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Synthesis, Characterization and Antioxidants Silver Nanoparticles Leaves of Prosopis Juliflora and Mentha Piperita

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Abstract

It is known that the green method is one of the environmentally friendly methods which attracted great attention by many researchers. So, our study focusing on the synthesis and characterization of silver nanoparticles (AgNPs) from stock solutions of silver nitrate, AgNO3 (0.001 M) of two aqueous solutions of extracted leaves of Prosopis Juliflora and Mentha Piperita. The structure, geometry, and sizes of the synthesised silver nanoparticles were identified by Ultraviolet-Visible (UV-Vis), Fourier transform infrared (FTIR), Scanning electron microscopy (SEM), Transmission electron microscopy (TEM), and electron dispersive X-ray (EDX) analysis. The formation of AgNPs was confirmed by the presence of a strong characteristic peak in the UV-Vis spectrum at around 425 nm. The FTIR showed the important functional groups. The synthesized silver nanoparticles have spherical shapes, individually ranging from 6.67-13.9 and 6.10-15.5 nm for Prosopis Juliflora (P. Juliflora) and Mentha Piperita (Mentha P.) extracts. The antioxidant activity of the obtained AgNPs was tested using the 1,1-diphenyl-2-picrylhydrazyl (DPPH) radical scavenging assay. It was found that they showed very powerful potent antioxidants.

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1. Introduction

In recent years, nanotechnology is one of the most important technologies that grew and studied widely by many researchers. This method has benefits as being applicable in various fields such as medical, engineering, biological sciences, physics and chemistry [1-7]. Generally, a particle is described as nano-scale if its diameter is in the range of 1 to 100 nanometers. Some environmentally unfriendly methods used to obtain nanoparticles (NPs) that can use metals such as gold, silver, platinum etc. [8-12]. Recently, green biosynthesis methods are using plant extracts, yeast, fungi, and bacteria are used to synthesis NPs, and they have interesting feature as excellent eco-friendly methods [13–15]. On the other hand, silver nanoparticles (AgNPs) produced by using plant extracts, as green method, received widely attention and reported by researches due to the stability, high dispersion, high surface areas, and small sizes of these nanoparticles [16,17]. The AgNPs can be synthesized from different types of plants including *Ziziphus Mauritiana*, *Carissa carandas* L. *Vigna unguiculata* stem, *Ficuscarica* Leave, and *Aquilegia Pubiflora*. Furthermore, these silver nanoparticles have different activities such as antioxidant and antibacterial activities [18-21]. The present study describes green method to prepare and characterize silver nanoparticles of aqueous extracts of *P. Juliflora* and *Mentha P.* plants leaves. Moreover, the antioxidants of two sources of AgNPs were also studied.

2. Materials and Methods

2.1. Plant materials

P. Juliflora and *Mentha P.* leaves were taken from Jear City, Abyan Governorate, Yemen. The leaves were cut, washed using distilled water, dry and grinded into powder form and kept at room temperature.

2.2. Preparation of plant extract:
The leaves powder was extracted by saturation of 40 g from powder onto 400 mL distill water, firstly. Then heat using a hotplate and stirrer at 40-45 °C for 25 minutes, and cool to room temperature. The prepared extracts were filtered with a piece of thick cotton cloth. The filtrates were kept for future use.

2.3. Synthesis of silver nanoparticles:
Gradually, using a burette, 30 mL of powder extract was mixed with 100 mL of silver nitrate ($AgNO_3$) solution (0.001M), then heated to 80 °C. After that, sodium hydroxide (NaOH) (1M) was added to the solution for neutralization. The solution color changed from yellow to brown precipitate. After 72 hours, separate the AgNPs with centrifuge for 10 min at 2800 rpm. Filtrate, wash with distilled water (three times), and dry in the oven for two hours at 60 °C.

2.4. Characterization of Silver Nanoparticles:
To confirm silver nanoparticles synthesis by taking 1 mL samples at 40-minute intervals and continuous scanning from 200 to 800 nm using UV–Visible spectroscopy Perkin Elmer Lambda 365. Moreover, to obtain FTIR spectra, Fourier transform infrared spectrometer (Perkin Elmer, Clarus 600 MODEL) from 450-4000 cm^{-1}, and the samples grinned with KBr.. SEM equipped with some detectors including TEM and EDX were used.

2.5. Testing antioxidant Activity:
Different concentrations of the two types of silver nanoparticles were prepared (50, 75, 100, 125, and 150μg/mL) in ethanol. The test tubes contained 3 mL of each concentration and 1 mL of freshly prepared 1,1-diphenyl-2-picrylhydrazyl (0.4 mM ethanolic solution). The reaction solutions were shaken and stored for thirty minutes. After that, the absorbance was determined along the wave lengths from 190 to 1100 nm. Percent activity was calculated by using a blank ascorbic acid solution. Percent antioxidant activity was determined from the following formula:

$$Percentage\ of\ inhibition = \frac{(A_o - A_1)}{A_o} \times 100\% \quad \dots (1)$$

where A_0 is the absorbance of DPPH alone and A_1 is absorbance of DPPH along with different concentrations of the samples. The IC_{50} value was calculated from the graph obtained by using the equation from the slope of the graph according to the following equation [22 and 23]:

$$y = mx + c \quad \dots (2)$$

3. Results and Discussion
The color change of the nitric acid solution from yellow to brown indicated the AgNPs formation. The color change indicated the electron transition and converted the Ag^+ to Ag^0.The UV-Vis spectra of Ag solution was determined in the range from 200to 800 nm to 72 hours and the absorbance was found to increase with time. The strong broad peak around 425 nm in the spectra indicated the surface resonance of Ag nanoparticles and confirmed the formation of AgNPs as shown in figures 1 and 2 [24].

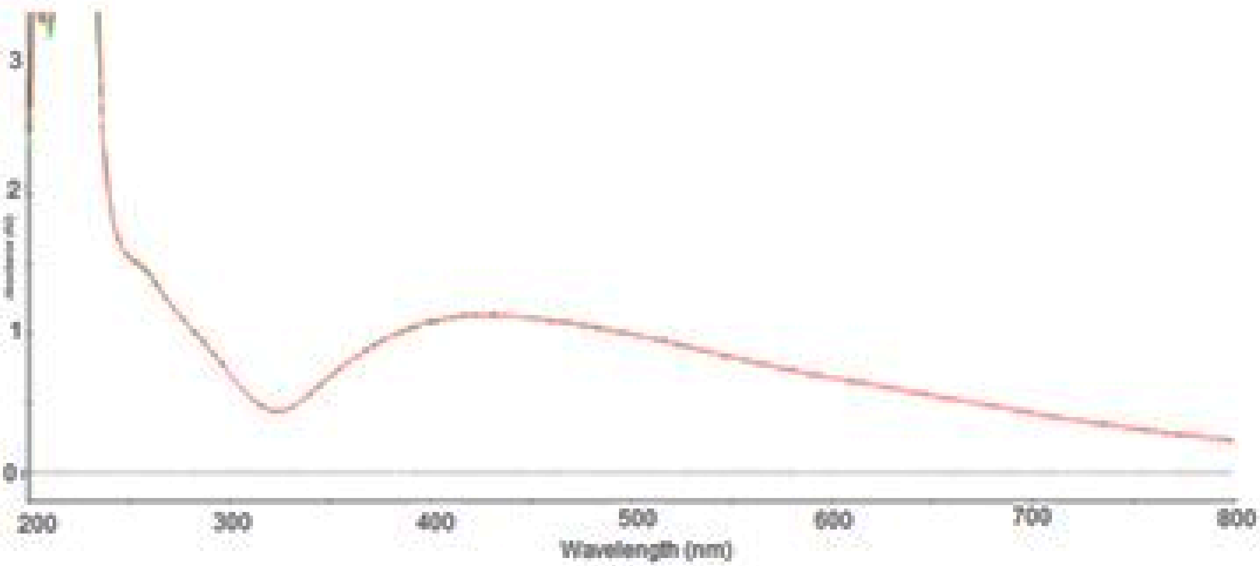

Figure 1. UV-Vis of AgNPs *Prosopis Juliflora* after 72 hours.

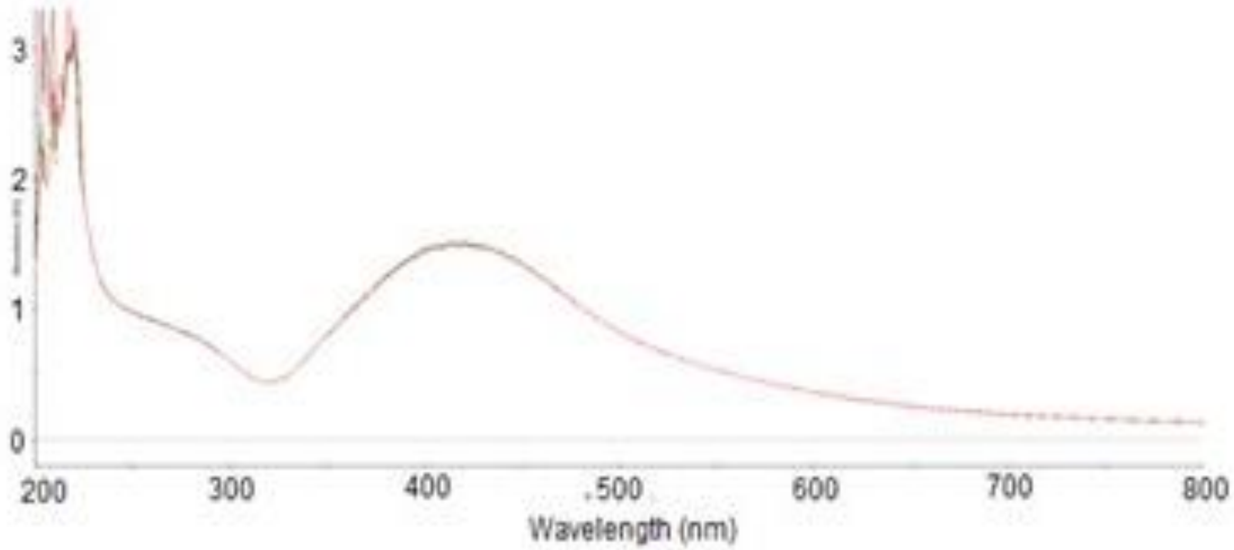

Figure 2. UV-Vis of AgNPs *Mentha piperita* .

Figures 3 and 4 exhibited the FTIR spectra, and showed the important absorptions of the functional groups in synthesized silver nanoparticles were contained, and confirmed the purity. The most characteristic frequencies in the FTIR spectrum of AgNPs of *P. Juliflora* were the O-H stretch at 3437.91 cm^{-1}, saturated -C-H stretch at 2913.50 cm^{-1}, C=O stretch at 1662.82 cm^{-1}, and conjugated C=C stretch at 1436 cm^{-1}. The FTIR spectrum of AgNPs of *Mentha P.* showed also stretching absorptions at 3350.58 cm^{-1}(O-H stretch), 2931.07 cm^{-1} (saturated -C-H stretch), 1713.15-1643.00 cm^{-1} (C=O stretch), 1451.14 cm^{-1} (unsaturated C=C). In both spectra the absorptions around 1200 cm $^{-1}$ were the frequencies of C-O bonds, the appearance of carbonyl and hydroxyl stretching frequencies indicated the carboxylic functional groups, and the N-H stretch frequencies centered around 3500 cm^{-1}. FTIR stretching absorptions indicate that the carbonyl, hydroxyle, amino, carboxylic groups, and unsaturated carbon double bonds were responsible in reduction of Ag^+ to Ag^0 nanoparticles.

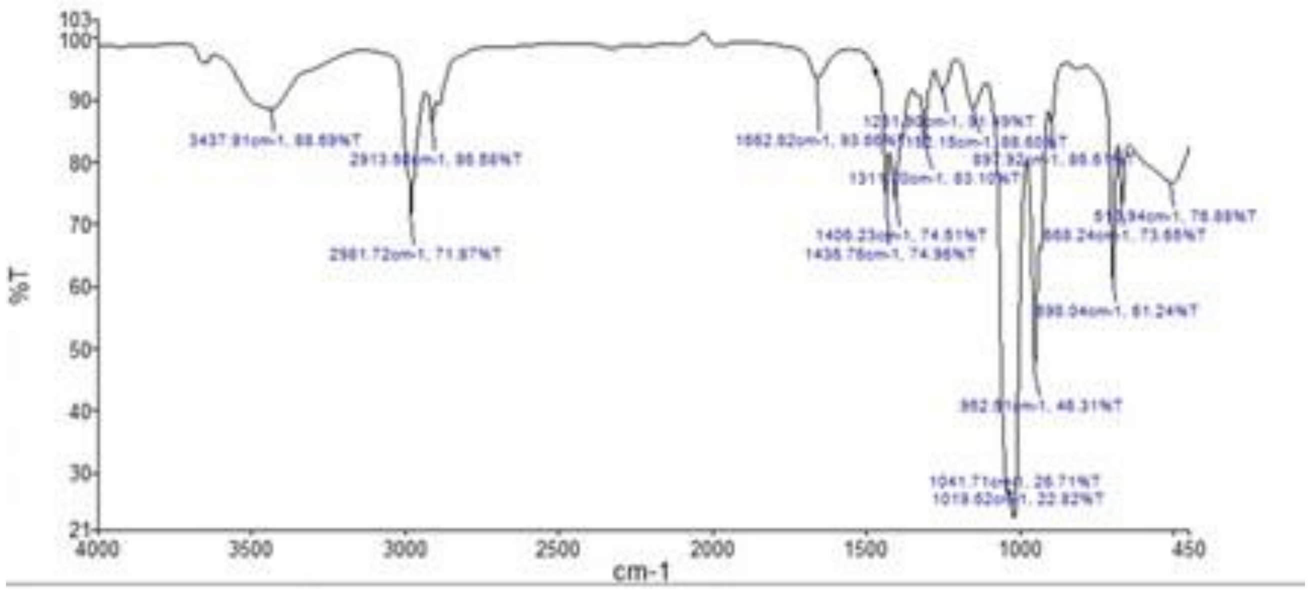

Figure 3. FTIR spectrum of AgNPs of *P. Juliflora*.

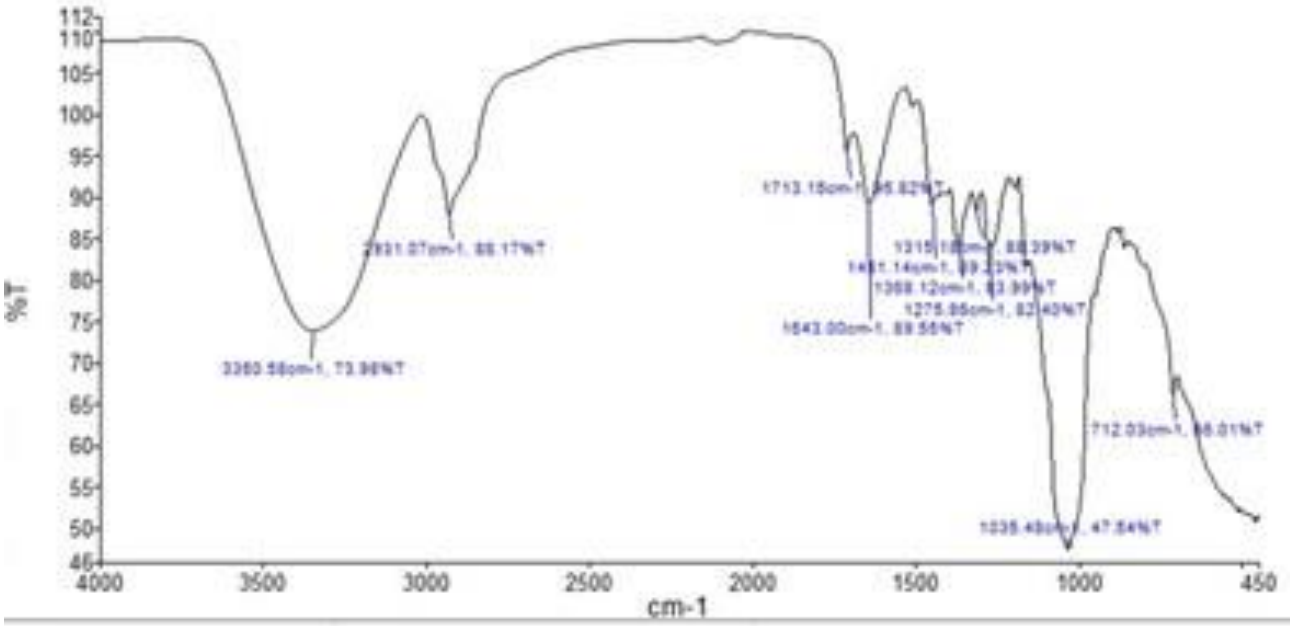

Figure 4. FTIR spectrum of AgNPs of *Mentha P.*

The morphological and elemental composition of the synthesized silver nanoparticles were identified from TEM and EDS analysis. The images of the TEM micrographs were observed in figures 5 and 6 which showed the spherical shapes with size 6.67-13.9 nm for *P. Juliflora* and 6.10-15.5 nm *for Mentha P.* The synthesized AgNPs were also elucidated by EDS spectra, figures 7 and 8. The peaks at 3 keV correspond to the surface plasma resonance of silver nanoparticles [21]. Moreover, the SEM micrographs in Figures 9 and 10 confirmed the morphological structure of the synthesized silver nanoparticles. The images showed the spherical structural nanoparticles in the levels of 6.67-13.9 and 6.10-15.5 nm for *Prosopis Juliflora* (P. *Juliflora*) and *Mentha Piperita* (*Mentha* P.) extracts, respectively.

The antioxidant data of *Prospis Juliflara*, *Mentha Piperita*, and ascorbic acid were mentioned in Tables 1, 2, and 3, respectively. The results showed that AgNPs from the *Prospis Juliflara* leaves extract were more active than the pure leaves of *Prospis Juliflara* extract [25]. In general, the substance is classified according to the IC_{50} value into very powerful, strong, or weak antioxidants. When the IC_{50} less than 50 ppm, the substance is described as very powerful antioxidant. They are described as strong or weak antioxidants if the IC_{50} value is 50-100 ppm, or 150-200 ppm, respectively [22]. The AgNPs from *Prospis Juliflara* exhibited IC_{50} equal 91.55µg/mL, figure 11, so they are very powerful antioxidants, and more active antioxidants compared with the pure leaves extract which were weak antioxidants [25]. Based on that, AgNPs from *Mentha Piperita* are also very powerful antioxidants. They showed IC_{50} value 89.13 µg/mL, figure 12, and this value slightly high than the IC_{50} of pure *Mentha* leaves extract [26]. However, compared with the standard ascorbic acid, figure 13, the results showed less antioxidant activity whether for AgNPs of *Prospis Juliflara* or *Mentha Piperita.*

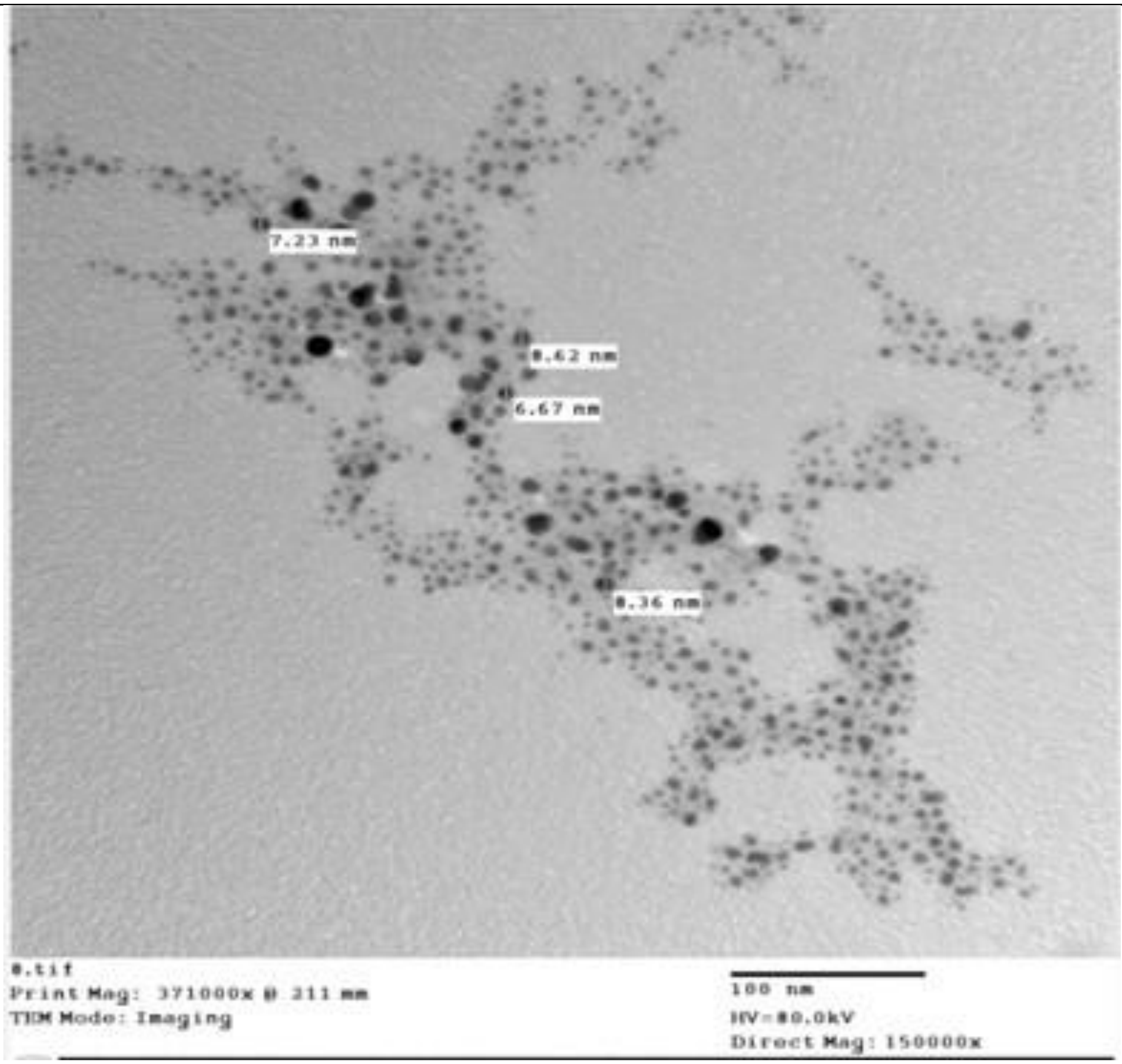

Figure 5. TEM Images of AgNPs of *Prosopis Juliflora*.

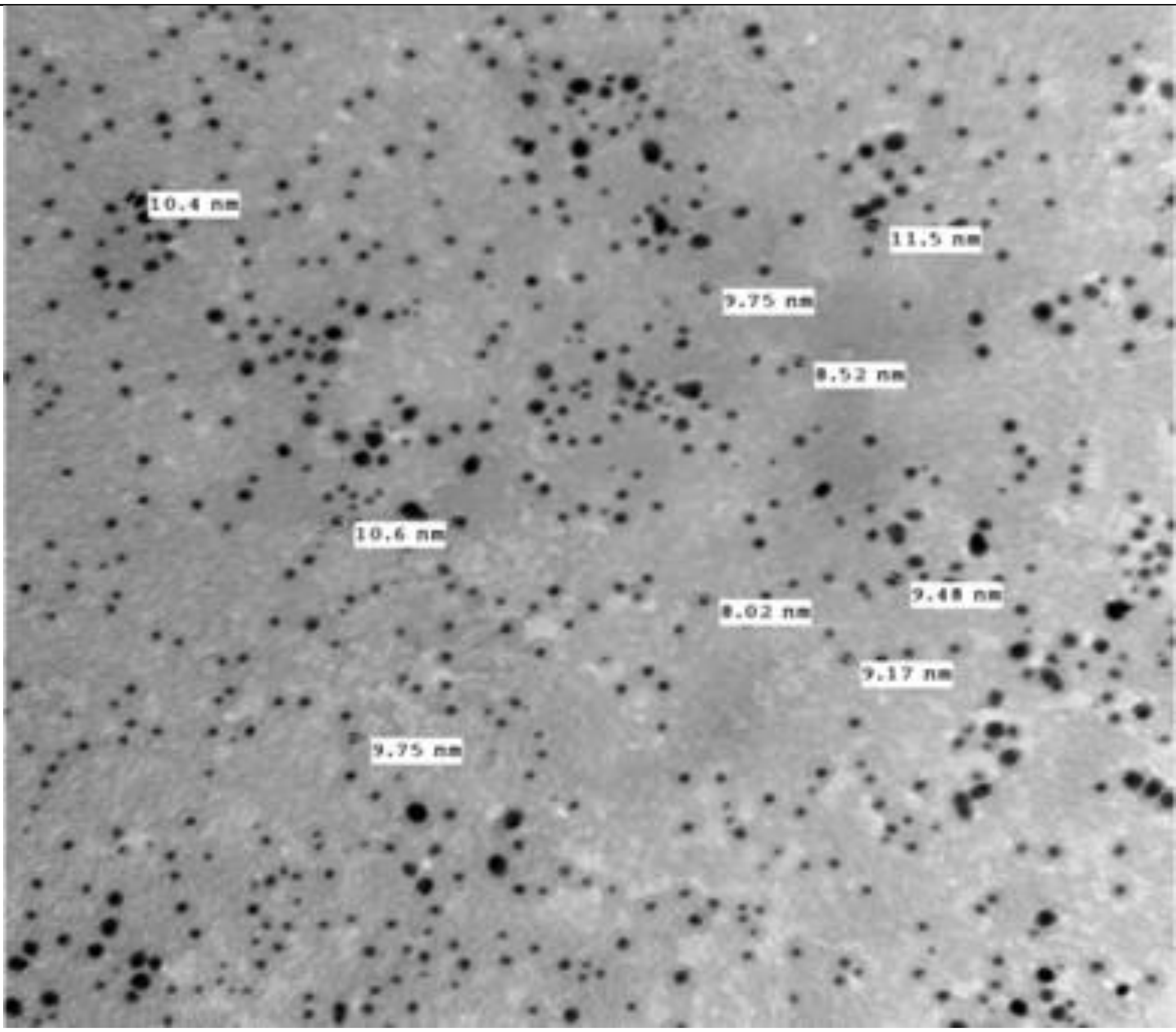

Figure 6. TEM Images of AgNPs of *Mentha P.*

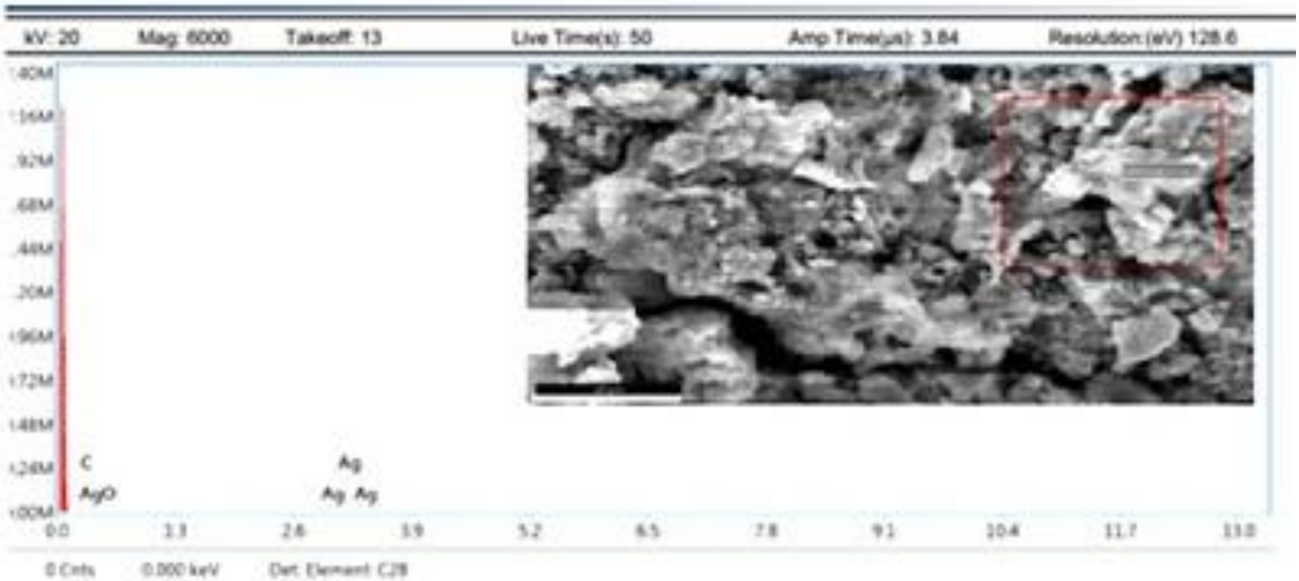

Figure 7. EDS of AgNPs of *P. Juliflora*.

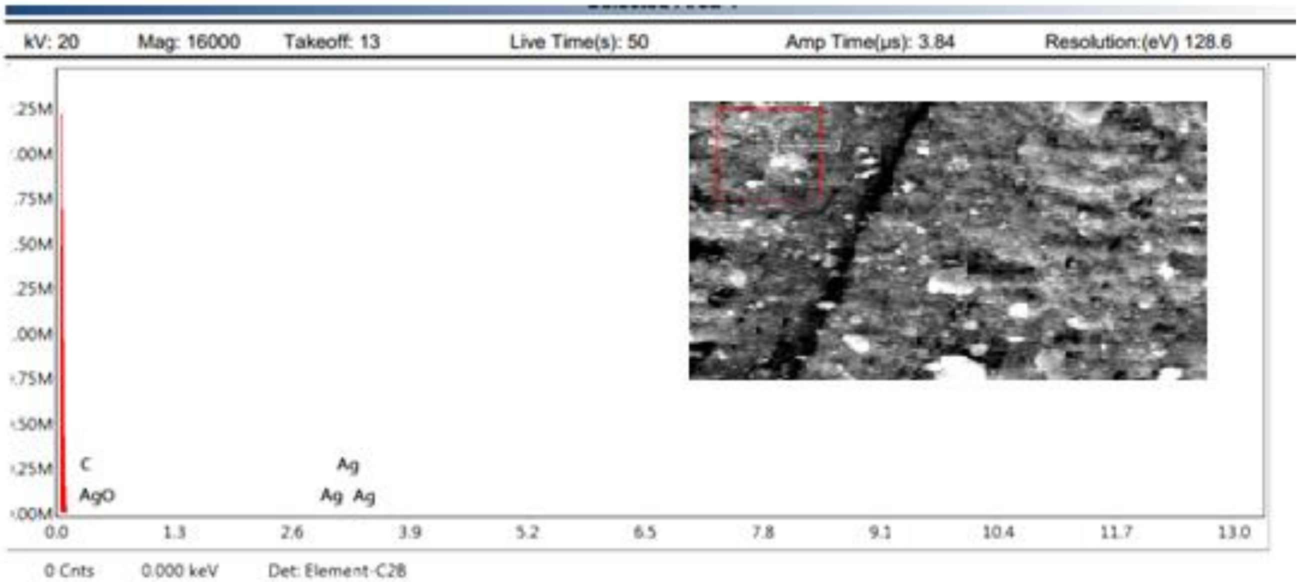

Figure 8. EDS of AgNPs of *Mentha P.*

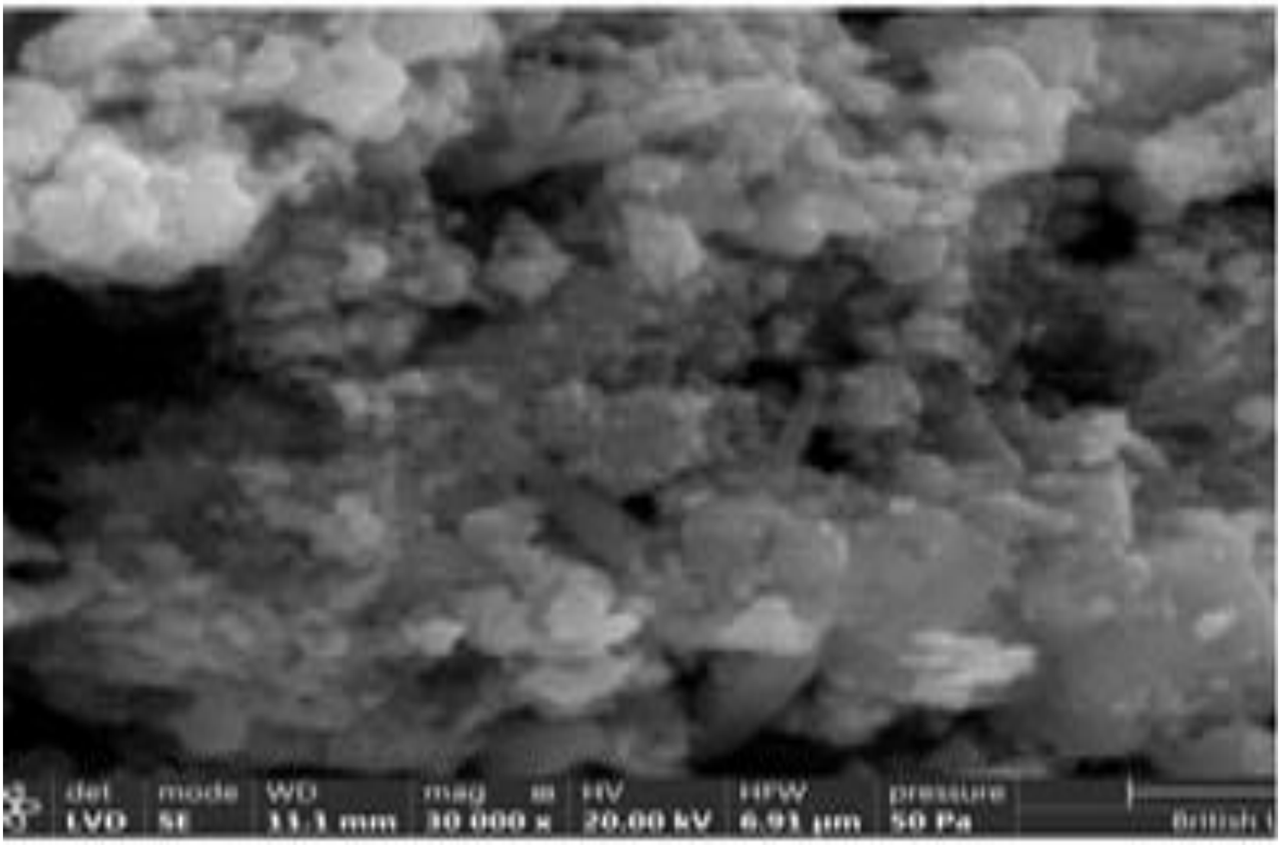

Figure 9. SEM image of AgNPs of *P. Juliflora*

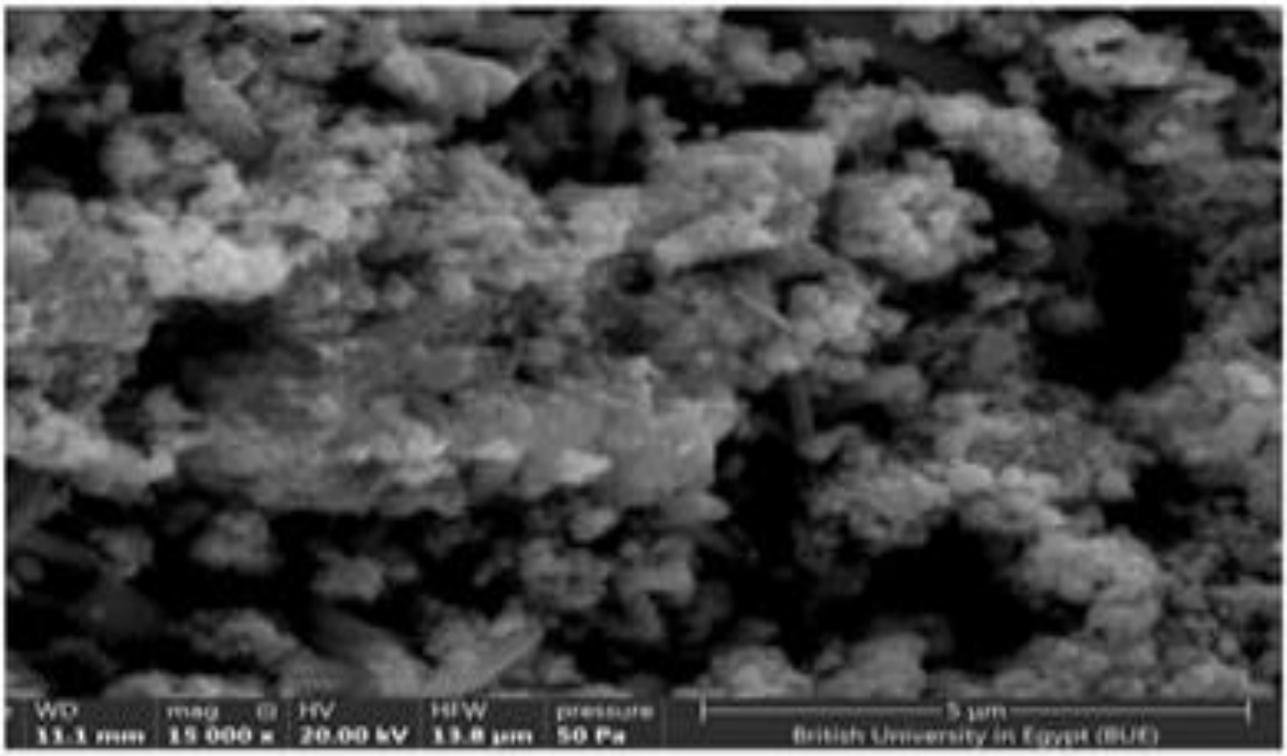

Figure 10. SEM image of AgNPs of *Mentha P.*

Table 1. Absorbance, percentage of inhibition value of AgNPs *Prospis Juliflara* (DPPH).

Concentration µg/mL	Absorbance	Inhibtion%
50	0.627	28.7
75	0.528	40.0
100	0.429	51.2
125	0.248	71.8
150	0.129	85.3

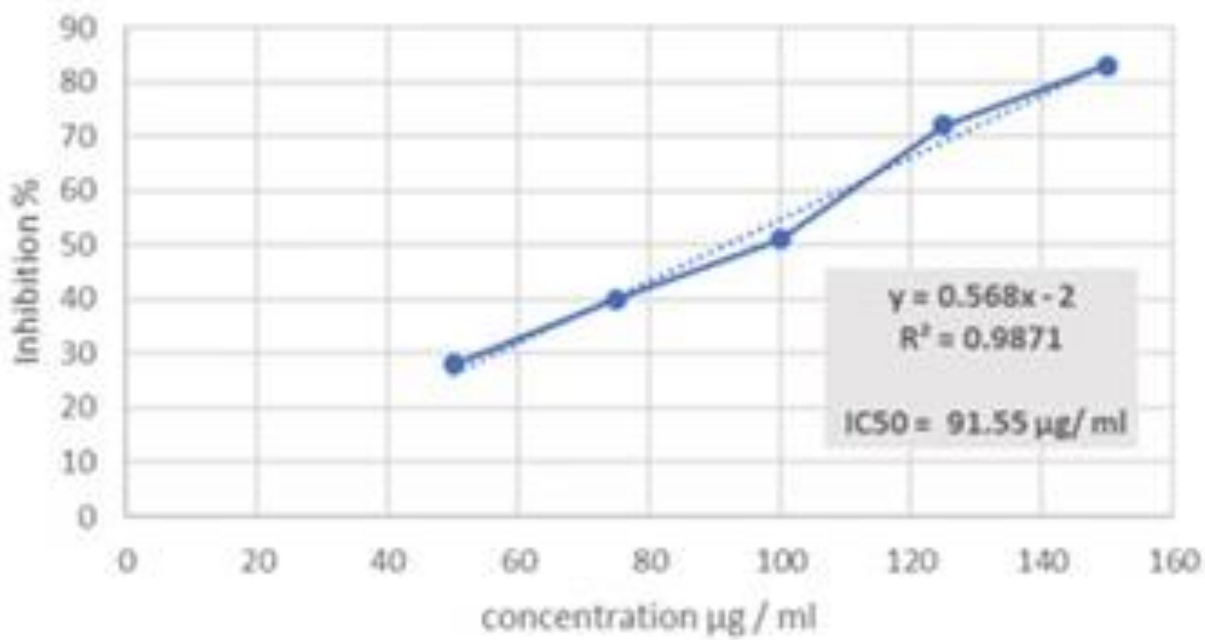

Figure 11. The DPPH scavenging of AgNPs *Prospis Juliflara*

Table 2. Absorbance, percentage of inhibition of AgNPs *Mentha piperita* (DPPH).

Concentration µg/mL	Absorbance	Inhibtion%
50	0.599	32
75	0.498	44
100	0.409	54
125	0.297	67
150	0.198	78

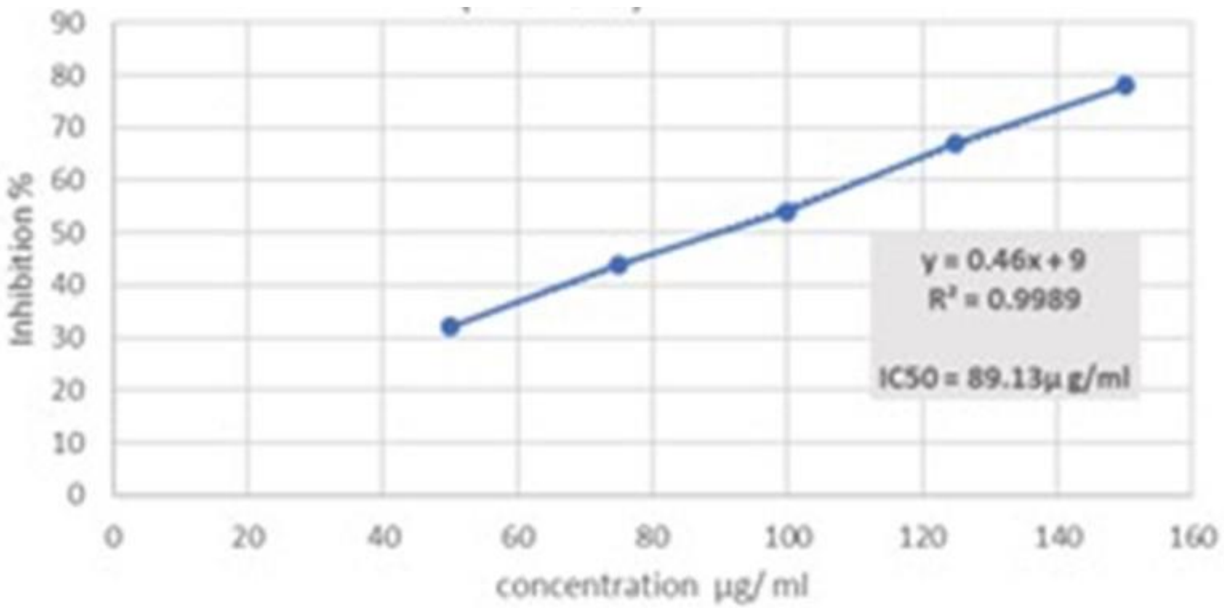

Figure 12. The DPPH scavenging of AgNPs *Mentha Piperita*.

Table 3. Absorbance, percentage of inhibition of ascorbic acid (DPPH).

Concentration µg/mL	Absorbance	Inhibtion%
50	0.692	20.0
75	0.554	37.0
100	0.418	51.0
125	0.245	73.2
150	0.065	92.2

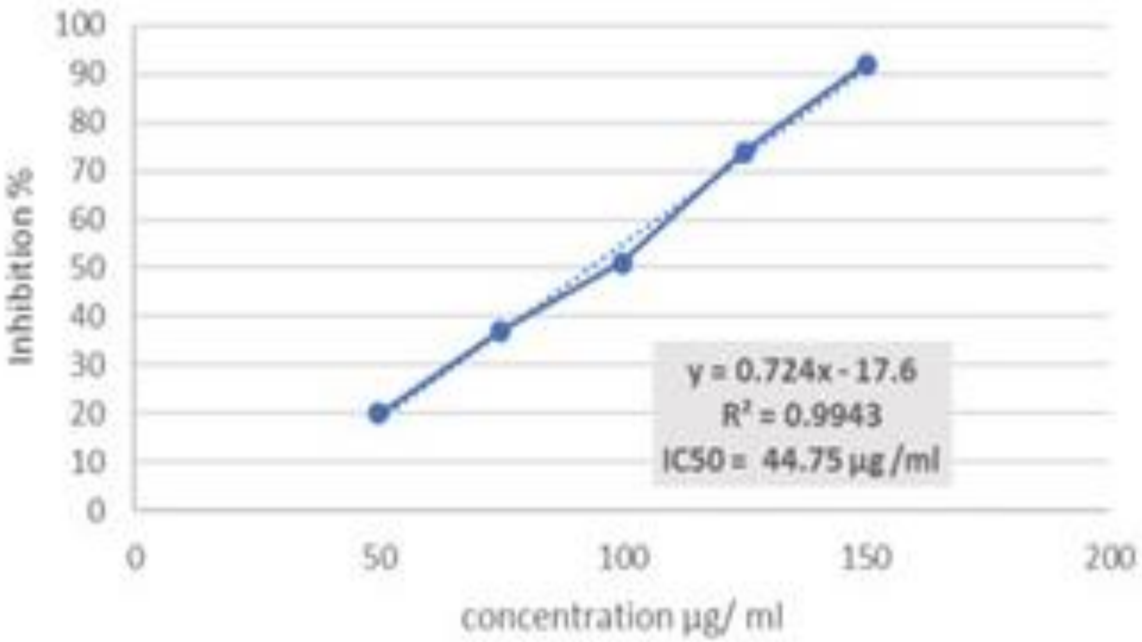

Figure 13. The DPPH scavenging of AgNPs ascorbic acid.

4. Conclusions

This work has reported the biosynthesis of AgNPs as eco-friendly method using two aqueous plant extracts, *P. Juliflora* and *Mentha P.* leaves. The converting of silver ions to nanoparticles was monitored via the development of a reaction mixture color to yellowish-brown, the appearance of characteristic peaks in UV-Vis and FTIR spectroscopy, as well as SEM, TEM micrographs, and EDS spectra to confirm the formation of the AgNPs. The formation of a peak around 425 nm in the UV-Vis spectra indicated the AgNPs production. The FTIR showed the most functional groups, and the chemical nature and phases of synthesized AgNPs were determined from EDS spectra. All SEM and TEM results showed that synthesized silver nanoparticles are spherical shape with 6.67 to 13.9 nm average size for of *P. Juliflora* and 6.10 to 15.5 nm of the *Mentha P.*AgNPs. The antioxidant test of the two prepared AgNPs was tested by using the DPPH method. The silver nanoparticles from *Prospis Juliflora* had the highest antioxidant compared to the pure *Prospis Juliflora* leaves extract. The results exhibited that AgNPs of *Prospis Juliflora* is a very powerful antioxidant with IC_{50} 91.55 ppm. The DPPH scavenging of AgNPs of *Mentha Piperita* also showed a very powerful antioxidation with IC_{50} equals 89.13 ppm.

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Conflicts of Interest
There are no conflicts of interest between the authors.

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