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Original Research Article

# Phytofabrication, and Characterization of $Ag/Fe_3O_4$ Nanocomposite from *Rosa Canina* Plant Extracts Using a Green Method

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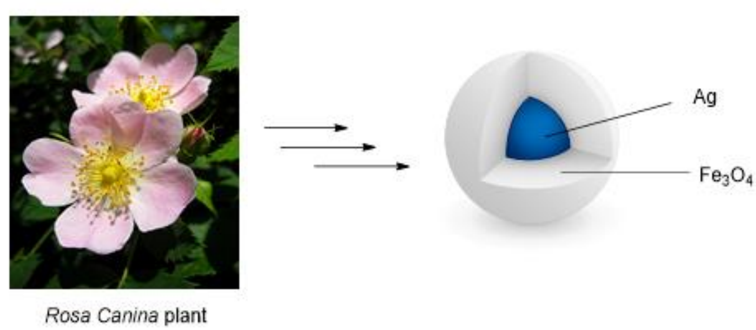
**ABSTRACT**

In this study, the $Ag/Fe_3O_4$ nanocomposite was prepared by *Rosa Canina* fruit extract as a suitable reducing source and stabilizing agent. The green synthesized nanostructure was characterized using UV–visible, X-ray diffraction analysis (XRD), field emission scanning electron microscope (FE-SEM), energy-dispersive X-ray spectroscopy (EDX), Fourier-transform infrared spectroscopy (FT-IR), and vibrating-sample magnetometer (VSM) techniques. TEM analysis of $Ag/Fe_3O_4$ nanocomposite showed a spherical shape (MNPs) with an average size of 8–28 nm.



## Graphical Abstract

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## Introduction

Research in the field of nanotechnology is growing rapidly due to their unique properties and their use in a wide range of applied programs in various sciences such as biological, chemical, and industrial sciences is expanding. The role of nanoparticles is increasing for use in cancer diagnosis, treatment, and the design of advanced drug delivery systems. Nanoparticles can be a potential alternative for treating diseases. Among the most important and widely used of these materials are silver nanoparticles [1].

Currently, it is believed that silver nanoparticles can be used as a therapeutic agent, in addition to be used in the fight against bacteria and the effect on wound healing, to fight against AIDS and viruses [2-5]. Likewise, Iron oxide nanoparticles are the foremost nanoscale materials with many practical applications such as biosensors, catalysts, and environmental remediation [6]. It plays a central role in a wide range of biomedical applications such as cell therapy, tissue engineering, drug delivery, photothermal effect, regenerative medicine, hyperthermia, and diagnosis [7-9]. There are several reports on the synthesis of magnetic nanoparticles where different reducing agents such as hydrazine, dimethylformamide, sodium borohydride, and carbon monoxide [10-14], etc. were used. These reducing agents are highly reactive chemicals that have adverse effects on the environment and hinder the biocompatibility of magnetic nanoparticles, which leads to the limited bio-medical applications of chemically reduced magnetic nanoparticles. To use magnetic nanoparticles in biomedical applications, they should be strictly biocompatible. Therefore, novel and environmentally friendly, biogenic reduction/greener synthesis methods are highly sought. Biogenic reduction methods which include the use of fungi, bacteria, and plant extracts, can be one of the best options to opt for the nanoparticles synthesis. Such greener methods are environment-friendly, cost-effective, provide good yield, and have decent reproducibility [15]. The availability of biogenic reductive materials in nature makes them a promising candidate for the nanoparticles synthesis. Such greenly synthesized biocompatible magnetic nanoparticles are particularly useful for magnetic separation of enzymatic catalysis for reuse [16]. Studies have shown that the bactericidal activity of transition metal NPs can be attributed to many different properties, the most important being the ability to generate ROS and their affinity to associate closely with R-SH groups [17, 18]. Today, drug-resistant bacteria have created serious problems in the treatment of many infectious diseases. Consequently, finding new ways to fight against these pathogens are important. Therefore, recent studies have focused on the study of the antimicrobial effects of plant origin [19]. The aim of this work is to synthesize the Ag/$Fe_3O_4$ nanocomposite using an aqueous extract of *Rosa Canina* fruit as a reducing agent and stabilizer.

## Experimental

### *Materials and methods*

All chemicals were of analytical grade purity and used as received. Ferric chloride hexahydrate, ferrous chloride tetrahydrate, ammonia solution (25%), were purchased from Merck company.

### *Preparation of Rosa Canina fruit extract*

20 g of dried powder of the *R. Canina* fruit was mixed with 100 mL of solvent (ethanol-water (1:1)). The mixture was stirred in a

shaker at 150 rpm away from light at room temperature for 48 h. The supernatant phase was removed and centrifuged 3 times at 3000 rpm for 10 minutes, and then filtered. After evaporating the solvent, the extract obtained through this method was poured into a glass container and stored at 255 K.

*Preparation of the $Ag/Fe_3O_4$ nanocomposite*

100 mL of 5000 ppm *Rosa Canina* fruit extract was taken in a 250 two-neck round bottom flask, and then 4 mmol $Fe_2(SO_4)_3.4H_2O$, 2 mmol $FeSO_4.7H_2O$, and 1.5 mmol $AgNO_3$ were added. After that, 10 ml of 9 M ammonia solution was injected dropwise into the mixture with vigorous stirring under $N_2$ atmosphere for 1 h at 333 K. The resultant solution was a dark brown color precipitate. The precipitate was separated by applying external magnetic field and washed with water for several times as well as dried in oven at 333 K for 24 h.

## Results and Discussion

Figure 1 displays the UV- visible spectrum of Rosa Canina fruit extract. This spectrum shows bonds at $\lambda_{max}$ 297 nm and 321 nm due to the transition localized within the ring of the benzoyl system and the cinnamoyl system, respectively. These bonds could be attributed to $\pi \rightarrow \pi^*$ transitions of the extract's aromatic system of phenolic compounds.

The UV-visible spectrum of the green synthesized $Ag/Fe_3O_4$ nanocomposite using the aqueous extract of *Rosa Canina* is depicted in Figure 2. The surface plasmon resonance (SPR) peak is centered at 421 nm which is demonstrating the nanocomposite formation.

Figure 3 demonstrates the X-ray diffraction pattern of the $Ag/Fe_3O_4$ nanocomposite. The patterns at $2\theta$ values 38.1°, 44.3°, 64.4°, and 77.2° can be assigned to (111), (200), (220), and (311) crystal planes in Ag cubic structure which agree with the standard Ag (JCPDS 04-0783) [20]. The diffraction peaks at $2\theta$ = 30.3°, 35.7°, 43.2°, 57.3°, and 63.03° can be indexed to (220), (311), (400), (511), and (440) planes of cubic $Fe_3O_4$ (JCPDS 19-0629), offering the formation of $Fe_3O_4$ particles [21]. The average size of the $Ag/Fe_3O_4$ nanocomposite was calculated using the Scherrer equation:

$$d = k\,\lambda / \beta \cos\theta$$

Where, d is the particle size of the crystal, k is Scherrer constant, λ is X-ray wavelength, β is the width of the XRD peak at half height, and θ is the Bragg diffraction angle. The average crystalline size of the $Ag/Fe_3O_4$ nanocomposite is calculated at 33 nm.

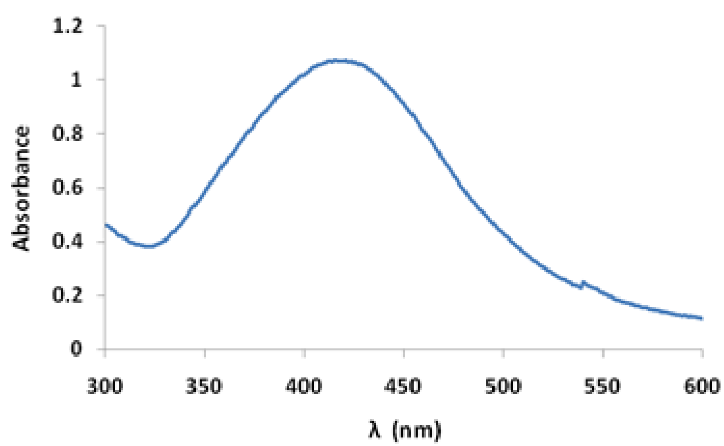


**Figure 1.** A UV-visible spectrum of *Rosa Canina* fruit extract

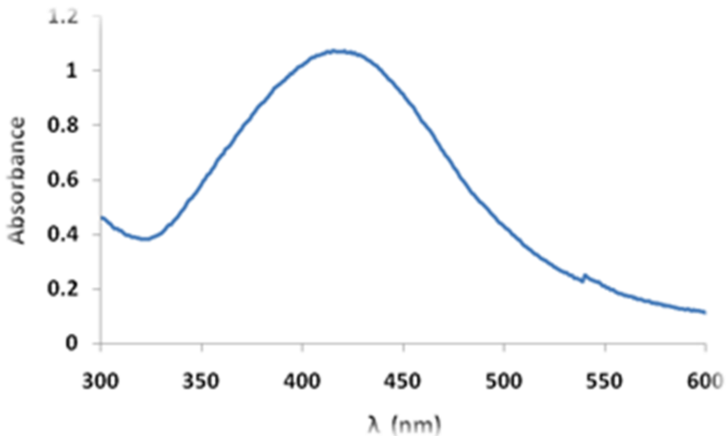


**Figure 2.** The UV-visible spectrum of Ag/$Fe_3O_4$ nanocomposite using *Rosa Canina* fruit extract

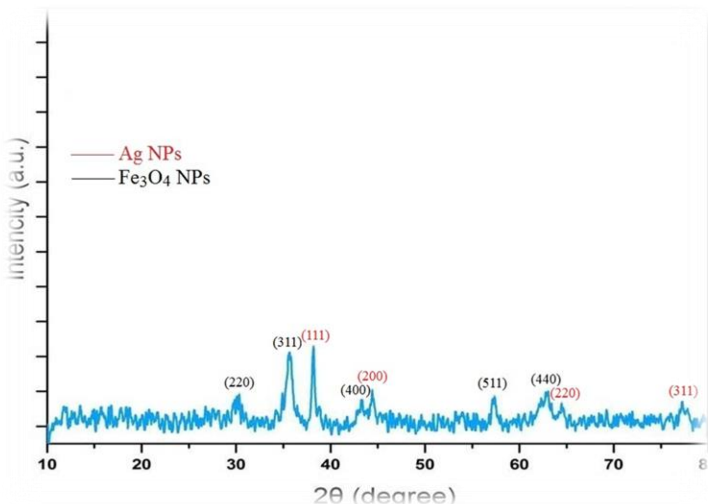


**Figure 3.** The XRD powder pattern of Ag/$Fe_3O_4$ nanocomposite

The FT-IR analysis was performed to recognize the possible biomolecules responsible for the reduction of the metal ions and capping of the bio-reduced nanoparticles. The FT-IR spectra of the *Rosa Canina* fruit extract and the Ag/$Fe_3O_4$ nanocomposite are illustrated in Figure 4. The FT-IR spectrum of the extract (Figure 4a) represented a broad peak at 3500 to 3000 which is attributed to free OH in the molecule and OH group forming hydrogen bonds [22]. The peaks at 1634, 1412, and 1098 $cm^{-1}$ in the spectrum of the extract represented a carbonyl group (C=O), stretching C=C aromatic ring, and C-OH stretching vibrations, respectively. These peaks could be ascribed to the presence of flavonoids and other phenolic compounds in the *Rosa Canina* fruit extract as responsible for the bioreduction of metal ions. Actually, the π-electrons of the carbonyl group of phenolic compounds in a Red/Ox mechanism can transfer to the free orbital of metal ions and change them to the zero-valent. The FT-IR spectrum of the Ag/$Fe_3O_4$ nanocomposite (Figure 4b), in addition to the peaks of phenolic compounds in the extract, represented the characteristic peak of Fe–O at 583 and 468 $cm^{-1}$, respectively [23].

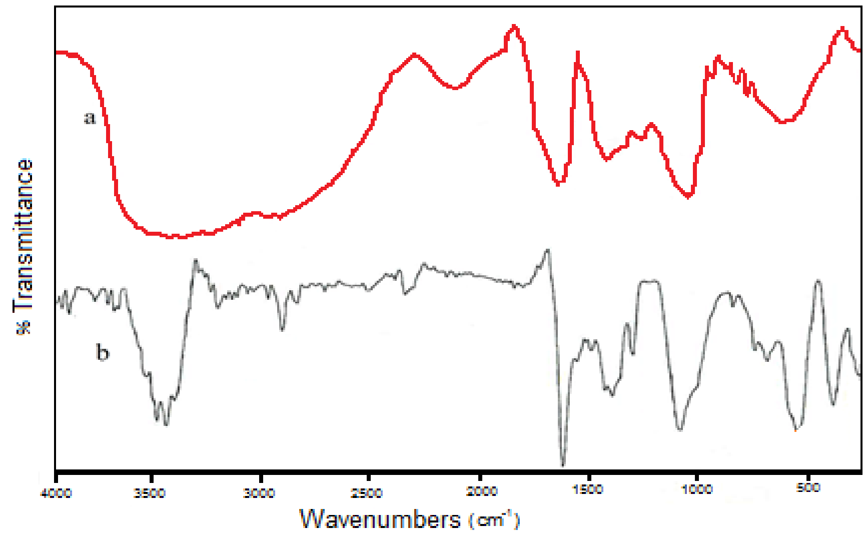


**Figure 4.** FT-IR spectra of (a) *Rosa Canina* fruit extract and (b) $Ag/Fe_3O_4$ nanocomposite

The morphology of $Ag/Fe_3O_4$ nanocomposite is revealed by field emission scanning electron microscopy (FE-SEM). FE-SEM analysis of the magnetic nanocomposite showed uniform-sized particles with spherical morphology (Figure 5).

Elemental analysis of synthesized nanocomposite ($Ag/Fe_3O_4$) was performed using the EDS technique. As demonstrated in (Figure 6), Fe, O, and Ag peaks indicate a successful synthesis. Furthermore, the presence of peak carbon in the EDS spectrum indicates the presence of organic compounds in a plant extract at the nanoscale.

TEM was another technique we applied to confirm the morphology of the $Ag/Fe_3O_4$ MNPs. In Figure 7, The transmission electron microscope image reveals the size of synthesized nanocomposite to be less than 30 nm.

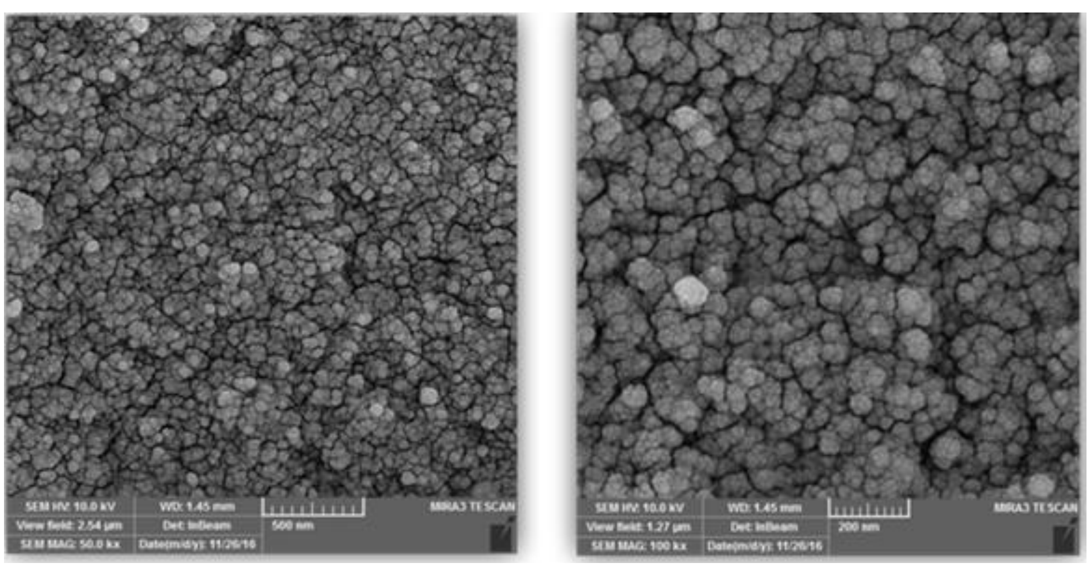

**Figure 5.** FE-SEM images of $Ag/Fe_3O_4$ nanocomposite

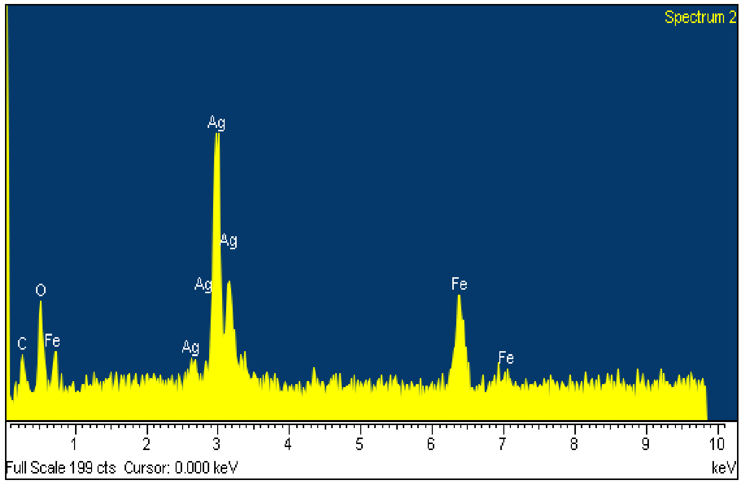


**Figure 6.** EDS image of the Ag/$Fe_3O_4$ MNPs

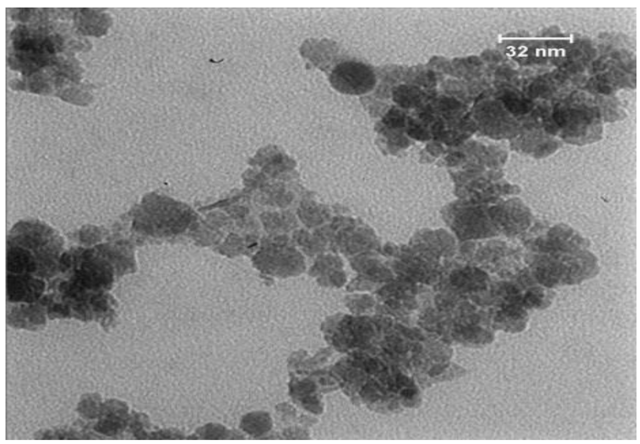


**Figure 7.** TEM image of the Ag/$Fe_3O_4$ MNPs

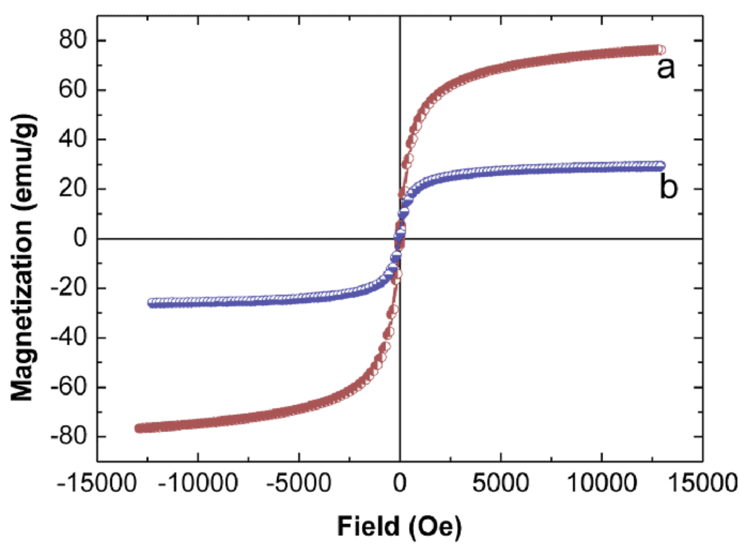


**Figure 8.** Magnetization loops of (a) $Fe_3O_4$ MNPs and (b) Ag/ $Fe_3O_4$ MNPs

Vibrating sample magnetometer (VSM) was used for the study magnetic property of catalyst at 300 °K by applying a magnetic field ranging from -15000 to 15000 Oe. The saturation magnetization (Ms) value $Fe_3O_4$ MNPs and Ag/$Fe_3O_4$ MNPs are 79.4 emu $g^{-1}$ and 22.5 emu $g^{-1}$, respectively. This experiment approves the superparamagnetic property of Ag/$Fe_3O_4$ MNPs and also confirmed that Ag coated well on the surface of $Fe_3O_4$ MNPs (Figure 8).

## Conclusion

The results of this research showed that *Rosa Canina* plant extract has the potential to regenerate iron and silver and is capable of synthesizing Ag/$Fe_3O_4$ nanocomposite, which was confirmed by different methods of nanocomposite synthesis. One of the advantages of this synthesis method is the use of plant extracts for nanocomposite synthesis, which is considered a green method and has advantages such as being environmentally friendly, cost-effective, simple, fast, and avoiding the presence of toxic solvents.

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## Disclosure Statement

No potential conflict of interest was reported by the authors.

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## Authors' contributions

All authors contributed to data analysis, drafting, and revising of the paper and agreed to be responsible for all the aspects of this work.

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