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Green Synthesis of Silver Nanoparticles Using Figs (*Ficus carica L.*) Leaves Extract Syrian

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The green nanoscale approach aims to advance development of clean technologies using nanotechnology, to minimize potential environmental and human health risks associated with the manufacture and use of nanotechnology products, and to encourage replacement of existing products with new nanoproducts, which are more environmentally friendly throughout their lifecycle. In addition, to being inexpensive, the easy implementation process and the advantages of synthesis without toxic chemicals are the main reasons of interest. Nanoparticles (NPs) are synthesized in many ways, including the biological method, which is an easy, fast, inexpensive and environmentally safe method and depends on using microorganisms or plant extracts. In this study, silver nanoparticles (Ag NPs) are successfully synthesized using figs (*Ficus carica*) leaf extract. The resulting nanoparticles are examined using scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDX). The spectrum (SEM–EDX) analysis shows that the Ag NPs are of cubical shapes and their elemental composition contains mostly silver. The size of the nanoparticles produced is approximately in the range of 16.9–39.5 nm.

Зелений наномасштабний підхід спрямований на просування розвитку чистих технологій з використанням нанотехнологій, мінімізацію потенційних ризиків для навколишнього середовища та здоров'я людини, пов'язаних з виробництвом і використанням нанотехнологічних продуктів, а також на заохочення заміни наявних продуктів новими нанопродуктами, які є більш екологічними протягом усього їхнього життєвого циклу. Крім того, що він недорогий, простий процес реалізації та переваги синтези без токсичних хемікатів є основними причинами інтересу. Наночастинки (НЧ) синтезуються різними способами, включаючи біологічний метод, який є простим, швидким, недорогим і екологічно безпечним методом і залежить від використання мікроорганізмів або рослинних екстрактів. У цьому дослідженні наночастинки срібла

(Ag-НЧ) успішно синтезуються з використанням екстракту листя інжиру (*Ficus carica*). Одержані наночастинки досліджують за допомогою сканувальної електронної мікроскопії (СЕМ) та енергодисперсійної рентґенівської спектроскопії (ЕДРС). Спектральна аналіза (СЕМ–ЕДРС) показує, що Ag-НЧ мають кубічну форму, а їхній елементний склад містить переважно срібло. Розмір одержаних наночастинок приблизно знаходиться в діяпазоні 16,9–39,5 нм.

Key words: *Ficus carica*, silver nanoparticles, biosynthesis, plant extracts, nanotechnology, SEM, EDX.

Ключові слова: *Ficus carica*, наночастинки срібла, біосинтеза, рослинні екстракти, нанотехнології, СЕМ, ЕДРС.

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1. INTRODUCTION

Nanotechnology is one of the modern technologies used recently in many fields such as medical, electronic, food and cosmetic industries, textile industry, agricultural applications and many other areas [1]. The main aspect of current nanotechnology research is the development of a reliable green process for the synthesis of metal NPs.

Biosynthesis of NPs using bacteria, fungi, yeast, algae, and plant extracts has more advantages over chemical methods as it is an environmentally benign process, cost-effective, and has the ability of large-scale production over chemical methods [2]. The physical and chemical methods are being provided in synthesis by using high amounts of energy and toxic chemicals, while biological source synthesis methods are cheaper and the process is simpler and easier. It also makes these methods more advantageous, because they do not contain toxic chemicals during the synthesis phase [1, 3].

Extracts of different plant parts (roots, stems, leaves, fruits, fruit peels, and seeds) are used to produce nanoparticles for many minerals, the most important of which are silver, gold and oxides of some minerals such as zinc oxide (ZnO), copper oxide (CuO), and ferric oxide (Fe_3O_4), in addition to other metals oxides [4]. Among nanomaterials, Ag NPs are getting high attention. Plant-mediated silver nanoparticle synthesis has various benefits; it is of very low cost and can be achieved under ambient temperature; the process is comparatively fast in comparison with bacteria. Silver NPs have been extensively used in the packaging of food, preservation, cosmetics, and medicine [5]. Fig (*Ficus carica L.*) has a wide range of medicinal and nutritional values [6]. Its fruit, root and leaves are used in the native system of medicine in different disorders [7], and the clinical studies have shown that fig-leaves' extract have antitumor, hypolipidemic, antioxidant, antibacterial, hypoglycaemic, and other functions. Because fig leaves contain a large number of flavonoids, they have a variety of pharmacological activities [6].

Thus, from the above perspective, the aim of this study is to synthesize Ag NPs with figs (*Ficus carica L.*) leaf extract in an inexpensive and simple way and to characterize this nanoparticle at different concentrations of Ag^+ ion.

2. MATERIALS AND METHODS

The study was conducted in the laboratories of Syrian Private University and Atomic Energy Commission of Syria, Syria.

Figs (*Ficus carica L.*) leaves used in the study was obtained from Tishreen park, Damascus city, Syria in June 2022.

2.1. Preparation of Plant

Fresh leaves of figs plants were picked and cleared with tap water, then, rinsed with distilled water several times and dried with absorbent paper for 15 days in the shade. Finally, the dried leaves were grinded by electric grinder to a fine powder that was used for the extraction.

2.2. Preparation of Figs (*Ficus Carica L.*) Leaves Extract

Extraction was done using method described by Corciova *et al.* in 2022 with some modification. The dried and powder leaf (twenty grams) of figs were mixed with 400 mL of distilled water for one hour at 25°C, then, heated to 60°C for one hour with continuous stirring by a magnetic stirrer. The extract aqueous plant solution was left to stand for 24 h at 4°C, then, filtered by using Whatman No. 1 filter paper. The filtered was kept at 4°C for synthesis [8].

2.3. Preparation Silver Nitrate ($AgNO_3$) Solution

The mother solution was prepared from silver nitrate 1 M, by dissolving 33.974 g of it in 200 ml of deionized water. Then, several diluted solutions of it were prepared in 100 ml and different concentrations 0.05, 0.1, 0.3, 0.5, 1 M by taking the following volumes of the main solution 5, 10, 15, 50, 100 ml and complete the volume with deionized water to 100 ml. The containers were kept away from light to prevent oxidation of the silver ions.

2.4. Synthesis and Characterization of Ag NPs

10 ml extract was mixed with 90 ml of each concentration from silver nitrate; the addition of extract to silver nitrate was conduct slowly at 25°C for 4 hours with continuous stirring by a magnetic stirrer. The colour change was monitored (colours to yellow brownish) that indicates the formation of Ag NPs depending on the time [9]. After that, the pH of solution adjusted to 7 [8]. The solutions were left for 24 h, then, filtered with Whattman No. 1 filter paper. The precipitates were transferred to the oven, where it was dried at 60°C for 8 h. The nanoparticles were removed, after, weighed and transferred to a closed container and kept for further analysis [10]. The characterization of silver nanoparticles was carried out as follows according to the methods described by Baran *et al.* earlier in 2018 [11]. The surface structure was visualized by SEM (TESCAN, Czech Republic) at an accelerating voltage of 30 kV, and elemental analysis measurement was done using EDX (EDAX, USA) at an accelerating voltage of 20 kV [11].

3. STATISTICAL ANALYSIS

The data was subjected to one way-ANOVA IBM SPSS software package for Windows (Version 20, SPSS Inc., Chicago, IL); the statistical significance was evaluated at $P \leq 0.05$. The results were presented as mean ± standard deviation based on three replications.

4. RESULTS AND DISCUSSION

Metals like Ag have a very strong surface plasmon resonance, which is vital in the synthesis of nanoparticles [5]. Plants (fig leaves) containing specific bioactive compounds are able to reduce the metallic salt used as precursor in silver nanoparticles synthesis. Some molecules found in the chemical structure of plants, flavones, ketones, aminoacids, phenolics, aldehydes or vitamins have the role of reducing the silver ions. The silver source is chosen as silver nitrate ($AgNO_3$), since it is the most suitable precursor for Ag NPs. Using a reducing agent, the silver ions become free, while they are reduced to atoms. The atoms will be transformed further into clusters by a nucleation reaction. The stabilizing agent is present from the nucleation step until the end of the process. It assures that colloidal silver particles synthesized will not agglomerate, and a nanosize will be maintained [12].

Phytochemicals are reducing agents, which act in the reduction of Ag ions during the process of green synthesis. These reagents involve the transformation of precursors in metallic silver (Ag^0), which will induce the formation of clusters by glomeration. The clusters are further grouped to form colloidal silver nanoparticles [13]. To be maintained and performed the reaction in the best conditions, stabilizing agents must also be present. They have the role of stabilizing the nanoparticles' suspension, while nanoparticles are formed and avoiding a possible agglomeration of particles. The mechanism of such reactions is depicted in Fig. 1 [12].

The formation of the Ag NPs was marked by an onset of colour changes upon mixing the reactants. The colour changed from a bright green to brown after 5 min and, then, to dark brown after 30 min with varying degrees of colour depending on the concentration of silver nitrate used. The change in the colour of the Ag NPs solution can be linked with the excitation process of the surface plasmon vibration within the biologically synthesized Ag NPs as evident from the previously published literature. This optical property of the synthesized Ag NPs is usually sensitive to concentration, shape, size, and the agglomeration state of the synthesized nanoparticles [5]. The result showed that the masses of the product nanoparticles were increased with increase in concentration of silver nitrate as shown Table.

The first step of characterization study after the synthesis of NPs is the crystal structure and its chemical composition. The analytical tools of scanning electron microscopy (SEM) were used to analyse the size and morphology of the Ag NPs, while energy dispersive x-ray diffractometer (EDX) was used to determine the elemental composition.

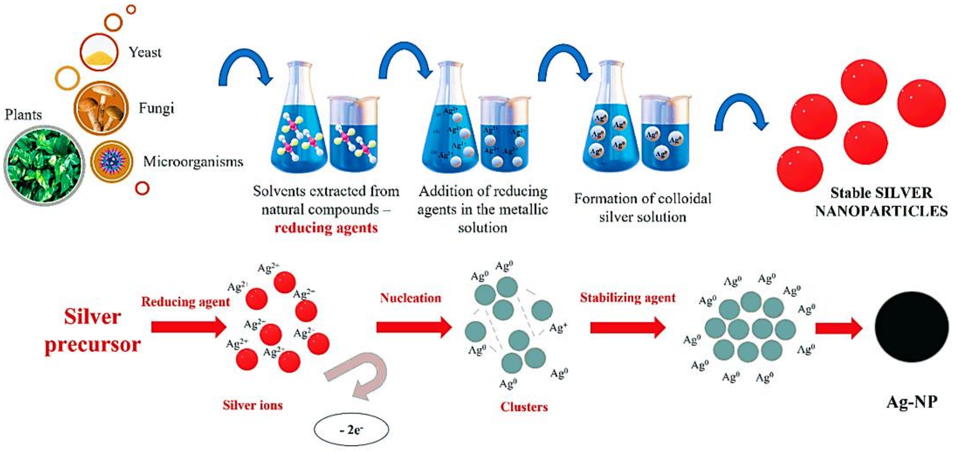


Fig. 1. Mechanism of chemical reduction of a silver precursor in Ag NPs formation [12].

TABLE. The change of the mass of the Ag NPs with the change of the used silver nitrate concentration.

Con. $AgNO_3$, M	0.05	0.1	0.3	0.5	1
Mass of Ag NPs, g	0.034±0.065	0.07±0.124	0.597±0.091	1.11±0.137	2.649±0.122

Note: *—values are mean ± standard deviation; **—different letters within the column indicate significant differences between the concentrations at every plant ($P \leq 0.05$).

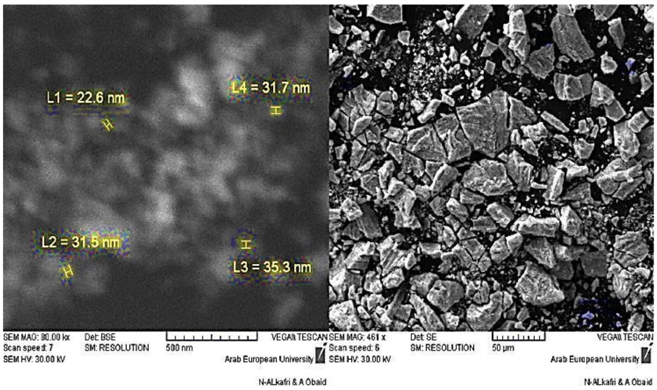


Fig. 2. SEM micrograph of the Ag NPs at 0.05 M $AgNO_3$.

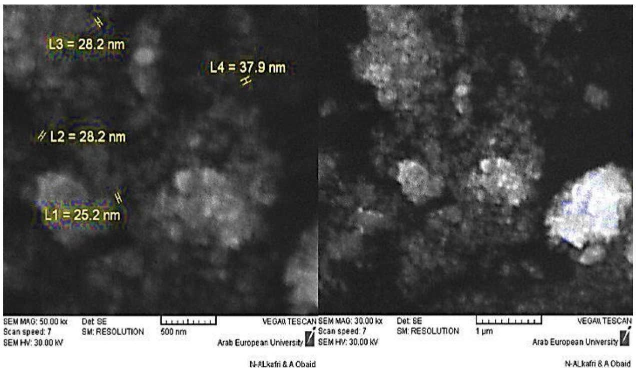


Fig. 3. SEM micrograph of the Ag NPs at 0.1 M $AgNO_3$.

When the SEM analysis data were examined, it was found that Ag NPs were crystalline in nature (of cubical shapes) at all concentration of silver nitrate. SEM analysis result was based on nanometre-scale imaging (Figs. 2–6). In other studies, it has been reported that Ag NPs have a spherical appearance [1, 9, 10], rod-like in shape [14]. The conditions of chemical reactions can influence on their shape and size as well [12]. Besides, the average particle size

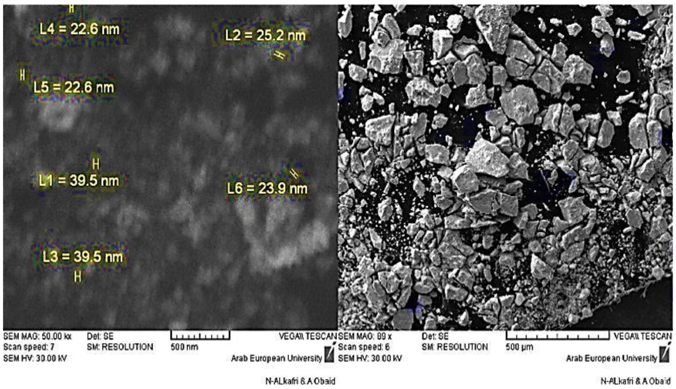


Fig. 4. SEM micrograph of the Ag NPs at 0.3 M $AgNO_3$.

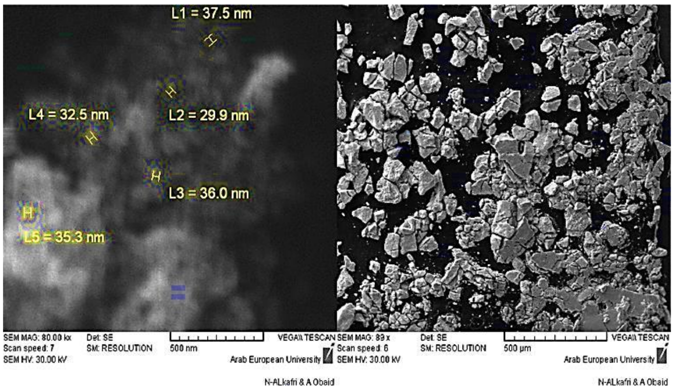


Fig. 5. SEM micrograph of the Ag NPs at 0.5 M $AgNO_3$.

as calculated using the ImageJ software from the SEM image was found to be between 22.6–35.3 nm, 25.2–37.9 nm, 22.6–39.5 nm, 29.9–37.5nm and 16.9–23.9 nm at 0.05, 0.1, 0.3, 0.5 and 1 M of $AgNO_3$, respectively. The research studies on silver-nanoparticle synthesis performed showed that the nanoparticles formed ranged in size from 80.11 to 157.01 nm [15], 72.656 nm [5], 17.3 nm [1].

In the EDX analysis, it was observed that the content of the element belonged largely to silver. Data analysis revealed that EDX spectra of Ag NPs mainly contained a specific and intense peak at $\cong$ 3 keV for Ag (79.85%, 69.80%, 95.69%, 93.27% and 95.15%) at 0.05, 0.1, 0.3, 0.5 and 1 M of $AgNO_3$, respectively.

In addition, it contained a peak for C and O; small quantities of P, S and Cl were observed at 0.05 and 0.1 M $AgNO_3$. Consequently, the results confirmed the synthesis of Ag NPs. The presence of other elements might be related to the breakdown of capping agents

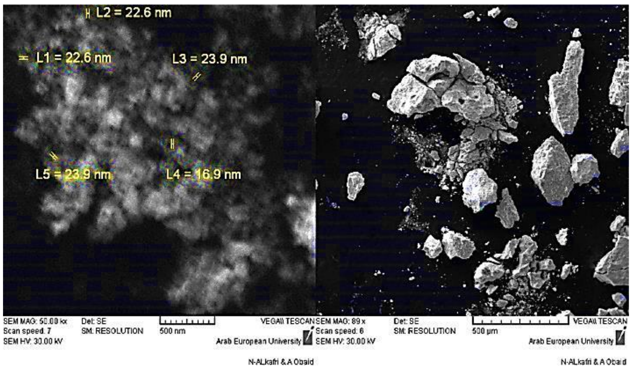
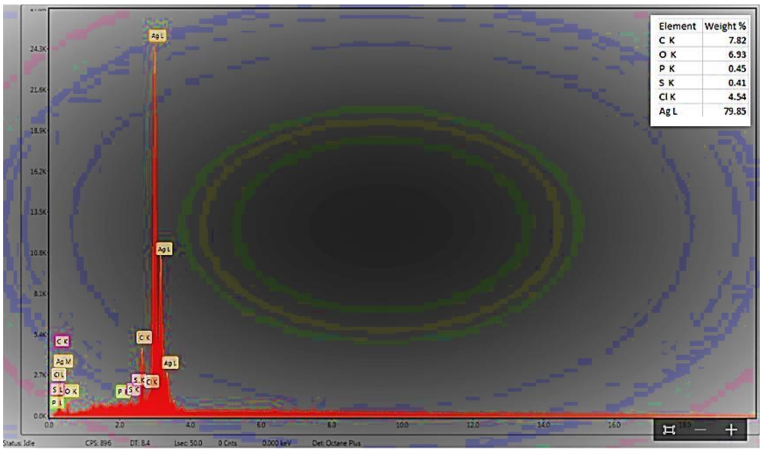


Fig. 6. SEM micrograph of the Ag NPs at 1 M $AgNO_3$.



Element	Weight %
C K	7.82
O K	6.93
P K	0.45
S K	0.41
Cl K	4.54
Ag L	79.85

Fig. 7. Analysis of the elemental composition by the EDX analysis of Ag NPs at 0.05 M $AgNO_3$.

from the surface of nanoparticles [8]; the presence of carbon indicates the presence of stabilizers [9]. Other concentration did not notice impurities from the heteroelements of silver, oxygen and carbon so these results indicated that the reaction product was composed of high purity Ag nanoparticles [16], in similar studies the element composition was evaluated [1, 17]. Figures 7–11 show the EDX spectrum of nanoparticles prepared with this bioreduction method.

From the previous results, it is noted that, after the concentration of 0.3 M of silver nitrate, there was no noticeable or significant change in the average size of nanoparticles during SEM analysis, nor in the elemental composition during EDX analysis; so, this concentration was dependent on it in subsequent studies.

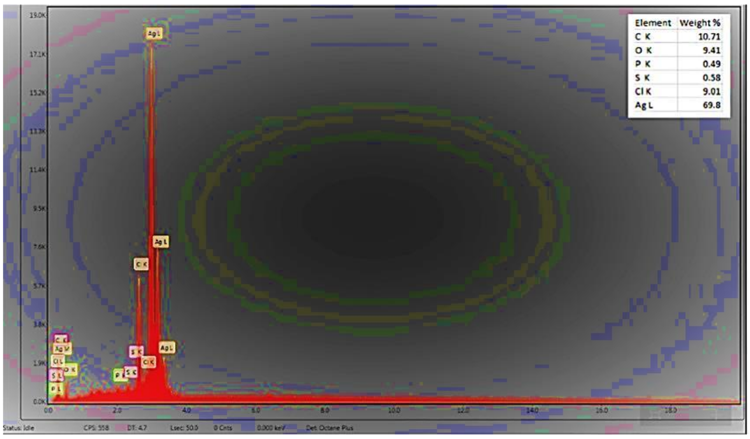


Fig. 8. Analysis of the elemental composition by the EDX analysis of Ag NPs at 0.1 M $AgNO_3$.

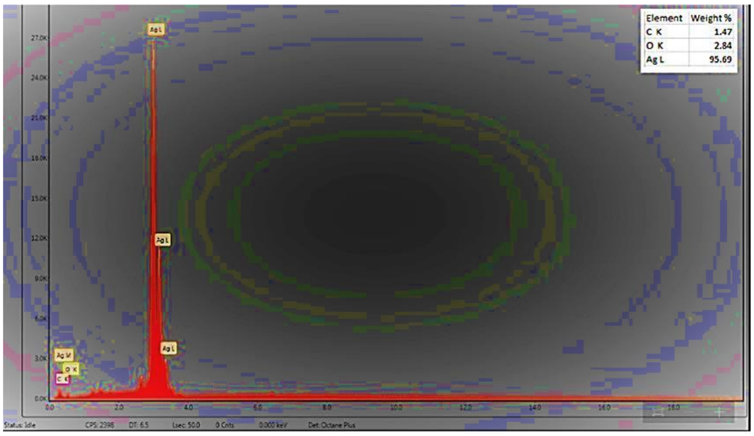
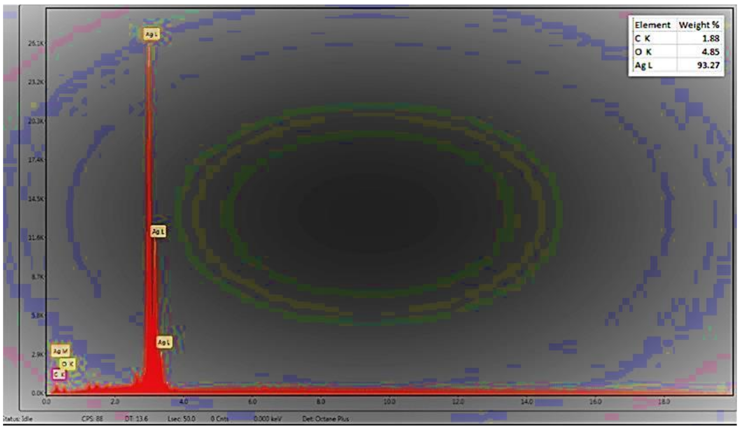


Fig. 9. Analysis of the elemental composition by the EDX analysis of Ag NPs at 0.3 M $AgNO_3$.

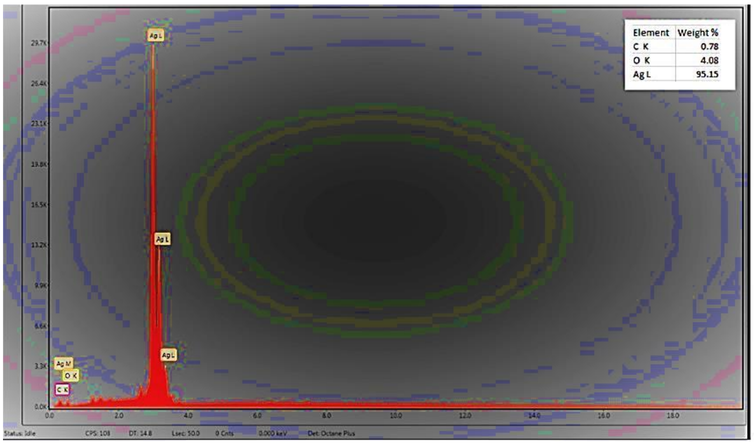
5. CONCLUSIONS

In this study, Ag NPs were synthesized by an eco-friendly and convenient method using agents—figs (*Ficus carica L.*) leaf extract. The characteristics of the obtained silver nanoparticles were studied using EDX and SEM techniques. The results confirmed the reduction of silver nitrate to silver nanoparticles with high stability and without any impurity at high concentration of silver nitrate; the presence of some C atoms suggests that some of the media components acts as a capping agent for stabilizing the nanoparticles.



Element	Weight %
C K	1.88
O K	4.85
Ag L	93.27

Fig. 10. Analysis of the elemental composition by the EDX analysis of Ag NPs at 0.5 M $AgNO_3$.



Element	Weight %
C K	0.78
O K	4.08
Ag L	95.15

Fig. 11. Analysis of the elemental composition by the EDX analysis of Ag NPs at 1 M $AgNO_3$.

Comparison of experimental results showed that particle size of synthesized silver nanoparticles is ranging between 16.9–39.5 nm. In addition, it is noted that changing the concentration of the silver nitrate solution contributed to a significant change in the amount of manufactured silver nanoparticles, but it did not have a significant effect on the size of these particles. As a result, we recommend the use of Ag NPs obtained from figs leaf extract as an alternative that can be developed and used in biotechnological and medical applications due to their small size.

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