEM)، مطيافية تشتت الطاقة بالأشعة السينية (EDS). كانت أقطار جسيمات الفضة النانوية في المدى 8.06-11.5 و 8.02-11.5 المتحصل عليها من نباتي السيسبان والنعناع على التوالي. من ناحية تم دراسة تأثير جرعة من المادة النانوية المازة، تركيز أيونات الرصاص في المحلول المائي، وزمن التلامس على عملية الامتزاز وذلك عند ظروف ثابتة من الحموضة (pH) والحرارة طوال الوقت.

الكلمات المفتاحية: جسيمات الفضة النانوية، Pb(II)، السيسبان، النعناع، الطريقة الخضراء.

How to cite this article:

E. A. Bin Selim, R. M. Ismail, S. Ruban, "REMOVAL OF LEAD IONS BY GREEN SYNTHESIZED SILVER NANOPARTICLES USING AQUEOUS EXTRACTS OF *PROSOPIS JULIFLORA* AND *MENTHA PIPERITA L. LEAVES*", *Electron. J. Univ. Aden Basic Appl. Sci.*, vol. 5, no. 2, pp. 217-224, June. 2024. DOI: <https://doi.org/10.47372/ejua-ba.2024.2.355>



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