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Silver nanoparticles coated by Schiff base as an adsorbent of lead from polluted water

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ABSTRACT

Synthesis of silver nanoparticles coated with Schiff base was achieved . Silver nanoparticles are reduced and coated using Schiff base. Schiff base serves as a capping to stop silver oxide from overgrowing. Schiff base produces silver nanoparticles with a novel form and a distinctive appearance. The presence of Schiff base as a reducing and capping agent can give a new shape for prepared silver nanoparticles. The structure of Schiff base was analyzed based on spectral data (^{1}H nuclear magnetic resonance (H^{1}NMR), Mass spectroscopy, and Fourier-transform infrared spectroscopy (FTIR). The formation of silver nanoparticles within a surface plasmon band at 461 nm was observed using UV-Vis Spectroscopy. Silver nanoparticles with a spherical shape and a particle size between 50 and 100 nm are visible in a scanning electron microscope image. For the first time, silver nanoparticles coated with Schiff base were used to adsorb lead from a sample of polluted water. The rate of adsorption was 69%.

1.Introduction

Azomethine is the main compounds in Schiff base which is importance in an organic synthetic field [1]. Schiff bases performance as intermediates in the synthesis of a number of compounds such as industrial and biological compounds through cycloaddition, ring closure and replacement reactions. Schiff base derivatives are beneficial compounds for synthesis of bioactive mediators such as lemon juice [1-15]. because of Schiff bases offer varied applications they have been used in different fields such chelating [16], inhibitors [17, 18], as a catalytic system and liquid crystal metal nanoparticles also is very common due to their varied applications in various fields [23]. Because of high surface area to volume ratio for nanomaterials, their properties will difference because the nano size such as physical, chemical and electrical properties [24]. In current years silver nanoparticles have attracted a lot of attentions due to their, chemical stability, good conductivity, and catalysis [25]. Incredible properties of nanomaterials strongly depend on size and, shape of nanopacticles (NPs), their interactions with stabilizers and surrounding media, and also on their preparation method.Controlling the synthesis of nanocrystals is therefore a major obstacle to understanding their better application features[26] . The shapes of nanoparticles depend on their interaction with stabilizers and capping agent such as surfactants, ligands, polymers or dendrimers [27 ,28] . High reactivity is a significant issue for analysis

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of some materials regardless of the particle size, however such effects might be improved for nanosized substances of materials that are typically less reactive. In some other conditions, environmental effects may lead to surprising nanomaterial properties [29-32] .

In this work, synthesis of silver nanoparticles were achieved using Schiff base as a reducing and capping agent. From our knowledge, this is the first time that capped silver nanoparticles with Schiff base prepared from banzothiozole used for purification of water from polluted heavy metals such lead.

2. Materials and Experiment

The compound of Schiff base was synthesized as can be seen in schematic in figure 1.

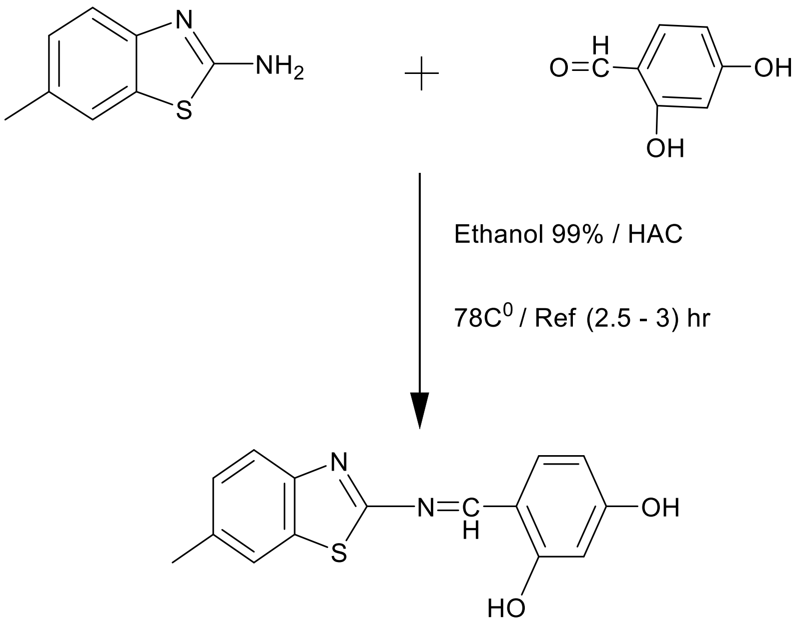

Fig.1 The schematic diagram for the schiff base synthesis

Chemicals and solvents were provided by Sigma-Aldrich. Using plates covered in silica gel 60 ultraviolet (UV) 254, TLC analytical properties were examined, and the resulting compounds were observed using UV light. On an Iraqi Shimadzu FTIR-8300 infrared spectrophotometer, FTIR spectra were captured, and the absorbance ranged from 3600-600 cm^{-1}. ^{1} HNMR spectra were recorded at room temperature in DMSO-d6 as solvent on a Bruker in (ovo AV-400 meter (Iran) with a signal peak of ^{1}H spectra. ^{1} H spectral signal peak at 2.50 ppm. The finished proton of decoupling values (J) is provided. Using a Gallen Kamp melting point device, melting points were measured in capillary tubes.. A Micro Mass LCT operating in Electrospray modetubes was used to make precise mass measurements. UV-Vis spectra were collected using a UV-160v Shimadzu spectrophotometer (400-700 nm). Images of scanning electron microscopy (SEM) were captured using a Zeiss instrument with a 200kv accelerating voltage (Iraq). The silver nanoparticles were subjected to energy dispersive analysis (EDX) (EDX instrument, (Iraq).

2.1.Synthesis of Schiff base

The synthesis of Schiff base 4-(((6- methylbenzo[d]thiazol-2-yl)imino)methyl)benzene-1,3-diol was done by mix 2-4 dihydroxy benzaldehyde (0.164 g, 0.001 mol) in 10 ml ethanol with 2-amino-6-methl benzothiazole (0.138 g, 0.001 mol) along with two drops of glacial acetic acid[[32,33]The resulting mixture was allowed to reflux for three hours. A yellow product was precipitated after the reaction was cooled. Compound of Schiff base was obtained as a yellow powder after being filtered, washed with cold ethanol, recrystallized from ethanol, and dried under vacuum desiccator. TLC silica gel was used to determine the purity of the Schiff base. 70 % yield, m.p. 251-254 ^{o}C. FTIR (KBr, v, cm^{-1}).

2.2 .Synthesis of silver nanoparticles

At the concentration of 0.1 (W/W%) of both silver nitrate and Schiff base were used to prepare silver nanoparticles. The resulting mixture was heated and stirred for 20 minutes at 60 degrees Celsius. The color of the mixture changed from yellow to light brown as a result of the reduction reaction of silver ions. The use of Schiff base as a reducing and stabilizing agent first reduced the metal ions and then stabilized the formed silver nanoparticles[34]

2.3.Adsorption of Lead using silver nanoparticles

For adsorption of lead by synthesized sample the standard solution were prepared in the range (1-20ppm) the concentration of lead 10 ppm was chosen for adsorption procedure. Silver nanoparticles coated by Schiff base was added for lead solution at concentration (10 ppm) [35] . The absorbance before and after adsorption was measured using atomic absorption spectroscopy type Phoenix-986 Biotech (UK)

3.Results and Discussion

3.1.spectrum

1.FTIR

Spectrum: 3024-3435 (OH) para phenols ,3059-3032 C-H aromatic, 2989-2825 (C-H) aliphatic, , 1631(N=CH), 1588(C=N) benzothiazole[36.37]

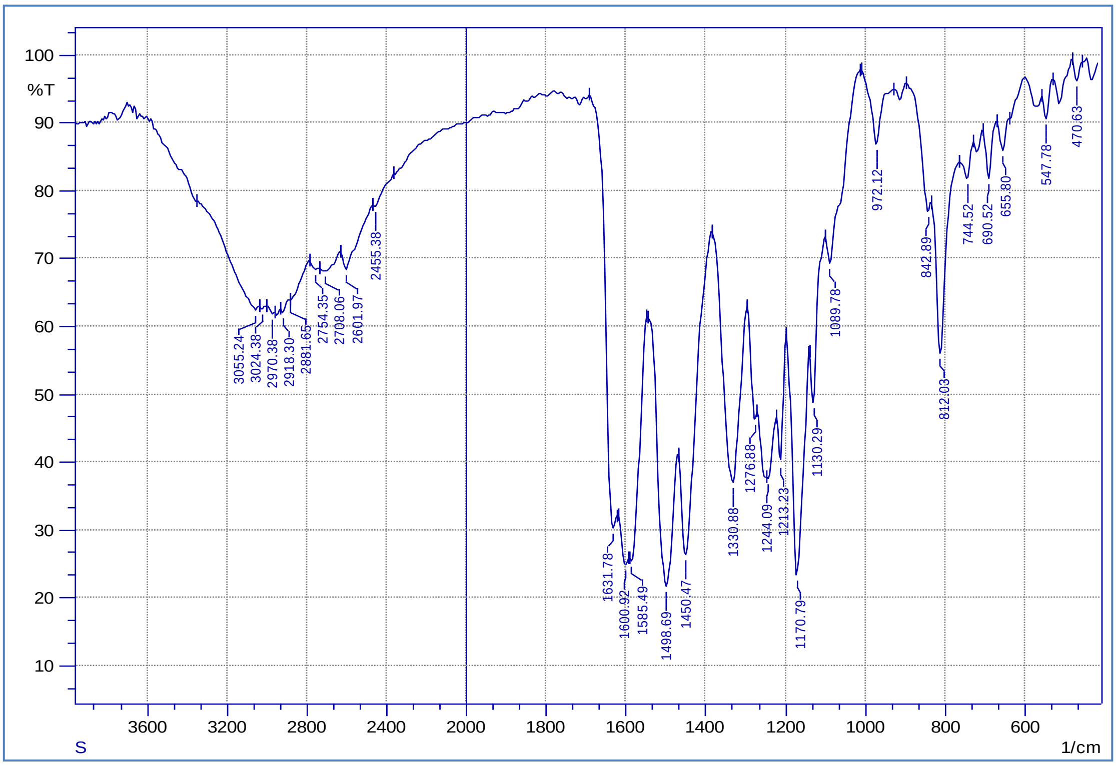

Fig. 2. Fourier-transform infrared spectroscopy spectrum of Schiff base

2. HNMR

1H NMR[36,37,38] in (Dimethyl sulfoxide, DMSO), : S(ppm) 11.99 ppm (s, 1H, OH ortho, 10.69 ppm (s, H, OH para), 9.92 ppm (s, H, CH=N), 6.4-7.9 ppm (m, 6H,H-Ar), 2.4 ppm (s,3H,CH3)

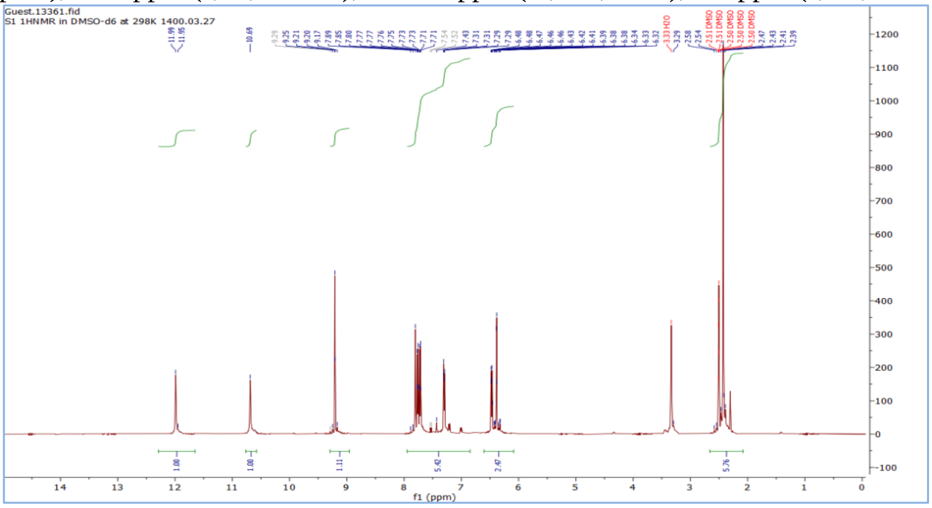

Fig. 3. ^{1} H nuclear magnetic resonance spectrum of Schiff base

3. Mass spectra

C15H12N2O2S Mass (EI) [39] (M+.) calculated: 284.33, found: 284.1

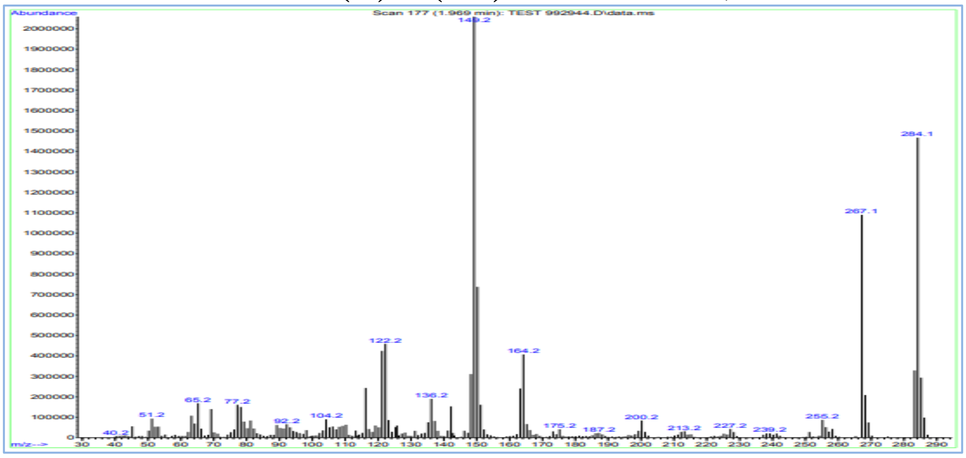

Fig. 4. Mass spectrum of Schiff base

4.Synthesis of silver nanoparticles

Schiff base consist of hydroxyl groups that have a strong capability to bind with silver ions. Hence, once the interaction occurred, silver ions is reduced to Ag^0 and therefore induces the creation of silver nanoparticles. Although the green method is presented in the recent research in case of preparation of metallic nanoparticles, the Schiff base prove the ability to reduce and stabilize nanoparticles eliminates the necessity of using several reagents that acts specifically as reducing and stabilizing agent [1] . Additionally, the Schiff base can be used as a masking agent for nanoparticles, giving them stability in solutions and the ability to the presence of active groups (such as amine) on the surface of the Schiff base. This makes the Schiff base suitable for electrostatic adsorption and interaction with nanoparticle surfaces as well as for adsorbing metal from contaminated water [40]. The schematic diagram of the synthesis of silver nanoparticles covered in Schiff base is shown in Figure 5.

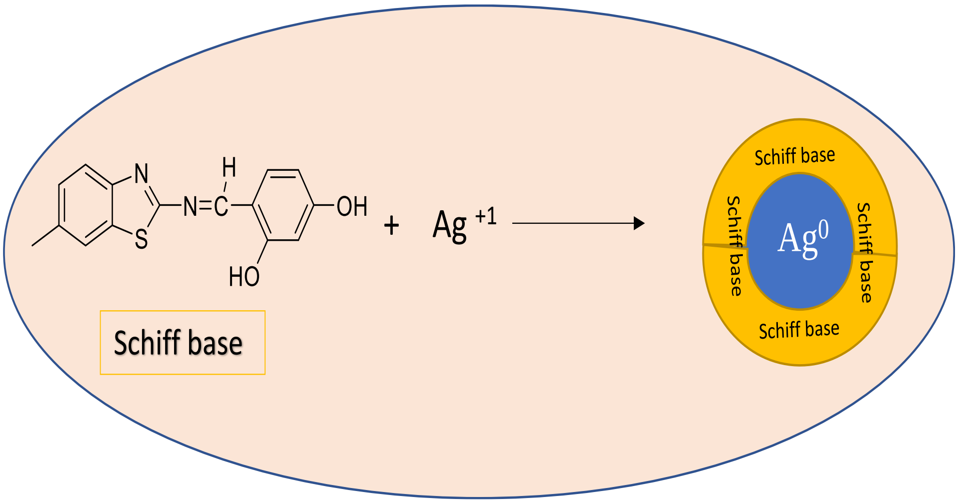

Fig.5 Schematic diagram of synthesis of silver nanoparticles coated by Schiff base.

4.1.UV-Vis spectroscopy of silver nanoparticles

Silver nanoparticle solutions were examined to find plasmon spectra using UV-Vis spectroscopy. The reduction reaction of Ag+ ions to create Ag0 was seen by recording the UV-Vis spectra of silver nanoparticles (1.0 mL) after diluting them with deionized water to (3.0 mL). In silver nanoparticles, a localised surface plasmon band is excited, which causes absorbance peaks to emerge at (400-800 nm). As shown in Fig. 6, the maximum absorbance at (461) nm is attributed to a Surface Plasmon band (SPB) caused by the formation of silver nanoparticles[41,42]. The Plasmon spectra is the phenomenon for metal that indicates the formation of nanomaterials and is expressed on the resonance of metal in the exact wavelength

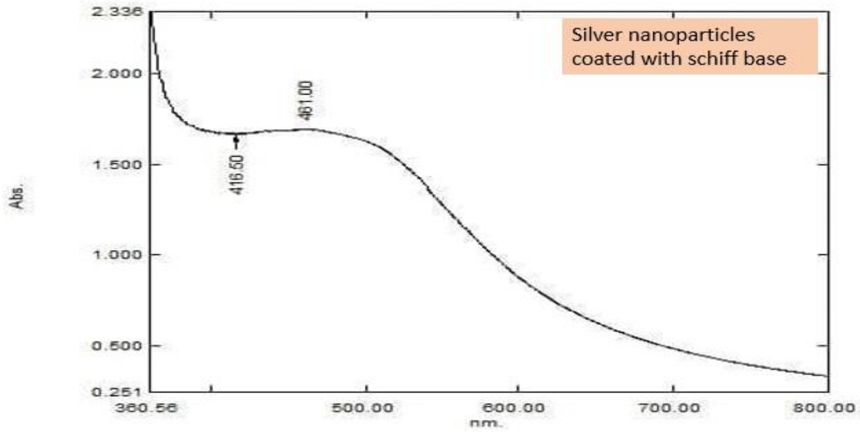

Fig. 6 . The Plasmon spectra of silver nanoparticles.

The Schiff base was measured in order to examine the peaks before the formation of silver nanoparticles. A can be seen from the Figure 7 that many of peaks for the Schiff baseswhich are disappeared and only the peak for plasmon appeared when silver nanoparticles were synthesized.

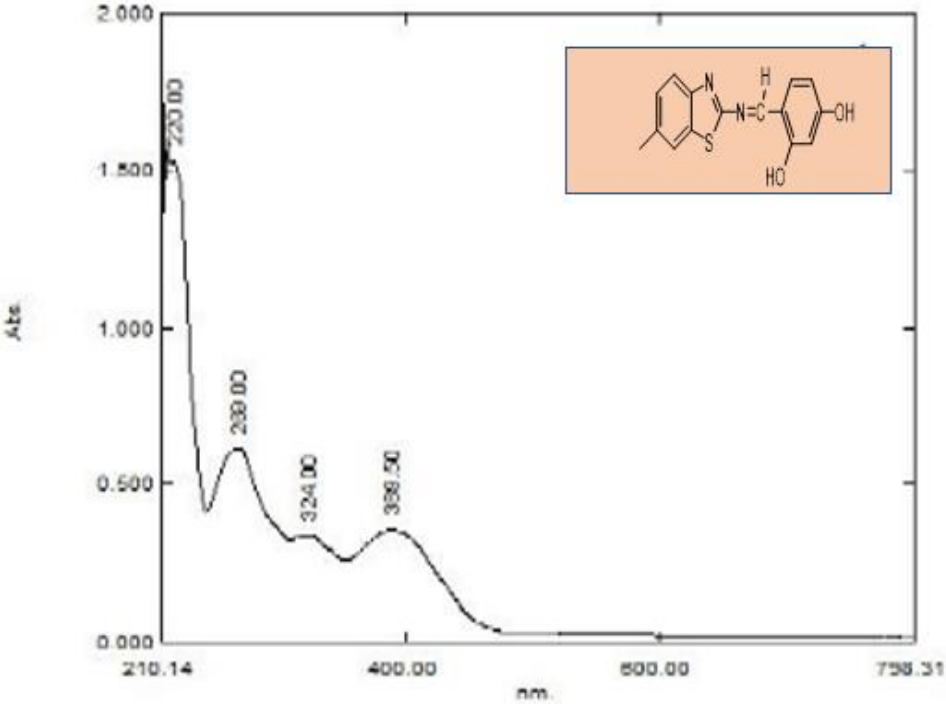

Fig.7 Uv-vissible spectra of Schiff base.

4.2.Scanning Electron Microscopy -EDX

SEM image was used to determine the particle size of silver nanoparticles. Schiff base is an important capping agent in the synthesis of silver nanoparticles because it can reduce and stabilize dispersive nanoparticles. Figure 8 illustrate a clear separation of the silver nanoparticles. These nanoparticles also have a consistent appearance in the solution. The particle size of silver nanoparticles was discovered to be (50-100) nm, and their shapes were spherical[43,44].

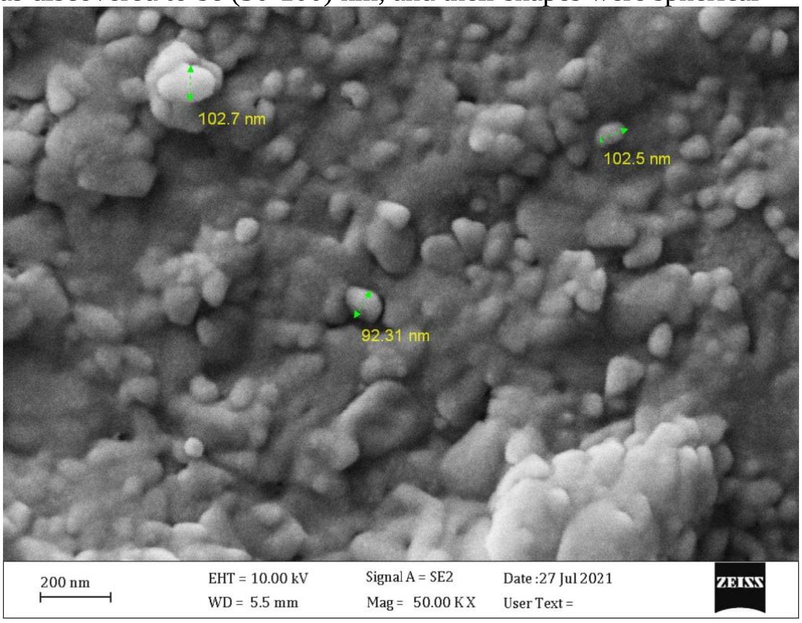

Fig. 8. Scanning electron microscopy image of silver nanoparticles in the presence of Schiff base.

Silver nanoparticles were examined using energy dispersive x-ray spectra. Figure 9 proves the formation of silver nanoparticles. Because of the high percentage of silver (52.9%) compare with the percentage of oxygen 39.5%.

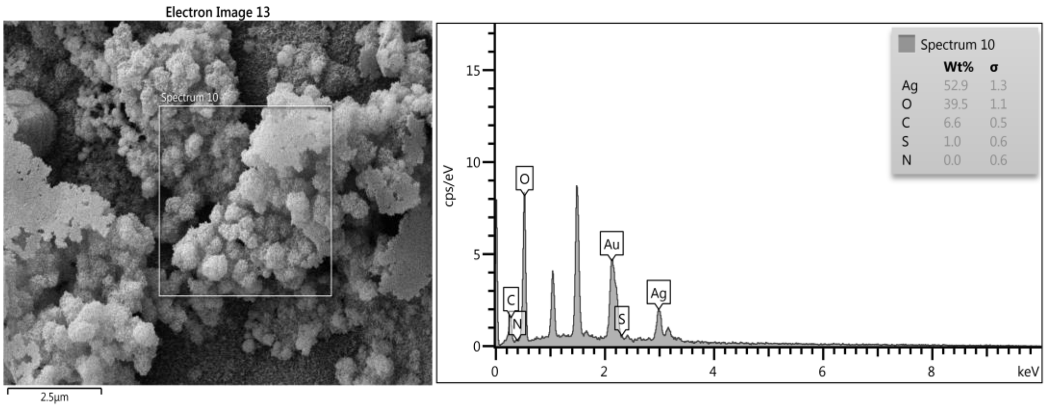

Fig.9. Energy dispersive analysis spectra of silver nanoparticles.

5. Application of synthesized silver nanoparticles in purification of water

The current synthesized silver nanoparticles were used as an adsorbent for purification of polluted water from one of the most popular heavy metals such as lead. Lead concentrations were measured using atomic absorption spectra before and after the adsorption of silver nanoparticles coated with schiff base. From our knowledge this is first time of using silver nanoparticles coated by Schiff base for releasing pollutant from polluted water table 1 and figure10, displays these results.

Table 1: Shows the adsorption % of lead ions on (silver nanoparticles coated by Schiff base) using AAS at optimum conditions (25°C, contact time 40 min, conc. of nano silver 0.1w/w%).

Lead conc.(mg.L^{-1})	**Lead absorbance before using of silver nanoparticles**	**Lead absorbance after using of silver nanoparticles**	**% Adsorption** $= \frac{(A^{\circ} - A_t)}{A_t} * 100$
10	**0.154**	**0.048**	**69**

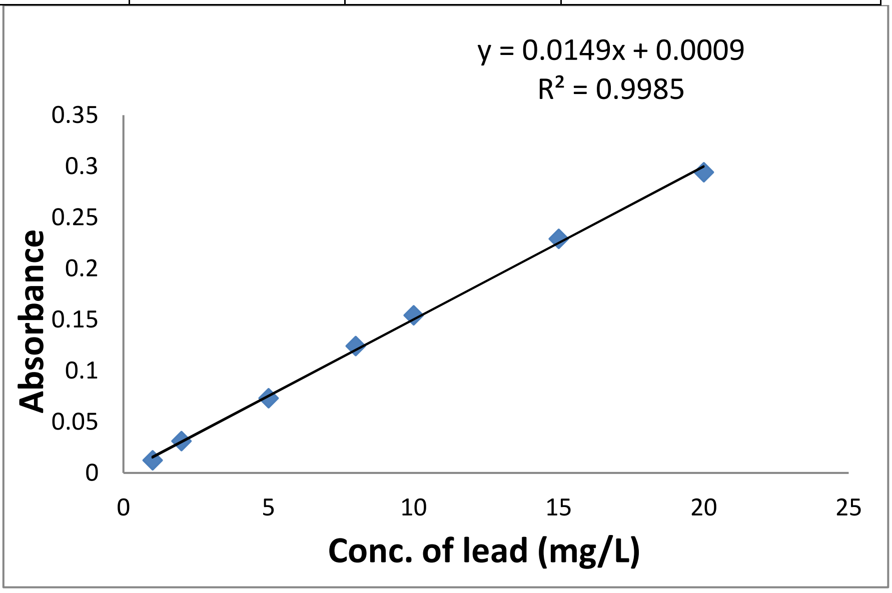

Fig. 10. Standard curve of lead using atomic absorption spectroscopy

The percentage of adsorption of lead was 69% using Schiff base as a capping agent for silver nanoparticles [45]. However, several factors need to be investigated, including the schiff base's structure, which may have an impact on adsorption. There may be more metal adsorption sites on nanoparticles with higher charges due to their capping. Such investigations should be conducted since nanoparticles can be created in a variety of shapes by altering the schiff base.

6. Conclusion

Silver nanoparticles capping by Schiff base can be produced by simple and fast method. The ability of Schiff base to reduce metal eliminates the need for multiple reagents that act specifically as a reducing or masking agent. Furthermore, the nanoparticles formed using Schiff base can be used in a variety of fields. Schiff base surface charge that allows for adsorbed materials such heavy metals. Because of that nanoparticles synthesized from Schiff base can be manufactured in a variety of applications. Moreover, the nanoparticles can be achieved in different shapes. Therefore, research could be conducted to precisely control the size and shape of nanoparticles capping with Schiff base. Furthermore, because nanoparticles are new to water treatment applications, various separation and purification techniques could be developed to increase their activity while decreasing toxicity.

7. Conflicts of interest

The authors declare that there are no conflicts of interest regarding this article.

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جسيمات الفضة النانوية المغطاة بقاعدة شيف كمادة مازة للرصاص من المياه الملوثة

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معلومات البحث

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الملخص

تم تخليق جسيمات الفضة النانوية وتغليفها باستخدام قاعدة شف تعمل قاعدة شف كغطاء لحماية النانوفضة ان وجود قاعدة شف كعامل اختزال وتغطية يمكن ان يعطي شكل جديد لجسيمات الفضة النانوية المحضرة تم تحليل بنية قاعدة شيف كعامل اختزال وتغطية يمكن ان يعطي شكلا جديدا لجسيمات الفضة النانوية المحضرةتم تشخيص الجسيمات المحضرة باستخدام تقنيات متعددة مثل الرنين النووي المغناطيسي وتحليل طيف الكتلة وطيف الاشعة تحت الحمراء والتحليل الطيفي ولوحظ طيف البلازمون للفضة النانوية عند 461نانومتر والتحليل باستخدام. تظهر الجسيمات النانوية الفضية ذات الشكل الكروي وحجم الجسيمات بين (50 - 100) نانومتر في صورة المجهر الإلكتروني الماسح. ولأول مرة ، تم استخدام جسيمات الفضة النانوية المطلية بقاعدة شيف لامتصاص الرصاص من عينة ملوثة الماء وكان معدل الامتزاز 69٪.

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