



Green Synthesis of Silver Nanoparticles Ag-NPs Using Tomato

(*Solanum lycopersicum*) Fruit Extract and Detection ThemSahar M. Mansur¹ and Rana T. Yahya²¹ Department of Biology, College of Science, University of Mosul, Iraq.Sahar.23scpl48@student.uomosul.edu.iq² Department of Medical Physics, College of Science, University of Mosul, Iraq.drranaaltaee@uomosul.edu.iq

Abstract

THE present study was conducted on the biosynthesis of silver nanoparticles from tomato fruits extract which were prepared from an aqueous solution mix of silver nitrate at a concentration of 1.0 and 0.2 mM with tomato fruit extract at specific dilutions. The concentration 2.0 mM at a dilution of 1:2 (10 silver nitrate solution and 20 aqueous tomato extract) gave dark orange appears as an indicator of the reduction of silver present in tomato extract and the formation of silver nanoparticles (AgNPs). Bio-synthesized silver nanoparticles were detected using ultraviolet-visible (UV) spectroscopy, which gave the best result for detection specially at the 2mM of silver nitrate solution and 1:2 dilution by the appearance of the peak of the curve at the wavelength of 263 nm with an absorbance of 1.8963 nm. These nanoparticles were also detected using the FTIR test, which indicates the presence of active groups at all concentrations used. The results showed the spectra of the biosynthetic nanoparticles with absorption bands at 1600 to 3500 cm⁻¹, so the 2.0 mM concentration of silver nitrate solution at 2:1 dilution gave the best result for the active groups band at the H=O bond reached to 3331 cm⁻¹ at spectrum frequency (2500- 3333) cm⁻¹ and the band was strong and very broad because the vibration force of water bond. The results also indicated the shape and size of the prepared Ag-NPs silver nanoparticles by scanning electron microscope (SEM) examinations, which showed a clear similarity in their size and shape with standard silver nanoparticles. Aim of study: The present study aimed to greenly synthesize AgNps using different dilutions of each of an aqueous extract of the tomato fruit and silver nitrate solutions in an easy and economical way. Also characterize the optical, structural and biological properties of nanoparticles AgNPs produced.

Keywords: Ecofriendly, Nanotechnology, Green synthesis, Silver nanoparticles.

Introduction

Nanotechnology is a technology that works to study, understand, and display matter with dimensions ranging between 1 and 100 nanometres which can be used in all different scientific fields, such as physics, chemistry, biology, materials science and engineering [1]. Nanoparticles can show significantly different physical and chemical properties than their larger material counterparts [2]. Advances in the field of nanotechnology largely depend on the ability to synthesize nanoparticles of various material sizes and shapes as well as to capably accumulate them

into complex architecture. The increasing use of NPs makes these structures more important every day [3]. These nanoparticles are synthesized in many ways, and the biological method is considered one of the easy, fast, cheap, and environmentally safe method, does not require energy [4]. A number of scientists have used plant materials in the biosynthesis of silver nanoparticles as a reducing agent such as *Aloe vera*, *Artemisia absinthium* and *Solanum lycopersicum* as a result of eco – friendly and simple processes required for this synthesis [5-6]. Silver nanoparticles have large surface areas and a high conductivity capacity and they can be synthesized using various biological

*Corresponding authors Sahar M. Mansur, E-mail: sahar.23scpl48@student.uomosul.edu.iq, Tel.: +9647512339141

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sources [7-8]. In addition, they can be synthesized both physically and chemically, however this method may have various disadvantages compared to biological methods as the chemical methods lead to the presence of some toxic chemicals adsorbed on the surface that may have adverse effects in medical applications [9-10].

Plants contain organic compounds such as flavonoids, amino and carboxylic acids, Ketones, phenols, and proteins (Fig.1)[11], as these substances play an important role in returning mineral salts and producing nanoparticles. Moreover, the synthesized MNPs are non-toxic, more stable and more uniform in size than their counterparts prepared by the traditional method. Thus, green preparation methods have become a hotspot for research in the field of MNPs synthesis [12]. Plant extracts not only possess reducing properties, but also possess antioxidant properties and act as a reducing agent for the preparation of MNPs while acting as a stabilizer to protect them from oxidation[13]. Also phytochemicals in plant reduce Ag^+ to AgO structure and provide the stability of AgNPs [14] and serve as a key to manufacturing green, which can be represented in three phases such as the reduction phase, the growth phase, and the completion phase [15-16-17]. In the field of using plant extract tomatoes (*Lycopersicon esculentum* Mill.) , the most popular vegetable fruit” contain vitamins, such as A, B and C; Beta carotene and Phytosterols, etc. Tomato juice have been reported to have anti-oxidant properties [18-19-20].

Material and Methods

Preparation of silver nitrate solution (AgNO_3)

Silver nitrate (AgNO_3)(Sigma –Aldrich, St Louis, MO,USA) was used as the precursor metal salt in aqueous solution to a final concentration of 1.0 and 2.0 mM [21], dissolved in a certain volume of distilled water then completing its dissolution, to the final volume at one liter with using a magnetic shaker (Hotplate Stirrer Lab Tech Lms1003, Korea) to complete the dissolution process.

Preparation of tomato extract

Tomato fruits were collected from Mosul local markets and washed several times with tap water , then rinsed with distilled water. After being dried at room temperature the fruits, 1kg of them is squeezed manually well, then filtered with filter paper, this was done by using the filtrate and removing the precipitate and all suspensions present in the extract placed in sterile tubes (Fig .2-A,B), and kept in the refrigerator at a temperature at 2°C to preserve them until use. This aqueous extract filtrate was used to produce the AgNPs.

Preparation of silver nanoparticles

An appropriate volume of silver nitrate solution and aqueous tomato plant extract prepared above was mixed in sterile tubes graduated according to the following dilutions:

A. 1:1 (10 ml AgNO_3 + 10 ml tomato extract)

B. 1: 2 (10 ml AgNO_3 +20 ml tomato extract)

C. 1: 2 (10 ml AgNO_3 + 20 ml tomato extract)

The first and second above dilution mixtures (A and B) were placed in a water bath (WNB10, memmert, Germany) at a 80°C for two hours to observe the change in colour of the solution mixtures refer to the colour results in the reduction of silver nitrate present in the extract to form silver nanoparticles. While the C dilution kept at room temperatures to compare the differences in colour.

Detecting the properties of biosynthetic silver nanoparticles

Many techniques Have been used for detecting and characterizing of the AgNPs biosynthetic including the spectrophotometer, (UV-Vis, Analytic Jena, Germany) was used to detect and diagnose found in Central laboratory, Collage of Science, University of Mosul which used to detect and diagnose silver nanoparticles prepared in a biological way and determine the maximum absorbance peaks, by placed the prepared nanoparticles solution in a quartz cell, then the absorbance was measured at the wavelength 350-550 nm. the results were recorded for each sample measured.

Detection of active aggregates of biosynthesized nanoparticles using Infrared Spectroscopy Technology (FTIR)

FTIR(Fourier transform infrared spectroscopy) technology is one of the important techniques for detecting the active groups present on the surface of the plant extract, from which we can infer the manufacture of nanocomposites. These active receptors are among the main factors for the biosynthesis of nanoparticle [22]. This examination was conducted by measured the prepared samples in the spectral range 450 – 40000 nm using an FTIR device (BRUKER , American) located in the Central laboratory, College of Science , University of Mosul.

Preparation of artificial AgNPs

Dissolve 1mM of artificial AgNPs (Sigma Aldrich –UK,<100 nm) in 10 ml of distilled water and add some drops of DiMethylsulfinylmethane Methyl sulfoxide (DMSO) to enhance their dissolution , put the solution on the magnetic shaker (Hotplate Stirrer Lab Tech Lms1003, Korea) to complete the dissolution process then add the distilled water up to 100 ml to prepare the solution for the examination by Scanning Electron Microscope(SEM).

Detecting the shape and size of biosynthesized nanoparticles using a Scanning Electron Microscope (SEM):

The examination was conducted for a sample of biosynthesized silver nanoparticles using a scanning electron microscope at the Casir Aladalah Company , (CAC Center) for Laboratory Tests / Baghdad, by placing 1ml of the solution on a glass slide, Thin films of the sample was prepared on a carbon coated grid by just dropping a very small amount of the sample (artificial and biosynthetic AgNPs) on the grid, extra solution was removed using a blotting paper and then the film on the SEM grid were allowed to dry by putting it under a mercury lamp for 5 min. Then samples were examined at several magnification levels to reveal the properties of these manufactured nanoparticles.

Results and Discussion

After 2 hours of reaction between silver nitrate AgNO₃ solution and the aqueous tomato extract, there was a change in colour, which is considered as indicative of the formation of AgNPs from an reduction reaction between aqueous extract of tomato plants and AgNO₃ solution at different dilutions with formation different colour according the dilution used (Fig.3)

In all reaction tubes, a yellow or orange colour colloidal suspension was observed after two hour from the reaction at 80C° in heating water bath according the dilution used(Fig.4).Which confirms that there has been an interaction between the active nanocompounds present in the plant extract with silver nitrate to cause the reduction process to occur and form Ag-NPs [23]. Between the samples tubes the results showed that the B sample (1:2 dilut) (Fig.3-B) gave the best colour indication (dark orange), after 2 hours of heating on the water bath. This means there are a lot of the active aggregates in this sample and the occurrence of the biosynthesis of activated silver nanoparticles, as silver nitrate reduced the silver present in the tomato extract which due to the increasing the temperature led to increase in the interaction between the free silver and the silver present in the active groups of the tomato extract, so a change in colour occurred (Fig.4) [24].

While the dilution sample put in the room temperature don't show any change in colour after two hours of reaction due to of no reduction reaction in this temperature, this is due to the non-availability of the suitable temperature for the reaction(Fig.5-A,B)[24].

The other dilutions of the other solution concentration (1mM AgNO₃) also gave the change in colour also showed gradual changes in colour according to the dilution used after the reaction happened from yellow to pink or orange (Fig. 6). The results of the study revealed the change in colour of

the dilution 1:1 (10 ml AgNO₃ + 10 ml tomato extract) to pink colour (Fig.6-A,C) and change from colourless to the pink colour, also at the same concentration of AgNO₃ the dilution (1: 2 ,10 ml AgNO₃ solution + 20 ml tomato extract), the increasing of the available temperature led to the reduction reaction result from increases the interaction between the free silver and the silver present in the active groups of the tomato extract[25], so a change in colour occurred from colourless to orange (Fig.6-B, D).

Detection the optical properties and structure of silver nanoparticles

The absorbance values of biosynthesis AgNPs solution were measured in the visible region of the electromagnetic spectrum(200-800 nm) using UV-Visible spectroscopy. Spectroscopy visible-ultraviolet which are used to indicate the optical properties of a sample so when light is passed through the sample, the amount of light absorbed by the sample is measured. Results show the differences in the absorbance of different dilutions which have been used (Fig.7) , the best result was given by the concentration 2mM of silver nitrate at 1:2 ratio. The dark orange discoloration is a characteristic data showing the formation of AgNPs due to vibrations on the plasma surface and the maximum peak value at 263 nm with absorbance 1.8963 and the absorption data also support this fact (Fig.7-A).The colour change as a result of mixing silver nitrate solution with tomato extract is due to the plasmon resonance peak, as it was examined by UV-vis at the wavelength 200-800 nm, the results agreed with the findings of Jagtap *et al.* , 2021 [26], who confirmed that the absorption peak of silver nanoparticles that were produced using tomato plant extract falls at a wavelength between 260 - 800 nanometre. Also the wavelengths result from the interaction of ultraviolet radiation with the metal present in the solution, so the electron moves from the low-energy state to the higher-energy state, which produces surface plasmon resonance, which gives an idea of the shape and size of the metal oxide nanoparticles by giving a specific wavelength [26].

The oval shape with its tip up and its base down is evidence of the nanomaterial, which begins to affect when exposed to visible ultraviolet radiation. The visible bands observed in the nanocomposites of the main compounds (tomato extract + silver nitrate)(Fig .7-B) clearly indicate the occurrence of an interaction between the two components form a stable nanocomposite [27].

Examination the active groups in the AgNPs solution by Fourier-Transform Infrared Spectroscopy (FT-IR)

FTIR measurement were carried out to identify the possible functional groups present in the biomolecules found in tomato extract and in all the reaction tubes also their role in the synthesis of

AgNPs as the role of molecules as coating agents for the biosynthesis of nanoparticles as phenol, alcohol, carboxylic acid, alkaloids and terpenoids which are responsible for reducing and stabilizing nanoparticles [28].

The results showed the spectra of the biosynthetic nanoparticles with absorption bands at 3331, 2109, 2116, 2111, 2128, 2113, 2112, 1636, 1635 cm^{-1} (Fig.8), so the band at 3331 nm is stretching band and it is attributed to the vibration of the elastic bond produced by water or alcohol, this package appears (OH) at 2500- 3333 cm^{-1} while the bands from (2109- 2112) are stretching low intensity bands and are attributed to the bound $\text{C}\equiv\text{C}$ tri-bonds in nitriles groups, these bands appear at a range between (2140- 2100) cm^{-1} as for the packages (1635- 1636) cm^{-1} the bands are attributed to compounds that contain the bond $\text{C}=\text{C}$ they are aband of medium intensity and appear at a range between(1626- 1662) cm^{-1} , this result agrees with [29] (Table 1).

Characterization the biosynthetic AgNPs by Scanning Electron Microscope (SEM) analysis

Scanning Electron Microscopy is the important techniques used to examine the surface appearance of nanomaterials and to photograph details, very small topography on the surface and is applied in the field of chemistry, biology and physics [30]. The results of microscopic examinations(SEM) indicated the shape of the two types of AgNPs : green synthetic AgNPs from the tomato extract and the artificial type (Fig.9-A,B) which gave the identical shape and size of the

two types .These results proved the success of the biosynthetic method from plant extract which represent the safer and faster methods to manufacture the nanoparticles by using the extracts of plant leaves, seeds, and others parts in preparing silver nanoparticles [31].

Conclusion

One of the major tests of current nanotechnology is to develop consistent experimental protocols for the synthesis of nanoparticles for their specialized properties. Compared to other green sources, plants are developing as advantageous because the presence of broad and variable bio-molecules in plants can act as capping and reducing agents and thus increases the rate of reduction and stabilization of nanoparticles. Since plants differentiate in their concentrations and combinations of organic reducing agents, the source of the plant extract is known to influence the characteristics and morphology of the green synthesized nanoparticles.

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Declaration of Conflict of Interest

There are no Conflict of Interest.

TABLE 1. Characteristic Infrared spectroscopy absorption of functional groups

Type of bond	Wave number (cm^{-1})	Intensity
O-H (carboxylic acid)	2500- 3300	Strong, very broad
$\text{C}\equiv\text{C}$	2140- 2100	Medium to weak
$\text{C}=\text{C}$	1662- 1626	Medium



Fig.1. Mechanism of AgNP Phytosynthesis by using concepts of metal reduction, AgNP phytosynthesis and reduction by reductases (20).

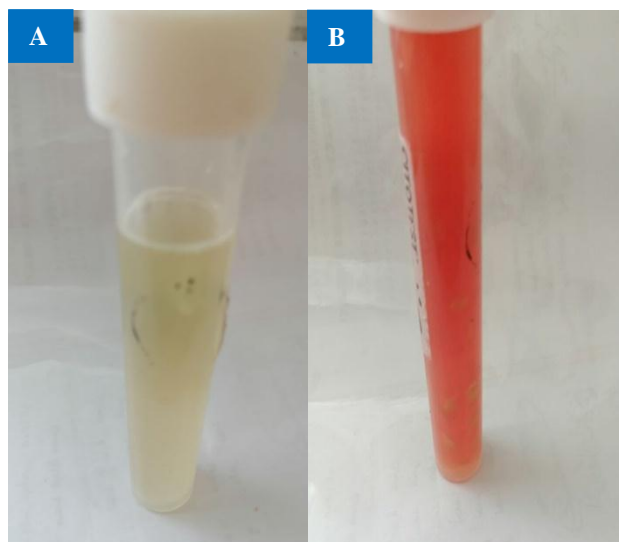


Fig. 2. A. tomato plant extract, B. Filtrate tomato extract

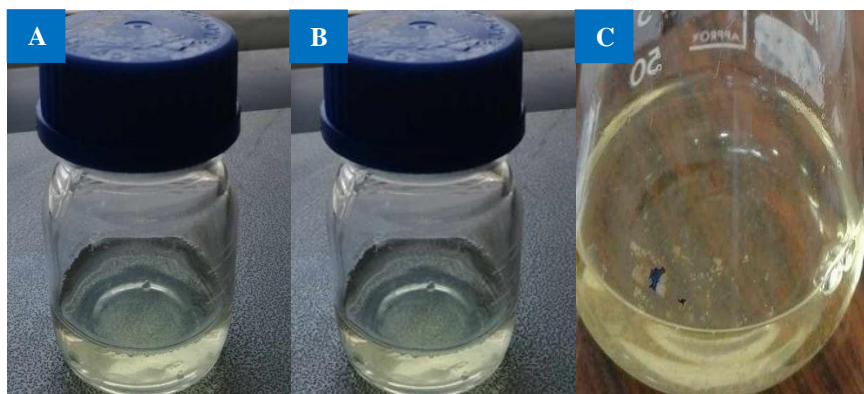


Fig. 3. Reaction tubes before the reaction containing an aqueous tomato extract and AgNO₃ solutions at different dilutions:

A. 1:1 (10 ml AgNO₃ + 10 ml tomato extract)

B. 1: 2 (10 ml AgNO₃ + 20 ml tomato extract)

C. 1: 2 (10 ml AgNO₃ + 20 ml tomato extract)



Fig. 4. Reaction tubes after 2 hrs. of reaction containing aqueous tomato extract and AgNO₃ solutions at different dilutions :

A. 1:1 (10 ml AgNO₃ + 10 ml tomato extract)

B. 1: 2 (10 ml AgNO₃ +20 ml tomato extract)

C. 1: 2 (10 ml AgNO₃ +20 ml tomato extract)

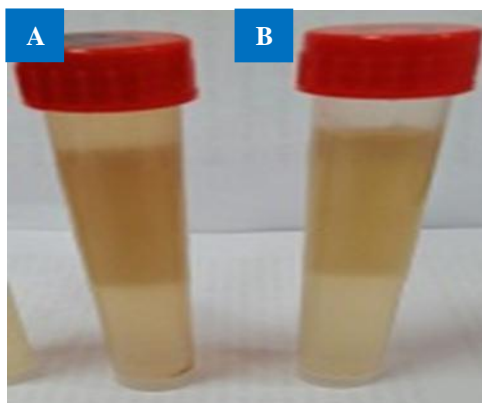


Fig. 5. Mixture of silver nitrate solution with aqueous tomato extract in all dilution put in room temperature before reaction (A) and after reaction (B).



Fig. 6. Mixture of aqueous tomato extract and silver nitrate (1mM) A,C (1:1, 10 ml AgNO_3 + 10 ml tomato extract) before and after reaction and B,D (1: 2, 10 ml AgNO_3 + 20 ml tomato extract) before and after reaction.

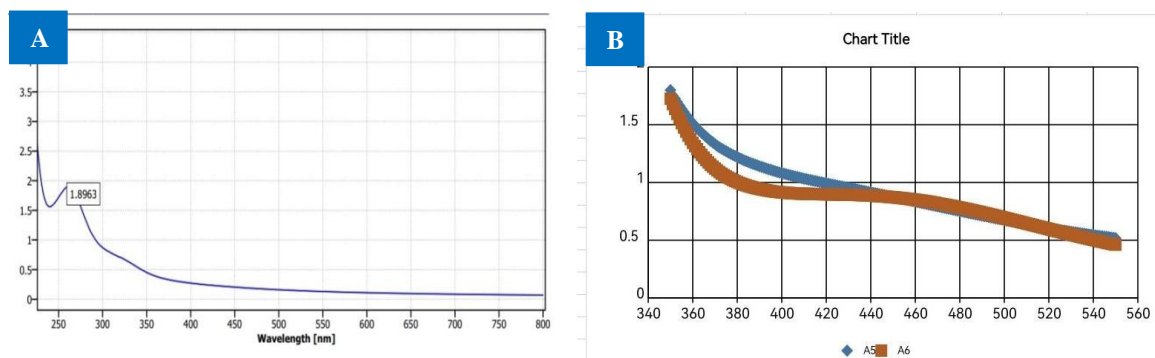


Fig. 7. Visible absorbance curve of AgNPs biosynthesized in 2 mM of AgNO_3 at dilution in wave length ranging from 200 nm to 800 nm (A) and controls (B) of :

- B1: aqueous tomato extract.
- B2: aqueous tomato extract with AgNO_3 solution.

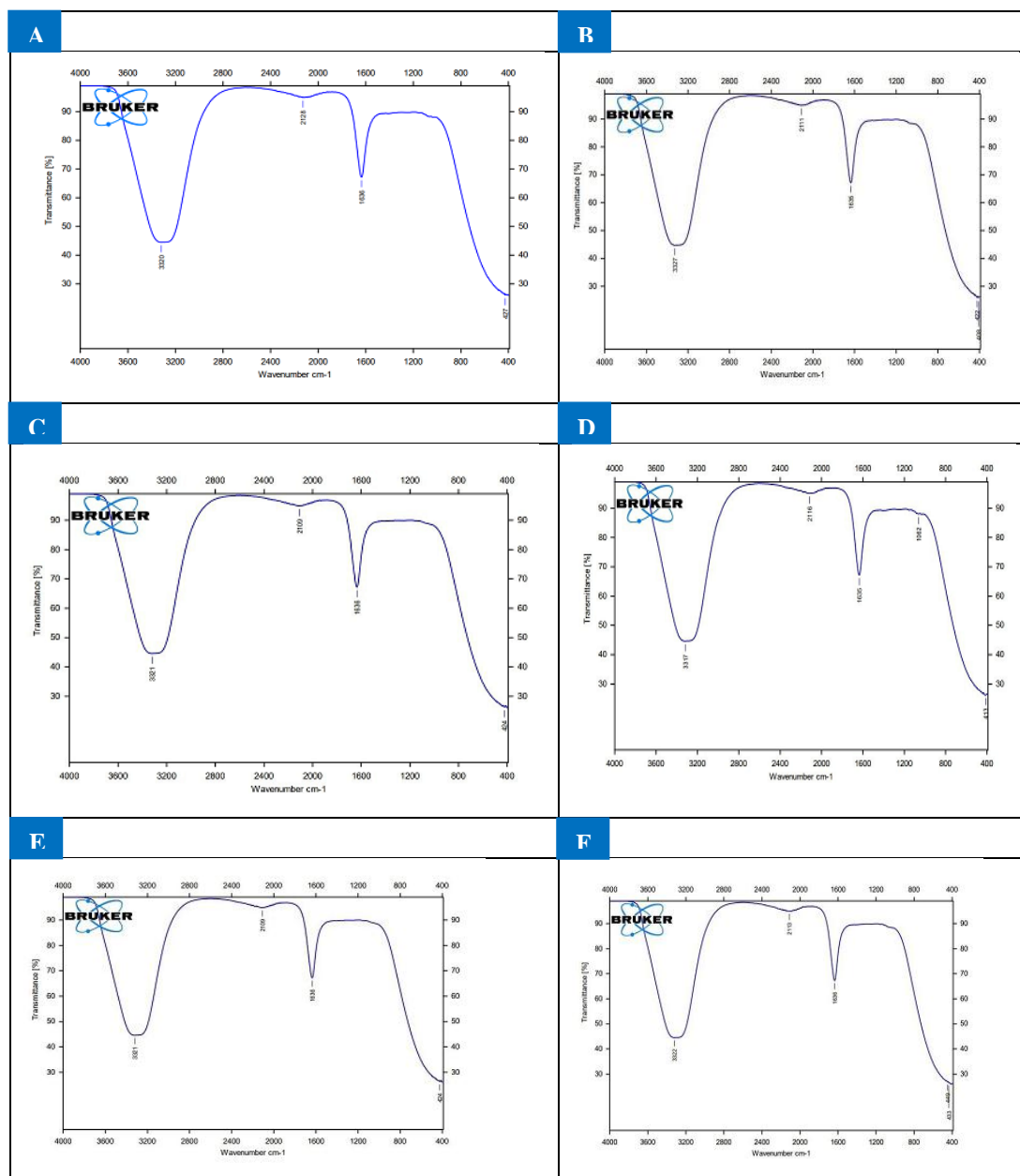


Fig. 8. FTIR spectra for the samples tested : A. Aqueous tomato extract, B. Mixture of silver nitrate solution at 2.0 mM with tomato extract at 1:1 dilution, C. Mixture of silver nitrate at 2.0 mM solution with tomato extract at 1:2 dilution, D. Mixture of silver nitrate at 2.0 mM with tomato extract at 1:3 dilution, E. Mixture of silver nitrate solution at 2.0 mM with tomato extract at 1:4, F. Mixture of silver nitrate solution at 2.0 mM with tomato extract at 1:5.

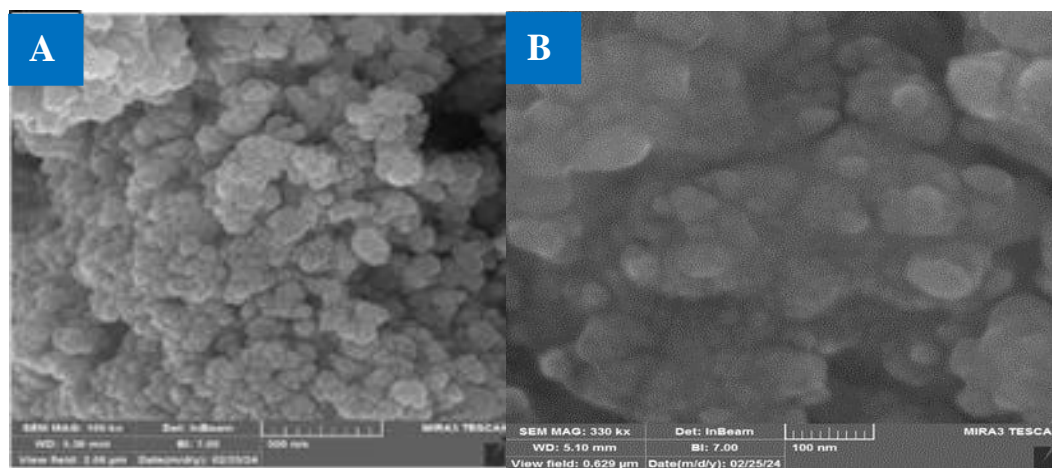


Fig. 9. Scanning Electron Microscope (SEM) analysis of artificial AgNPs (A) and biosynthetic AgNPs (B).

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التصنيع الأخضر لدقائق الفضة النانوية باستخدام مستخلص ثمار الطماطم *Solanum lycopersicum* والكشف عنها

سحر محمد منصور¹ و رنا طارق يحيى²

¹ قسم علوم الحياة , كلية العلوم , جامعة الموصل , العراق.

² قسم الفيزياء الطبية , كلية العلوم , جامعة الموصل , العراق.

الملخص

اعتمدت الدراسة الحالية التصنيع الحيوي لدقائق الفضة النانوية من مستخلص ثمار الطماطم. بسبب التكلفة المنخفضة لهذه الطريقة وتوافرها الحيوي الجيد. تم تحضير جزيئات الفضة النانوية من مزج المحلول المائي لنترات الفضة بتركيز 1.0 و 0.2 ملغم مايكرون مع مستخلص ثمار الطماطم و بتخافيف محددة لكل منهما ، وأعطى التركيز 2.0 ملي مايكرون عند التخفيف 1:2 (10 محلول نترات الفضة و 20 المستخلص المائي لثمار الطماطم) أوضح دلالة لونية بظهور اللون البرتقالي الداكن كمؤشر على اختزال الفضة الموجودة في مستخلص الطماطم وتكوين جزيئات الفضة النانوية AgNPs ولم تظهر العينة المختبرة عند درجة حرارة الغرفة أي دلالة لونية لعدم حدوث تفاعل الاختزال وعدم تكوين الدقائق النانوية.

وتم الكشف عن دقائق الفضة النانوية المصنعة حيويًا باستخدام التحليل الطيفي المرئي فوق البنفسجي (UV) والذي أعطى أفضل نتيجة للكشف عنها وبالأخص عند التركيز 2 ملي مايكرون من محلول نترات الفضة و بالتخفيف 1:2 من خلال ظهور قمة المنحني عند الطول الموجي 263 نانومتر بامتصاصية 1.8963، ان التغير في رنين المحلول المحضر من الأصفر إلى البرتقالي المحمر في التخفيفات المحضرة المختلفة يرجع إلى ذروة رنين البلازمون التي تقع بين 250-280 نانومتر والموجودة على سطح المادة النانوية. كما تم الكشف عن الدقائق النانوية المحضرة باستخدام اختبار FTIR الذي يشير إلى وجود المجاميع الفعالة في كل التراكيز المستخدمة. إذ أظهرت النتائج ان طيف الدقائق النانوية المصنعة حيويًا مع خطوط امتصاص تقع بين 1600-3000 سم⁻¹ وأعطى التركيز 2.0 ملي مايكرون من نترات الفضة عند التخفيف 1:2 أفضل نتيجة للمجاميع الفعالة كانت عند الرابطة H=O التي بلغت 3331 سم⁻¹ عند طيف تردد 3300 - 2500 سم⁻¹ كانت هذه الحزمة قوية وواسعة جدًا وذلك بسبب قوة الاهتزاز لرابطة الماء. أشارت النتائج أيضًا إلى شكل وحجم الدقائق النانوية للفضة المحضرة AgNPs بواسطة فحوصات المجهر الإلكتروني الماسح (SEM) التي أظهرت تطابقًا واضحًا في حجمها وشكلها مع دقائق الفضة النانوية القياسية.

الكلمات الدالة: صديقة للبيئة ، تكنولوجيا النانو ، التصنيع الأخضر ، دقائق الفضة النانوية.