Research Article

Tunay Karan*

Biogenic synthesis of silver nanoparticles using *Consolida orientalis* flowers: Identification, catalytic degradation, and biological effect

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Abstract: Silver nanoparticles have attracted great attention due to their important usage areas recently. Silver nanoparticles were synthesized *via Consolida orientalis* flowers by green approach. The spectroscopic analyses characterized the synthesized silver nanoparticles (AgNPs@Co). The surface plasmon resonance of AgNPs@Co was determined as 425 nm by UV-Vis. The particle size was determined by X-ray diffraction (XRD) as 9.7 nm using the Scherrer equation. XRD analysis at 2θ with the angle of 38.17°, 44.29°, 57.49°, and 77.36° corresponded to planes [111, 020, 202, and 131] demonstrating the fcc structure. In addition, transmission electron microscopy analysis presented the particle size to be 11.9 nm as spherical. The functional moiety of bioactive compounds was displayed by Fourier-transform infrared spectroscopy analysis, and a characteristic hydroxyl was detected at 3,274 cm^{-1}. The zeta potential revealed the stability of nanoparticles as −20.3 mV. The signals at 2.3–3.4 keV in energy-dispersive X-ray spectroscopy proved the nanostructure. The catalytic activity of AgNPs@Co was executed using methylene blue in the treatment of sodium borohydride and degradation was determined as 71% in 45 min. Antioxidant of extract and nanoparticles was carried out using 2,2-Diphenyl-1-picrylhydrazyl (DPPH), 2,2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) and superoxide assays. The nanoparticles and extract exhibited good antioxidant activity with the values of 9.3 ± 0.2 and 11.2 ± 0.6 in the DPPH assay, respectively, in comparison with the standard butyl hydroxyanisole (6.5 ± 0.4). The silver nanoparticles may be a good antioxidant agent for drug development and the food industry.

Keywords: *Consolida orientalis*, silver nanoparticles, spectroscopy, natural products, antioxidant

*** Corresponding author: Tunay Karan**, Department of Genetics, Faculty of Veterinary Medicine, Yozgat Bozok University, Yozgat, Turkey, e-mail: tunay.karan@bozok.edu.tr
ORCID: Tunay Karan 0000-0002-9114-8400

1 Introduction

Plants have been employed in medicine and food for a long time [1–4]. The importance of plants is due to their bioactive compound content. Natural products refer to a chemical compound or substance that is derived from a living organism, such as plants, animals, or microorganisms. Natural products have been a basic resource of drugs and therapeutics throughout history. They often possess diverse chemical structures and biological activities, making them valuable in fields such as medicine, agriculture, and cosmetics [5]. Moreover, natural products often behave as principals for the growth of new drugs through isolation, structural modification, and synthesis [6–8].

Nanotechnology has gained significant curiosity lately owing to its extensive application areas including medicine, cosmetics, agriculture, textile, electronics, optics, energy, and water purification [9]. The particle size of nanoparticles is assumed to be 1–100 nm. Physical and chemical methods are commonly used for nanoparticle synthesis. Nevertheless, these conventional methods contain toxic chemicals that harm organisms and the environment. Therefore, natural products have been preferred for nanoparticle synthesis since this method is cost-effective, sustainable, and eco-friendly [10]. Nanoparticles possess unique properties and behaviors owing to their surface area. The properties of nanostructures can differ significantly from the same material in bulk form, making them valuable for many application areas [11]. Enzymes, DNA, and receptors are about 100–10,000 times smaller than cells. Silver nanoparticles with functionalized surfaces are effective and promising tools for drug release purposes. Functionalized silver nanoparticles can exert a synergistic antiproliferative effect in various cancer cell lines and can be employed in the cure of various kinds of cancer. The silver nanoparticles produced using natural sources were stated to demonstrate substantial activities including antioxidant, anticancer, and antibacterial effects [12].

Consolida orientalis belonging to the Ranunculaceae family is distributed to Europe, Asia, and Africa. Phytochemical study of this plant displayed the isolation of norditerpenealkaloids, delcosine, 18-demethylpubescenine, 18-hydroxy-14-*O*-methylgadesine, takaosamine, gigactonine, and 18-methoxygadesine [13,14]. In addition, various extracts of this plant were reported to present substantial pharmaceutical effects such as antioxidant [15], anticancer [16], and pesticidal activity [17]. A reported study presented that the diterpenoid alkaloid, norditerpenoid alkaloids delsoline, delcosine, gigactonine, and takaosamine were isolated and identified from *C. orientalis* [18].

Free radicals are extremely reactive compounds having an unpaired electron [19]. This unpaired electron makes them unstable and highly reactive, as they seek to pair up with another electron to achieve stability. Free radicals can be generated through various processes, including normal metabolic reactions in the body, exposure to environmental factors such as pollution or radiation, and certain lifestyle choices such as smoking or excessive alcohol consumption [20]. Antioxidants are substances that can neutralize free radicals by providing an electron without becoming unstable themselves. They facilitate and protect cells from the detrimental effects of free radicals [21]. Antioxidants can be obtained from a healthy diet such as a variety of fruits, vegetables, and beverages. Additionally, the body produces its antioxidants to decrease the negative effects of free radicals [22]. Natural antioxidants are substances found in nature that can help protect cells from the damaging effects of free radicals. They work by neutralizing free radicals, preventing, or minimizing oxidative stress and the associated cellular damage [23]. Natural substances known as secondary metabolites are not directly related to survival, growth, and proliferation. Secondary metabolites are largely produced by organisms for ecological goals, such as protection against predators, competition for resources, or attracting pollinators, in contrast to primary metabolites, which are necessary for fundamental life processes [24]. These compounds are often synthesized in specialized cells or tissues and may accumulate in specific plant parts or be secreted into the surrounding environment. Secondary metabolites are found in various organisms, including plants, bacteria, and marine organisms. They exhibit lots of chemical structures and biological activities. Some common classes of secondary metabolites include alkaloids, terpenoids, phenolic compounds, flavonoids, glucosinolates, and saponins. Each class of secondary metabolites has unique chemical properties and biological functions [25].

Natural products play an important role in nanoparticle synthesis. The cobalt oxide nanoparticles were synthesized using *Muntingia calabura* leaves and it was reported that these nanoparticles displayed high activity against methylene blue (MB) [26]. *Tabebuia aurea* leaf extract mediated synthesis of α-Fe_2O_3 nanoparticles was achieved and these nanoparticles have the potential to be used in biomedical function [27]. In addition, silver nanoparticles were synthesized using cocoa pod shell and their potential usage in nanocatalysts and antifungals was investigated [28].

Herein, AgNPs@Co were synthesized using *C. orientalis* flowers, and their antioxidant, and catalytic properties were examined. *C. orientalis* includes significant bioactive compounds. In addition, this plant displayed important biological effects. Hence, the reduction, stabilization, and capping of silver by the bioactive compounds found in *C. orientalis* could be a promising agent for the food and pharmaceutical industry and raw material for the drug development process.

2 Materials and methods

2.1 Plant material

C. orientalis (Gay) Schrodiger was collected in June 2022 in Artova-Taspinar village, Tokat, Turkiye, at 40°07′46.6″N 36°19′12.4″E. The botanical identification of the plant was carried out by Dr. Murat Unal at the Department of Biology, Van Yuzuncu Yil University, and a voucher specimen was deposited at the herbarium (No: VANF 20295).

2.2 Synthesis of silver nanoparticles

Air-dried *C. orientalis* leaves (5.0 g) were heated in deionized water (100 mL) at 45°C for 3 h. After the temperature of the solution had decreased to room temperature, it was filtered (Whatman No. 1) and then reacted with $AgNO_3$ (100 mL, 1. 0 mM) at 40°C for 1 h. The solution was subjected to centrifugation for 15 min and dried by the lyophilization process to yield the product [29].

2.3 Identification of nanoparticles

The identification of AgNPs@Co was carried out *via* advanced techniques. The absorbance was established by a UV-Vis spectrophotometer (Hitachi U-2900). This is the most valuable and accessible technique to confirm the formation of nanoparticles. The wavelength range was applied to 300–800 nm. Fourier-transform infrared spectroscopy (FT/IR 4700) was utilized to identify the natural compounds. The particle size and the crystal structure were determined by X-ray diffraction

(XRD) analysis (Panalytical) using HighScore Plus software. Transmission electron microscopy (TEM, Hitachi HT-7700) was utilized to reveal the dimensions and structure of nanoparticles. Zeta potential was evaluated by Zetasizer Nano ZSP. The energy-dispersive X-ray spectroscopy (EDX) detector was employed to determine the elemental composition.

2.4 Catalytic activity

The catalytic activity of nanoparticles was determined by the treatment of MB (10.0 ppm, 2 mL) with sodium borohydride ($NaBH_4$) (1.5 mL) in the presence of AgNPs@Co (60.0 ppm, 150 µL). UV-Vis spectrophotometer (663 nm) was employed for the reaction progress. Formula (1) was used to calculate percentage degradation

$$\text{Degradation}(\%) = (Ax - Ay/Ax) \times 100 \quad (1)$$

where Ay is the last time and Ax is the first-time absorbance [30].

2.5 Superoxide radical scavenging activity

Hydroxylammonium chloride was utilized for the oxidation of radicals. A spectrophotometer at 530 nm was employed for the detection of nitrite formation. The phosphate buffer (pH 7.4, 1.0 mL, 65 mM), xanthine (0.1 mL, 7.5 mM), hydroxylammonium chloride (0.1 mL, 10 mM), sample (0.1 mL, 2.0 mg·mL^{-1}), and deionized water (0.4 mL) were added in a reaction flask. Moreover, xanthine oxidase (0.3 mL, including 60 µg of protein) was added to the flask. The reaction mixture was incubated for 25 min at room temperature (rt). Sulfanilic acid (0.5 mL, 20 mM) and naphthylamine (1.0%, 0.5 mL) were added and stirred for 5 min. After stirring for 25 min at rt, the optical density of the mixture was measured at 530 nm. The radical scavenging activity of α-tocopherol at different concentrations (1–10 µg) was measured and used for presenting the superoxide radical scavenging effect of extract and nanoparticles [31].

2.6 DPPH$^\bullet$ activity

The radical scavenging activity of extract and nanoparticles was determined using the 2,2-diphenyl-1-picrylhydrazyl (DPPH) assay. Briefly, sample extracts (3.0 mL, 5–50 µg·mL^{-1}) were mixed with the 1.0 mL of 0.26 mM DPPH solution. The mixture was stirred and incubated at rt for 30 min. The absorbance was measured (517 nm) [32].

2.7 ABTS$^{\bullet+}$ activity

The radical cation scavenging activity 2,2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) (ABTS$^{\bullet+}$) of nanoparticles and extract was performed. The ABTS$^{\bullet+}$ radical cation was produced by reacting 7 mM ABTS with 2.45 mM potassium persulfate in incubation at rt in the dark for 16 h. The various concentrations of AgNPs@Co (5–50 µg·mL^{-1}) were reacted with ABTS$^{\bullet+}$ solution. The absorbance was measured at 734 nm [33].

2.8 Statistical analysis

The statistical analysis of antioxidant activity was carried out using GraphPad Prism (v.8.0). The results were satisfied the homogeneity and normality of distribution, and one-way ANOVA followed by Turkey's test was utilized. The results were stated as mean values with standard deviations ($P < 0.05$).

3 Results and discussions

3.1 Synthesis and UV-Vis analysis

C. orientalis leaves were used for the synthesis of AgNPs@Co. *C. orientalis* leaves include secondary metabolites acting as reducing, capping, and stabilizing agents. The reaction between silver nitrate and the compounds in the plant extract is an oxidation–reduction reaction. While the bioactive compounds in the plants are oxidized, silver ions (Ag^+) are reduced to silver metal (Figure 1) [34]. Plant extracts were effectively used for nanoparticle synthesis. Silver nanoparticles synthesized using *Tagetes erecta* leaf extract displayed high antioxidant activity [35]. *Origanum onites*-mediated synthesis of silver nanoparticles was achieved, and these particles displayed significant antioxidant activity [36]. The present study accords with the reported ones. Each plant includes different secondary metabolites in combination with different quantities. So, nanoparticles synthesized from each plant would display the various biological effects.

UV-Vis analysis is known as a powerful procedure to present the metal nanoparticle formation. The characteristic absorption signals appeared at 400–450 nm. The color change and UV-Vis analysis confirmed the accomplishment in the synthesis of AgNPs@Co. The maximum absorption at 425 nm indicated the surface plasmon resonance of nanoparticles (Figure 2).

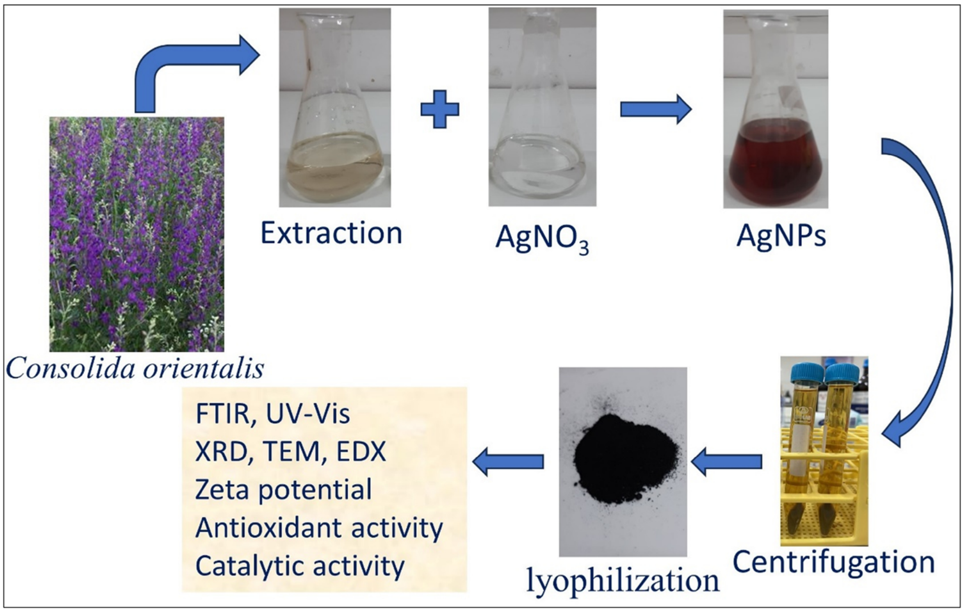

Figure 1: Synthesis of AgNPs@Co using *C. orientalis.*

3.2 FTIR analysis

The possible interfacial groups between the capping agents and silver nanoparticles were identified by FTIR analysis. FTIR spectroscopy was employed to present the functional group's presence on the nanoparticles synthesized by *C. orientalis* flowers. The absorption signals were assigned to OH stretching vibrations in phenolic (3,274 cm^{-1}), carbon–carbon double bond stretching of disubstituted alkene (1,636 cm^{-1}), stretching of N–O (1,515 cm^{-1}), O–H bending of alcohol (1,399 cm^{-1}), and C–O stretching of alkyl aryl ether (1,022 cm^{-1}) (Figure 3). The interface of silver metal with phenolics, flavonoids, and terpenoids yielded the colloidal suspension [37]. Therefore, the reduction of silver ions by corresponding compounds was identified by FTIR measurement. There is agreement between this study and the reported study regarding the FTIR signals. FTIR vibration signals of silver nanoparticles produced from *Macrotyloma uniflorum* are consistent with the present study [38].

3.3 XRD analysis

XRD is a robust procedure to reveal the structure of a material. It is based on the principle that when X-rays pass through a crystalline sample, they interact with the

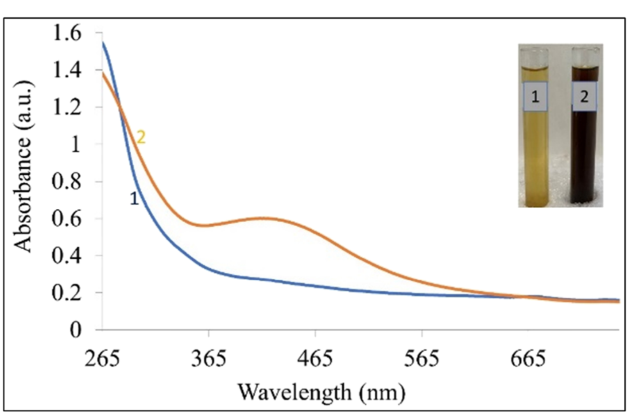

Figure 2: UV-Vis spectrum of extract (1) and AgNPs@Co (2).

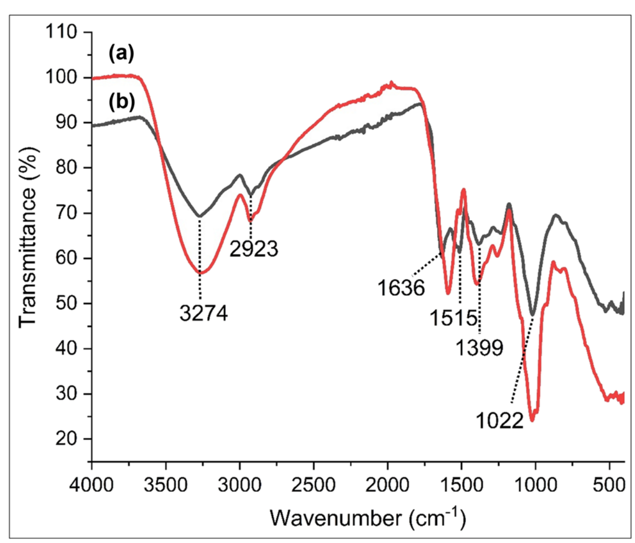

Figure 3: FTIR spectrum of extract (a) and AgNPs@Co (b).

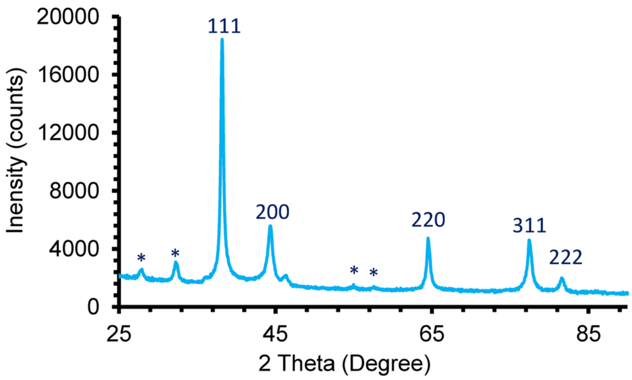

Figure 4: XRD pattern of AgNPs@Co.

atoms and cause the X-rays to disperse in diverse directions. The resulting scattering pattern can be used to reveal information about the alignment of atoms within the crystal lattice. The signals with 2θ at certain angles [38.17°, 44.29°, 57.49°, 77.36°, 81.54°] related to 111, 020, 202, 131, 222 planes, respectively, presenting the face-centered cubic structure that accorded to the JCPDS data card (01-087-0597) [39]. The Scherrer equation (Eq. 2) was employed for calculating the particle size of AgNPs@Co.

$$D = K\lambda/\beta\cos\theta \tag{2}$$

where D is the average diameter, β is the half maximum intensity, θ is the Braggs angle, and λ is the wavelength. The particle size was calculated as 9.7 nm by XRD analysis, but TEM analysis revealed the particle size to be 11.9 nm. This difference was thanks to the variation of the spherical (Figure 4). The lattice constant and d-spacing were calculated as 4.086 and 2.35 Å, respectively. Unassigned peaks may be due to the compounds in the plant extract causing the crystallization independently [40].

3.4 TEM analysis

TEM is an effective method to observe the ultrastructure of materials at very high resolution. It allows scientists to study the morphology, crystal structure, and nanoscale details of various specimens, including biological samples, nanomaterials, polymers, and inorganic materials. TEM analysis of AgNPs@Co was executed, and the sizes were determined ranging from 9 to 30 nm. The average particle size was calculated to be 11.9 nm with spherical (Figure 5). EDX and elemental analysis are shown in Figure 6. The intense signals at 2.3–3.4 keV in the EDX spectrum confirmed the nanostructure formation.

3.5 Zeta potential

ζ potential is a term employed in colloid and surface chemistry to describe the electrostatic potential that exists at the shear plane (the interface) between a solid surface and a surrounding liquid medium. This concept is particularly relevant in the study of colloidal systems, where particles are dispersed in a liquid medium, and the interactions between these particles and the surrounding fluid are crucial to their stability and behavior. The magnitude of the zeta potential is an essential parameter in colloidal systems, as it provides insights into the stability and behavior of the particles. The strong repulsive force between the particles was due to the high zeta potential, leading to increased stability due to reduced chances of aggregation or flocculation. Conversely, low or close-to-zero zeta potentials suggest weaker repulsive forces and may lead to particle agglomeration and settling. The ζ potential of AgNPs@Co was determined as −20.3 mV exhibiting moderate stability

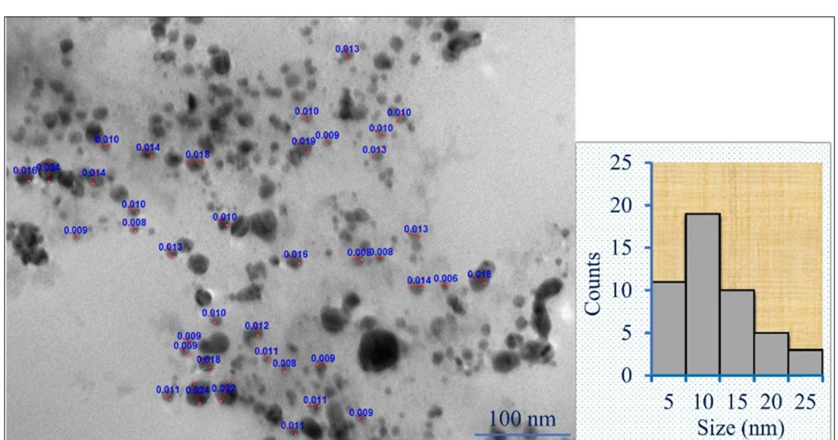

Figure 5: TEM image of AgNPs@Co and particle size distribution.

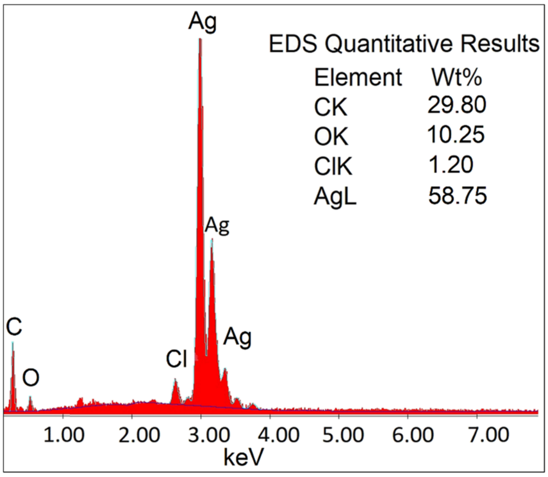

Figure 6: EDX spectrum and elemental analysis of AgNPs@Co.

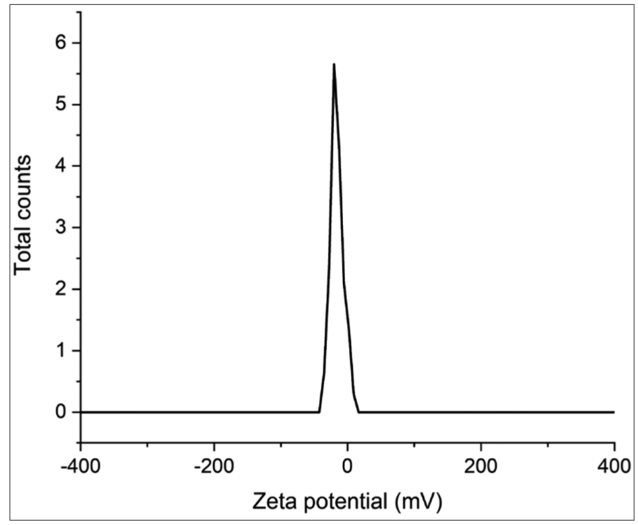

Figure 7: Zeta potential of AgNPs@Co.

(Figure 7). AgNPs synthesized from *Ocimum sanctum* had the zeta potential to be −55 mV that presented the repulsion among AgNPs and their dispersion stability [41].

3.6 Catalytic activity

Degradation of MB using AgNPs is a promising approach for wastewater treatment and environmental remediation. Silver nanoparticles possess unique catalytic properties that can be utilized to efficiently degrade various organic pollutants, including dyes like MB. The positively charged surface of silver nanoparticles attracts the negatively charged MB molecules, leading to their adsorption onto the nanoparticle surface. The adsorbed MB molecules undergo reduction reactions in the presence of AgNPs. The surface of the AgNPs provides catalytic sites that facilitate the transfer of electrons from the dye molecules to the silver nanoparticles. This electron transfer process leads to the degradation of MB into smaller byproducts. The silver nanoparticle-mediated degradation efficacy of MB by sodium borohydride was determined as 71% within 45 min. Still, the degradation was found to be 19% absent AgNPs@Co. The degradation mechanism was determined by Langmuir-Hinshelwood [42]. Hydrogen and electrons were released by sodium borohydride. After adding AgNPs@Co into the reaction medium, adsorption of both reactants on the catalytic surface took place. Moreover, nanoparticles facilitate the electron transfer, resulting in the degradation of MB. Silver nanoparticles synthesized from natural sources have been reported to reveal considerable catalytic activity on MB (Figure 8).

Degradation kinetics and percentage degradation of MB dye were calculated (Figure 9). $\mathrm{Ln}(A/A_0)$ with a significant R^2 value of 0.9886 presenting first-order kinetics according to Eq. 3. The rate constant was calculated as 0.0258 min^{-1}.

$$\mathrm{Ln}\frac{A}{A_0} = kt \quad (3)$$

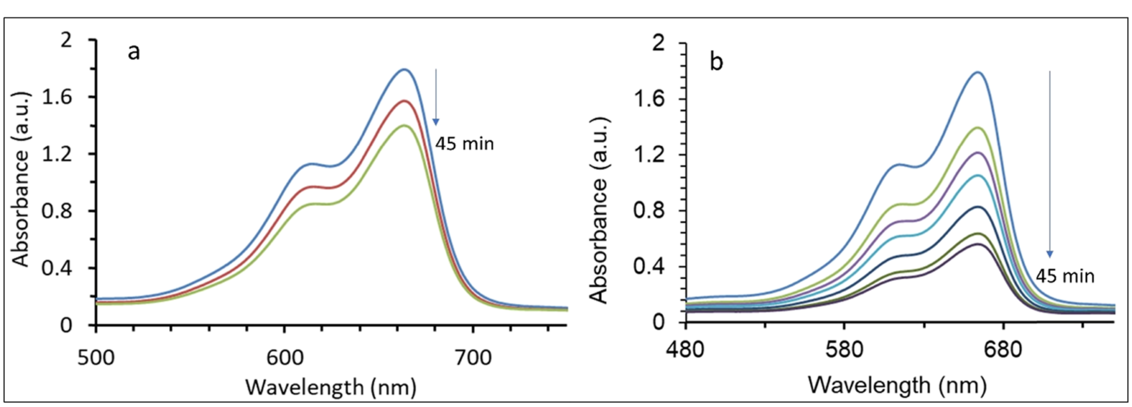

Figure 8: Catalytic degradation of MB by $NaBH_4$ (a) reaction mixture without AgNPs@Co and (b) reaction mixture with AgNPs@Co.

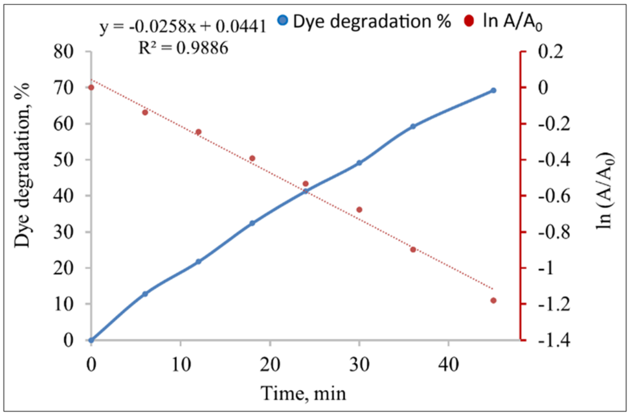

Figure 9: Photocatalytic degradation kinetics.

$$\%\text{Degradation} = \frac{A_0 - A}{A_0} \times 100 \quad (4)$$

where A_0 and A are the initial and final absorbance of dye and k is the first-order rate constant.

AgNPs synthesized from some plant extracts have been reported to exhibit good catalytic activities. The aqueous beetroot extract was used for silver nanoparticle synthesis, and they displayed good catalytic activity [43].

3.7 Antioxidant activity

Antioxidant activity of nanoparticles and extract was performed using various assays, including superoxide radical ($O_2^{-\bullet}$) scavenging, DPPH$^\bullet$ scavenging, and ABTS$^{\bullet+}$ radical cation scavenging tests. The AgNPs@Co and extract revealed considerable DPPH$^\bullet$ activity with the values of 9.3 ± 0.2 and 11.2 ± 0.6 in comparison to the standard BHT (8.7 ± 0.2). The same trend was observed for the ABTS$^{\bullet+}$ effect. The activity of AgNPs@Co was detected to be higher (7.2 ± 0.1) than the extract (9.4 ± 0.4) significantly. Regarding the superoxide radical scavenging effect, it was determined that the nanoparticles and the extract had an excellent superoxide radical scavenging effect. The effect of AgNPs@Co was detected better than the extract with the values of 10.3 ± 0.1 and 12.5 ± 0.3, respectively, in comparison to the BHT (11.4 ± 0.2). The results were presented as inhibitory concentration (IC_{50}). The percentage of radical scavenging activity was calculated, and half-maximal IC_{50} was calculated *via* a calibration curve.

The nanoparticles synthesized using medicinal plants were reported to exhibit important antioxidant activity. AgNPs obtained from *Echinacea purpurea* displayed excellent antioxidant activity [44]. In another study, oleuropein was used for silver nanoparticle synthesis, and nanoparticles displayed significant antioxidant activity [45]. *Origanum majorana*-mediated synthesis of silver nanoparticles was achieved, and they showed excellent antioxidant activity [46]. The particle size has an important effect on biological activities. It has been reported that silver nanoparticles with small particle sizes show high biological activity [47]. Therefore, in this study, synthesized small particle sizes of silver nanoparticles demonstrated excellent antioxidant activity (Figure 10).

4 Conclusion

A novel eco-friendly, cost-effective, fast, scalable method for silver nanoparticle synthesis using *C. orientalis* flowers was developed. The natural compounds in the corresponding

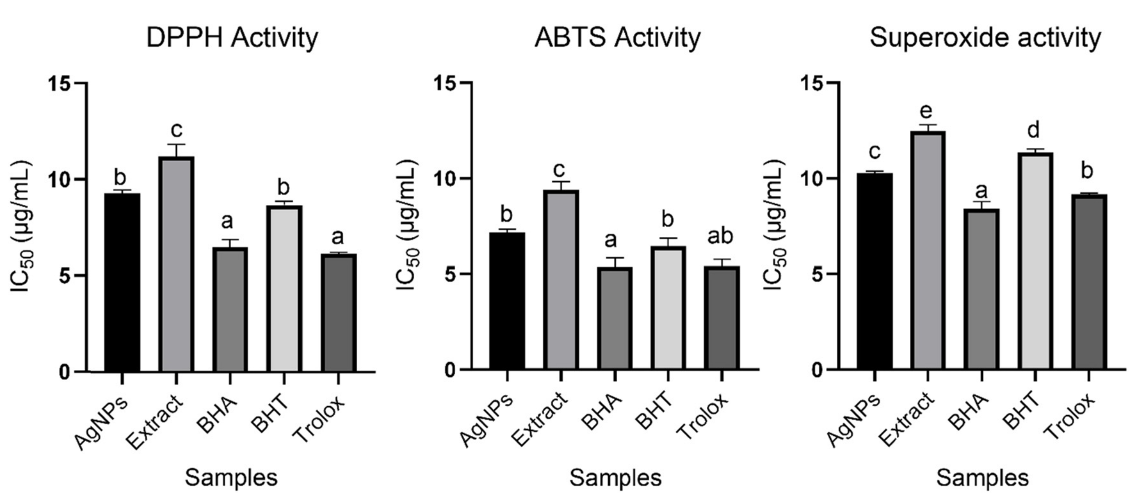

Figure 10: Antioxidant activity of nanoparticles and extract.

plant extract behaved as stabilizing and reducing agents. The green synthesized AgNPs@Co were interpreted by UV-Vis, FTIR, EDX, TEM, and XRD analyses. *C. orientalis* has great medicinal importance due to its bioactive compound contents. Thus, AgNPs@Co might be a pharmaceutical drug candidate for diseases caused by oxidative stress. Moreover, AgNPs@Co may be useful for developing new and more potent antioxidants. The catalytic effect of green synthesized nanostructures depends on their composition and size. The high surface-to-volume ratio reveals higher activity. AgNPs@Co showed good catalytic activity for dye reduction and removal. AgNPs@Co acted as an effective catalyst for the reduction of MB by $NaBH_4$.

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Data availability statement: All data generated or analyzed during this study are included in this published article.

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