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Microwave-assisted green synthesis of gold nanoparticles using MAE-derived aqueous *Rubus caesius* L. Fruit extract: characterization and UV–Vis spectral stability

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Abstract

Gold nanoparticles (AuNPs) were synthesized through a microwave-assisted green route using an MAE-derived aqueous *Rubus caesius* L. fruit extract as a natural reducing and stabilizing medium. The effects of precursor concentration, extract volume, and microwave power were systematically evaluated within the tested experimental range. The combination of 0.075 mM HAuCl₄·0.3 H₂O, 0.4 mL fruit extract, and 180 W microwave irradiation produced the most distinct and symmetric surface plasmon resonance response. FT-IR analysis suggested that oxygen-containing functional groups in the extract may have contributed to Au³⁺ reduction and AuNP surface stabilization. TEM analysis revealed spherical, triangular, and hexagonal nanoparticles with an average core size of 11.6 ± 3.2 nm. DLS showed a hydrodynamic diameter of 71.92 ± 4.12 nm and a polydispersity index of 0.425, while the near-neutral zeta potential indicated limited colloidal stability. UV–Vis monitoring showed that the RC-AuNPs largely retained their spectral characteristics for up to 45 days. The synthesized nanoparticles exhibited a core size comparable to previously reported *Rubus*-mediated AuNPs. The specific contribution of this study is the use of an MAE-derived aqueous *R. caesius* L. fruit extract together with the systematic evaluation of key synthesis parameters affecting AuNP formation, optical response, and colloidal behavior.

Keywords *Rubus caesius* L., Green nanotechnology, Microwave-assisted extraction, Plant-mediated synthesis, Surface plasmon resonance, AuNP

1 Introduction

In recent years, the synthesis of gold nanoparticles (AuNPs) has increasingly shifted from conventional chemical routes toward greener and more sustainable approaches. Traditional chemical methods, although effective in controlling nanoparticle formation, often rely on toxic reducing agents, stabilizers, or organic solvents that may limit their environmental compatibility and biomedical applicability. In contrast, plant-mediated synthesis uses naturally derived materials and bioactive phytochemicals, including



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phenolics, flavonoids, tannins, sugars, proteins, and organic acids, which may participate in metal-ion reduction and nanoparticle stabilization [1]. By serving multiple roles within a single reaction system, these phytochemicals make plant-based synthesis routes attractive in terms of simplicity, reduced toxicity, and lower environmental burden. Phytochemical-rich plant extracts have been used as effective reducing and stabilizing systems for the green synthesis and functional application of metallic nanoparticles, including AuNPs and Au–Ag-based nanostructures [2, 3]. Within this context, microwave-assisted green synthesis has emerged as a rapid, energy-efficient, and relatively controlled approach for AuNP formation using plant-derived extracts and phytochemical-rich reducing systems [4–7]. Recent studies have further demonstrated the applicability of catechin-based and fruit-extract-mediated systems for the sustainable green synthesis of AuNPs, emphasizing the influence of phytochemical composition and synthesis conditions on nanoparticle formation and physicochemical properties [4, 8]. Plant-mediated synthesis may also offer lower cytotoxicity, improved biocompatibility, and potential applications in biomedical, catalytic, sensing, and environmental fields [9]. However, the exact composition of plant extracts may strongly influence nanoparticle nucleation, growth, morphology, and colloidal behavior. Therefore, each plant-based synthesis system should be evaluated individually rather than considered interchangeable with other botanical sources. Within this broader context, phytochemical-rich fruits have emerged as promising bioresources for sustainable nanomaterial production. Fruits contain diverse secondary metabolites, including phenolics, flavonoids, anthocyanins, organic acids, and sugars, which may act as reducing, chelating, and capping agents during nanoparticle synthesis [10, 11]. Underutilized or less extensively studied fruit species may possess distinct phytochemical profiles that result in different nanoparticle characteristics and therefore warrant further investigation.

In parallel with the development of plant-mediated synthesis strategies, microwave-assisted extraction (MAE) has gained increasing attention as an efficient green extraction technology for recovering bioactive compounds from plant materials. Compared with conventional extraction techniques, MAE can offer important advantages, including reduced extraction time, lower solvent consumption, improved extraction efficiency, rapid heating, and enhanced disruption of the plant matrix. Since the biochemical composition of an extract is one of the key determinants of nanoparticle nucleation, growth, morphology, and stabilization, the extraction method may indirectly influence the physicochemical properties of the resulting nanomaterial. In this regard, combining MAE-based extract preparation with green nanoparticle synthesis integrates two sustainability-oriented processes within a single experimental framework. However, the present study did not include a direct chemical comparison between the MAE-derived extract and an extract prepared using a conventional extraction method. Therefore, the MAE-derived extract cannot be claimed to possess a definitively different phytochemical composition based on the present findings. Rather, MAE is considered a rapid extraction approach that may promote plant-tissue disruption and the recovery of water-soluble phytochemicals while reducing extraction time and prolonged thermal exposure. Previous studies have demonstrated the feasibility of *Rubus*-mediated AuNP synthesis using different species and extract systems. For example, Demirbaş et al., (2019) reported the synthesis of long-term stable AuNPs using *R. idaeus* and *R. fruticosus* fruit extracts, with particle sizes generally ranging from approximately 10 to 15 nm [12]. Similarly,

Gurrola-Rios et al., [13] synthesized AuNPs using anthocyanins obtained from *R. palm-eri* and reported an average particle size of approximately 13 nm. These studies confirm the potential of Rubus-derived phytochemicals as reducing and stabilizing agents in AuNP synthesis. However, they did not investigate *R. caesius* L. fruit extract, use an MAE-derived aqueous fruit extract, or systematically evaluate the combined effects of $\text{HAuCl}_4 \cdot 0.3 \text{ H}_2\text{O}$ concentration, fruit extract volume, and microwave power on nanoparticle formation and colloidal behavior. Therefore, the present study differs from previous Rubus-mediated AuNP studies by focusing on MAE-derived *R. caesius* L. fruit extract and by establishing a microwave-assisted comparative evaluation framework for examining the optical and colloidal properties of the synthesized AuNPs. Although plant-mediated AuNP synthesis is already well represented in the literature, the contribution of the present study lies in combining *Rubus caesius* L., a relatively underexplored plant source in this field, with MAE-based aqueous extraction and subsequent microwave-assisted synthesis of AuNPs. Rather than claiming a fundamentally new concept in green nanotechnology, the present work provides additional evidence for a specific and insufficiently explored plant-extract system. To the best of our knowledge, the use of an MAE-derived aqueous extract of *R. caesius* L. for the synthesis of AuNPs has not been clearly documented in previous studies. Thus, the significance of this work arises from extending the diversity of plant-based systems available for green AuNP production through a defined and systematically evaluated experimental approach. More specifically, the study investigates how key synthesis variables, including $\text{HAuCl}_4 \cdot 0.3 \text{ H}_2\text{O}$ concentration, extract volume, and microwave power, influence nanoparticle formation, SPR characteristics, and colloidal behavior. This process-oriented evaluation helps clarify the relationship between the selected experimental conditions and the physicochemical properties of the nanoparticles. The study also evaluates the capacity of the MAE-derived extract to function as a reducing and capping medium during microwave-assisted AuNP synthesis.

Based on this rationale, the present study aimed to evaluate the potential of the MAE-derived aqueous fruit extract of *Rubus caesius* L. as a green reducing and stabilizing medium for the microwave-assisted synthesis of AuNPs. The study specifically focused on determining the effects of $\text{HAuCl}_4 \cdot 0.3 \text{ H}_2\text{O}$ concentration, fruit extract volume, and microwave power on the SPR response, nanoparticle formation, and colloidal behavior of the synthesized AuNPs. In addition, the study aimed to identify the most favorable synthesis condition within the investigated experimental range for producing AuNPs with nanoscale core dimensions and largely retained UV–Vis spectral characteristics during storage. It was hypothesized that the MAE-derived aqueous fruit extract of *Rubus caesius* L. contains sufficient phytochemical constituents to reduce Au^{3+} ions and contribute to the stabilization of the resulting AuNPs. It was further hypothesized that increasing microwave power would accelerate nanoparticle formation by enhancing reduction kinetics, while an appropriate balance between $\text{HAuCl}_4 \cdot 0.3 \text{ H}_2\text{O}$ concentration and fruit extract volume would result in a more defined SPR response. Conversely, excessive $\text{HAuCl}_4 \cdot 0.3 \text{ H}_2\text{O}$ concentration or fruit extract volume was expected to produce broader spectral profiles and less controlled nanoparticle formation.

2 Materials and methods

2.1 Materials

All reagents and chemicals used in this study were of analytical grade and used without further purification. Chloroauric acid trihydrate ($\text{HAuCl}_4 \cdot 0.3 \text{ H}_2\text{O}$) was purchased from a certified supplier and used as the gold precursor for nanoparticle synthesis. Deionized water was used in all experimental procedures. Glassware was cleaned with nitric acid and rinsed thoroughly with deionized water prior to use.

2.2 Plant collection and preparation of extract

Rubus caesius L. specimens were collected in August 2024 from the Alazlı region of Düzköy, Trabzon, Türkiye (Voucher No: A7; 1825 m; 30.VIII.2024). The taxonomic identification of the plant material was carried out by Prof. Dr. Salih Terzioğlu. A voucher specimen was deposited in the Herbarium of the Department of Forest Engineering, Faculty of Forestry, Karadeniz Technical University, Trabzon, Türkiye, under the herbarium number KATO 24,461. For aqueous fruit extraction, 20 g of cleaned fresh fruits was mixed with 80 mL of deionized water and continuously stirred using a magnetic stirrer at 25 ± 2 °C for 240 min to facilitate the diffusion of bioactive phytochemicals from the fruit material into the aqueous medium. The mixture was subsequently kept in the dark at 4 °C for 24 h to improve extraction efficiency. Thereafter, microwave-assisted extraction was performed using a domestic microwave oven operated at 900 W for 8 min. This condition was selected to promote fruit tissue disruption and enhance the release of phytochemical constituents into the aqueous medium while limiting prolonged thermal exposure. After microwave treatment, the aqueous fruit extract was allowed to cool naturally to room temperature before filtration. No external temperature-control system was used during microwave extraction.

2.3 Synthesis of gold nanoparticles (AuNPs)

Gold nanoparticles (AuNPs) were synthesized through a green and sustainable route using the aqueous fruit extract of *Rubus caesius* L., which served simultaneously as a reducing and stabilizing medium. Chloroauric acid trihydrate ($\text{HAuCl}_4 \cdot 0.3 \text{ H}_2\text{O}$) solutions were freshly prepared in deionized water at three different concentrations (0.05, 0.075, and 0.1 mM). For each experiment, varying volumes of the fruit extract (0.2, 0.4, and 0.8 mL) were added dropwise to 50 mL of the gold precursor solution under continuous stirring at room temperature. The reaction mixtures were subsequently subjected to microwave irradiation in a domestic microwave oven at two power levels (90 and 180 W) to promote nanoparticle formation. Under the selected synthesis condition (0.075 mM $\text{HAuCl}_4 \cdot 0.3 \text{ H}_2\text{O}$, 0.4 mL fruit extract, and 180 W), the pH of the reaction medium was measured in three independent synthesis experiments and ranged from 6.8 to 7.2. The progression of the reaction was evidenced by a distinct color change from pale yellow to purple–pink, indicating the reduction of Au^{3+} ions to metallic Au^0 by the phytochemical constituents of the fruit extract. This visual transformation is characteristic of the surface plasmon resonance (SPR) phenomenon associated with AuNP formation. Following synthesis, the AuNP suspensions were centrifuged at 15,000 rpm for 15 min prior to characterization. The resulting AuNP samples were stored at 4 °C until further characterization. A negative control experiment was performed by exposing an aqueous $\text{HAuCl}_4 \cdot 0.3 \text{ H}_2\text{O}$ solution without *Rubus caesius* L. fruit extract to the

same microwave irradiation conditions used for AuNP synthesis. The control sample was visually monitored and analyzed by UV–Vis spectroscopy to verify the absence of spontaneous AuNP formation.

2.4 Characterization of gold nanoparticles

The biosynthesized gold nanoparticles (AuNPs) were characterized by UV–Vis spectroscopy, Fourier transform infrared (FT-IR) spectroscopy, dynamic light scattering (DLS) coupled with zeta potential measurements, and transmission electron microscopy (TEM) to evaluate their optical properties, surface chemistry, colloidal behavior, and morphological characteristics. AuNP formation was initially evaluated by UV–Vis spectrophotometry using a Shimadzu UVP-1240 UV–Vis spectrophotometer (Shimadzu, Japan). Absorption spectra were recorded over the wavelength range of 300–800 nm to identify the characteristic surface plasmon resonance (SPR) band associated with AuNP formation. FT-IR analysis was performed using a PerkinElmer FT-IR spectrometer (PerkinElmer) in the spectral range of 4000–550 cm^{-1} to determine the functional groups present in the aqueous *Rubus caesius* L. fruit extract that may contribute to the reduction of Au^{3+} ions and the surface stabilization of the synthesized nanoparticles. The FT-IR results were also used to evaluate possible interactions between phytochemical constituents from the fruit extract and the AuNP surface.

The hydrodynamic diameter, polydispersity index, and zeta potential of the AuNP suspensions were measured using a Malvern Zetasizer Nano ZSP system (Malvern Instruments) to assess particle size distribution, surface charge, and colloidal behavior in aqueous medium. In addition, the morphology and particle size of the synthesized AuNPs were examined by transmission electron microscopy using a Hitachi HT-7700 TEM instrument (Hitachi, Japan). TEM analysis provided images for evaluating nanoparticle shape, core size distribution, and morphological characteristics at the nanoscale.

3 Results and discussion

The progress of AuNP formation was initially monitored through the visible color change occurring in the reaction medium. As illustrated in Fig. 1, the mixture containing *Rubus caesius* L. extract and aqueous $\text{HAuCl}_4 \cdot 0.3 \text{H}_2\text{O}$ solution displayed a pale yellow color before microwave exposure (Fig. 1b), reflecting the presence of unreduced Au^{3+} ions in the precursor solution. After microwave irradiation, the solution rapidly turned purple–pink (Fig. 1c and d), providing clear visual evidence for the reduction of Au^{3+} to Au^0 and the subsequent formation of colloidal gold nanoparticles. This characteristic



Fig. 1 Visual observation of AuNP formation using the aqueous fruit extract of *Rubus caesius* L.: (a) fresh fruits used for extract preparation; (b) initial reaction mixture containing $\text{HAuCl}_4 \cdot 0.3 \text{H}_2\text{O}$ solution and the aqueous fruit extract before microwave irradiation; (c, d) reaction mixture after microwave treatment, showing the development of a distinct purple–pink color that visually suggests the formation of gold nanoparticles (AuNPs)

color transition is attributed to the surface plasmon resonance (SPR) of AuNPs and is generally associated with the formation of colloidal gold nanoparticles, particularly those containing predominantly spherical particles [14]. The rapid development of the purple–pink coloration further indicates that microwave irradiation effectively promoted the reduction process and accelerated nanoparticle nucleation. This observation is consistent with previous reports demonstrating that microwave-assisted green synthesis enhances reaction kinetics by improving energy transfer, intensifying molecular collisions, and enabling rapid and homogeneous heating, thereby facilitating faster nucleation and controlled nanoparticle growth [4, 7, 15, 16].

The influence of metal ion concentration on AuNP formation was systematically evaluated by UV–Vis spectroscopy using a plant extract-mediated green synthesis route. In these experiments, the extract volume (0.2 mL) and microwave power (90 W) were kept constant, while the temporal evolution of the absorbance spectra was monitored at four different precursor concentrations (0.05, 0.075, 0.1, and 1 mM). As shown in Fig. 2a, the reaction system containing 0.05 mM $\text{HAuCl}_4 \cdot 0.3 \text{ H}_2\text{O}$ exhibited a weak SPR band centered around 540 nm, with only a slight increase in intensity over time. This behavior suggests that the low precursor concentration provided an insufficient number of Au^{3+} ions to sustain effective nucleation and subsequent nanoparticle growth, thereby limiting overall AuNP formation. Increasing the concentration to 0.075 mM (Fig. 2b) led to a more pronounced SPR band and higher absorbance values, indicating enhanced nanoparticle generation. Among the tested precursor concentrations, 0.075 mM $\text{HAuCl}_4 \cdot 0.3 \text{ H}_2\text{O}$ (Fig. 2b) produced the most favorable spectral profile and was therefore selected for subsequent experiments. From the 10th minute onward, a well-defined, intense, and symmetric SPR band was observed, reaching maximum intensity at 20 min

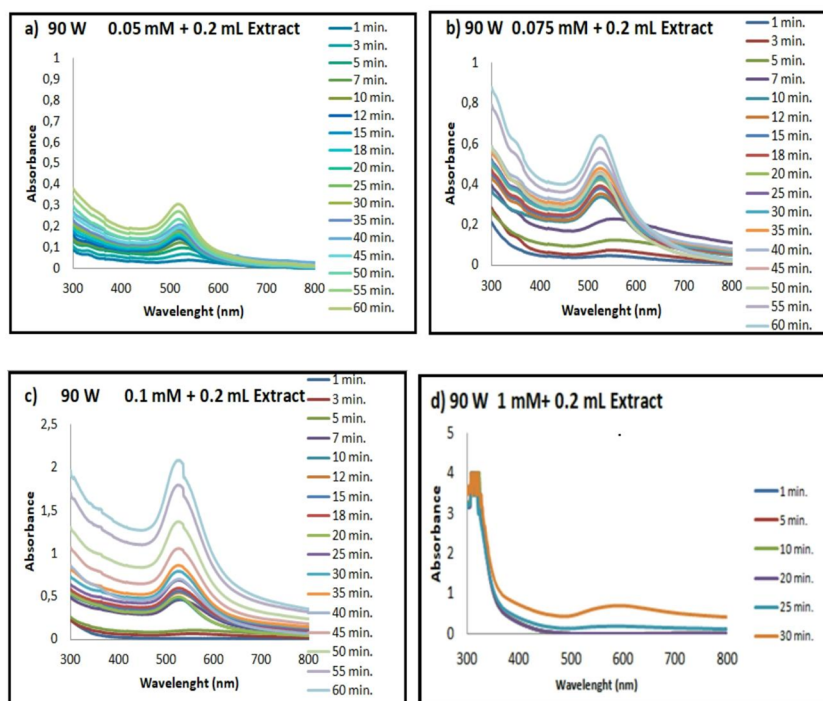


Fig. 2 Surface plasmon resonance (SPR) absorption spectra of gold nanoparticles (AuNPs) synthesized using different $\text{HAuCl}_4 \cdot 0.3 \text{ H}_2\text{O}$ concentrations (0.05, 0.075, 0.1, and 1 mM), 0.2 mL aqueous *Rubus caesius* L. fruit extract, and 90 W microwave irradiation at different reaction times

and remaining clearly detectable throughout the monitoring period. The emergence of such a distinct and well-defined SPR signal suggests a balanced reduction environment in which the availability of Au^{3+} ions and phytochemical reducing agents was well matched, enabling controlled nucleation and subsequent growth of relatively uniform nanoparticles. Moreover, the persistence of the SPR band up to 60 min indicates that the spectral characteristics of the system were largely retained during the monitored reaction period, without substantial deterioration of the SPR response. In contrast, the highest precursor concentration (1 mM; Fig. 2d) resulted in a markedly different spectral behavior. Although the overall absorbance increased substantially, particularly in the 300–350 nm region, the characteristic SPR band of AuNPs became indistinct or completely masked. This spectral pattern points to an uncontrolled reaction pathway, in which excessive Au^{3+} ion concentration likely promoted rapid and non-uniform particle growth, resulting in aggregation, precipitation, or the formation of polydisperse structures. Under these conditions, the fixed extract volume was evidently insufficient to provide the reducing and stabilizing capacity required to maintain controlled nanoparticle synthesis, leading to oversaturation of the reaction medium. Similar concentration-dependent behavior has been reported previously, where low precursor concentrations were associated with limited nucleation efficiency, whereas excessively high concentrations favored uncontrolled particle growth and agglomeration [17]. Overall, the UV–Vis results clearly demonstrate that metal ion concentration plays a decisive role in regulating nanoparticle nucleation, growth, and colloidal stability. Within the experimental range investigated, 0.075 mM was identified as the most suitable precursor concentration, as it provided the most balanced reaction environment for the reproducible formation of AuNPs with a well-defined SPR response. This interpretation was further supported by the visual appearance of the suspensions, where the 1 mM sample displayed a darker and less homogeneous color compared with the lower precursor concentrations (Fig. 3).

As the precursor concentration increased, the color of the suspensions became progressively darker. In particular, the 1 mM sample exhibited a darker and less homogeneous appearance, supporting the UV–Vis results and suggesting less controlled nanoparticle formation, possibly associated with aggregation, precipitation, or increased polydispersity.

The effect of extract volume on AuNP formation was further investigated by UV–Vis spectroscopy using reaction systems containing a fixed gold precursor concentration of 0.075 mM $\text{HAuCl}_4 \cdot 0.3 \text{ H}_2\text{O}$ and two different volumes of aqueous *Rubus caesius* L. fruit extract (0.4 and 0.8 mL) under constant microwave irradiation at 90 W. The time-dependent SPR spectra recorded over 1–60 min are presented in Fig. 4. As shown in

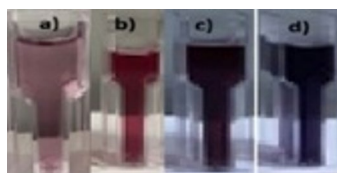


Fig. 3 Visual appearance of gold nanoparticle (AuNP) suspensions synthesized using 0.2 mL aqueous *Rubus caesius* L. fruit extract and different $\text{HAuCl}_4 \cdot 0.3 \text{ H}_2\text{O}$ concentrations under 90 W microwave irradiation: (a) 0.05 mM, (b) 0.075 mM, (c) 0.1 mM, and (d) 1 mM

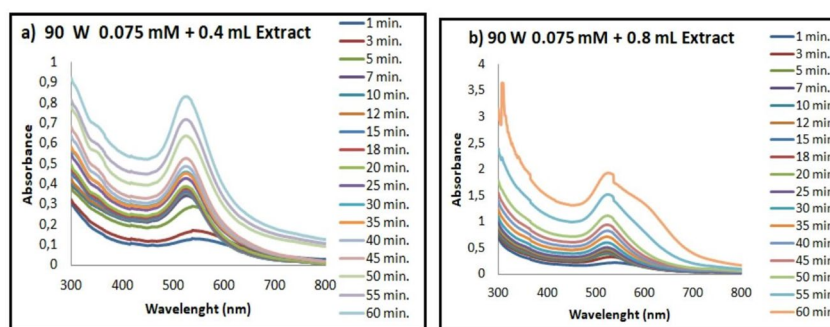


Fig. 4 Surface plasmon resonance (SPR) absorption spectra of gold nanoparticles (AuNPs) synthesized using 0.075 mM HAuCl₄·0.3 H₂O with (a) 0.4 mL and (b) 0.8 mL aqueous *Rubus caesius* L. fruit extract under 90 W microwave irradiation at different reaction times

Fig. 4a, the system containing 0.4 mL extract exhibited a distinct and relatively narrow SPR band centered at approximately 524–528 nm, with a gradual increase in intensity up to about 25–30 min. This spectral behavior suggests a more favorable nucleation and growth process, indicating that the amount of phytochemicals present in the fruit extract was sufficient to reduce Au³⁺ ions and contribute to the stabilization of the resulting nanoparticles without promoting excessive aggregation. The well-defined SPR profile also suggests the formation of relatively uniform AuNPs under these conditions. In contrast, increasing the extract volume to 0.8 mL (Fig. 4b) resulted in a different spectral response. Although an SPR band was still observed, the spectra showed markedly higher absorbance values, particularly in the 300–400 nm region during the early stages of the reaction, together with a broader and less distinct SPR profile. This behavior suggests that the excess amount of phytochemicals in the fruit extract may have accelerated the initial reduction process, leading to rapid nucleation but a less controlled subsequent growth stage. As a result, the system likely favored the formation of nanoparticles with broader size distribution and reduced uniformity. The broadened SPR band may also reflect enhanced interparticle interactions or partial aggregation, possibly induced by the high concentration of polyphenolic and other bioactive constituents in the extract.

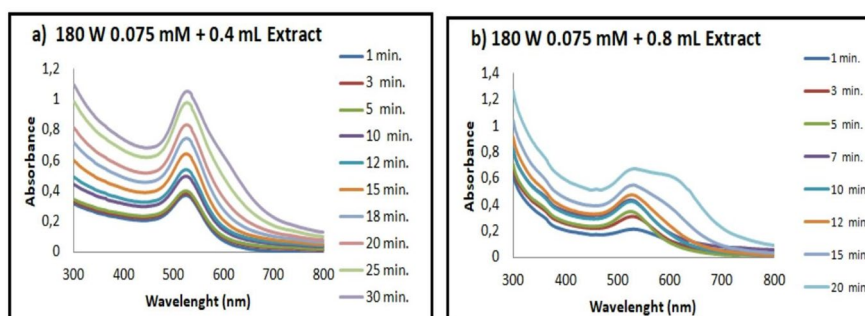
These findings suggest that, although increasing extract volume enhances the availability of reducing and capping agents, excessive phytochemical content may disrupt the balance between nucleation, growth, and stabilization, ultimately reducing colloidal uniformity [5]. Overall, the UV–Vis results demonstrate that extract volume is a critical parameter in regulating the biosynthesis of AuNPs. Within the conditions examined, the use of 0.4 mL aqueous *Rubus caesius* L. fruit extract in combination with 0.075 mM HAuCl₄·0.3 H₂O provided a more favorable reaction environment, yielding a sharper and more consistent SPR response.

All synthesis experiments were performed in triplicate, and the reproducibility of AuNP formation was evaluated by calculating the mean ± standard deviation of the SPR maximum wavelength (λ_{max}) and absorbance intensity for each tested condition. As summarized in Table 1, the condition containing 0.075 mM HAuCl₄·0.3 H₂O and 0.4 mL aqueous *Rubus caesius* L. fruit extract produced a distinct and reproducible SPR response with low variation in both λ_{max} and absorbance intensity. In contrast, the 1 mM HAuCl₄·0.3 H₂O condition produced a broad and indistinct SPR band, for which λ_{max} could not be reliably determined. These findings support the selection of 0.075

Table 1 Reproducibility of UV–Vis/SPR responses of RC-AuNPs synthesized under different experimental conditions

Synthesis condition	λ_{max} (nm)	Absorbance intensity	n
90 W, 0.05 mM HAuCl ₄ 0.3 H ₂ O, 0.2 mL extract	525.00 ± 1.00	0.29 ± 0.01	3
90 W, 0.075 mM HAuCl ₄ 0.3 H ₂ O, 0.2 mL extract	526.33 ± 0.58	0.64 ± 0.01	3
90 W, 0.1 mM HAuCl ₄ 0.3 H ₂ O, 0.2 mL extract	527.00 ± 2.65	2.05 ± 0.05	3
90 W, 1 mM HAuCl ₄ 0.3 H ₂ O, 0.2 mL extract	595.00 ± 5.00	0.68 ± 0.02	3
90 W, 0.075 mM HAuCl ₄ 0.3 H ₂ O, 0.4 mL extract	526.33 ± 0.58	0.83 ± 0.01	3
90 W, 0.075 mM HAuCl ₄ 0.3 H ₂ O, 0.8 mL extract	526.33 ± 0.58	1.90 ± 0.01	3
180 W, 0.075 mM HAuCl ₄ 0.3 H ₂ O, 0.4 mL extract	527.00 ± 1.00	1.05 ± 0.01	3
180 W, 0.075 mM HAuCl ₄ 0.3 H ₂ O, 0.8 mL extract	529.67 ± 0.58	0.67 ± 0.01	3

Values are presented as mean ± SD

**Fig. 5** Surface plasmon resonance (SPR) absorption spectra of gold nanoparticles (AuNPs) synthesized using 0.075 mM HAuCl₄0.3 H₂O with (a) 0.4 mL and (b) 0.8 mL aqueous *Rubus caesius* L. fruit extract under 180 W microwave irradiation at different reaction times

mM HAuCl₄0.3 H₂O and 0.4 mL aqueous *Rubus caesius* L. fruit as the most favorable synthesis condition among the tested experimental range.

The formation of AuNPs using 0.075 mM HAuCl₄0.3 H₂O and two different volumes of aqueous *Rubus caesius* L. fruit extract (0.4 and 0.8 mL) under microwave irradiation at 180 W was evaluated by UV–Vis spectroscopy. The time-dependent SPR absorption spectra recorded over 1–30 min are presented in Fig. 5. For comparison, Fig. 4a shows the UV–Vis spectra of AuNPs synthesized under 90 W microwave power using the same precursor concentration and 0.4 mL extract over a longer reaction period (1–60 min). Under the lower microwave power condition, the absorbance increased gradually and the SPR band appeared at approximately 524–528 nm, indicating a relatively slower reaction rate and progressive nanoparticle formation. By contrast, increasing the microwave power to 180 W markedly accelerated the synthesis process, as evidenced by the earlier development of a more intense and better-defined SPR band. As shown in Fig. 5a, the system containing 0.4 mL extract at 180 W exhibited a sharp and symmetric SPR peak centered at approximately 524–528 nm, which became clearly distinguishable between 10 and 15 min and reached a maximum absorbance of about 1.2. This spectral behavior indicates rapid and well-defined nanoparticle formation under the tested conditions, suggesting that constituents of the fruit extract may have contributed to Au³⁺ reduction and surface stabilization under increased microwave power. The narrow and symmetric nature of the SPR band further suggests a more uniform optical response under these conditions.

In contrast, the use of 0.8 mL extract under 180 W microwave power (Fig. 5b) resulted in a broader and less well-defined SPR profile. Although the absorbance reached its maximum at around the 10th minute, the strong background absorption observed in the 300–400 nm region suggests that the reaction environment became less controlled, likely due to the excessive concentration of phytochemicals in the medium. Such conditions may have promoted overly rapid reduction, irregular particle growth, and increased interparticle interactions, ultimately resulting in broader size distribution and possible aggregation. Thus, while higher extract volume accelerated the initial formation process, it may also have reduced nanoparticle uniformity. Overall, the results demonstrate that microwave power and extract volume act together in determining the SPR characteristics of the synthesized AuNPs. Under 180 W irradiation, the use of 0.4 mL aqueous *Rubus caesius* L. fruit extract provided the most favorable spectral response, characterized by a sharp SPR band and rapid formation kinetics. In contrast, increasing the extract volume to 0.8 mL led to spectral broadening and signs of less controlled nanoparticle formation. These findings highlight that, in addition to precursor concentration, careful evaluation and adjustment of microwave power and extract volume are important for reproducible AuNP synthesis. However, although the UV–Vis results indicated that 180 W microwave irradiation accelerated AuNP formation and produced a sharper SPR response compared with 90 W, TEM characterization was focused on the condition selected based on the UV–Vis/SPR results.

All synthesis experiments were performed in triplicate, and the reproducibility of AuNP formation was evaluated by calculating the mean $\pm$ standard deviation of the SPR maximum wavelength (λ_{max}) and absorbance intensity for each tested condition. As summarized in Table 1, the condition consisting of 0.075 mM $\text{HAuCl}_4 \cdot 0.3 \text{ H}_2\text{O}$, 0.4 mL aqueous *Rubus caesius* L. fruit extract, and 180 W microwave irradiation produced the most favorable and reproducible SPR response, with a λ_{max} of 527.00 ± 1.00 nm and an absorbance intensity of 1.05 ± 0.01 ($n = 3$). Therefore, this condition was selected for subsequent FT-IR, TEM, DLS, zeta potential, and UV–Vis spectral stability analyses.

Under the selected synthesis condition, the pH of the reaction medium ranged from 6.8 to 7.2 across three independent synthesis experiments, indicating near-neutral reaction conditions. Previous research has demonstrated that pH can influence the colloidal behavior of *Rubus*-mediated AuNPs. Demirbaş et al. (2019) investigated pH values of 2, 5, 8, and 13 and reported that *R. idaeus*-mediated AuNPs remained dispersed at pH 5 and above, whereas *R. fruticosus*-mediated AuNPs were reported to remain dispersed across all the investigated pH conditions. Thus, the near-neutral pH range observed in the present study lies between the pH 5 and 8 conditions previously investigated for other *Rubus*-based AuNP systems.

The UV–Vis spectral behavior of RC-AuNPs was evaluated during storage. The UV–Vis spectra showed no substantial changes during the first 45 days, suggesting that the optical characteristics of the suspension were largely preserved over this period (Fig. 6). In contrast, a decrease in absorbance intensity was observed on day 60, indicating reduced UV–Vis spectral stability. However, it should be noted that this assessment was based only on UV–Vis spectral changes. The observed retention of the SPR band should therefore be interpreted as UV–Vis spectral stability.

FT-IR spectroscopy was used to examine the functional groups potentially associated with the synthesis and surface stabilization of RC-AuNPs. Figures 7(a) and 7(b) show

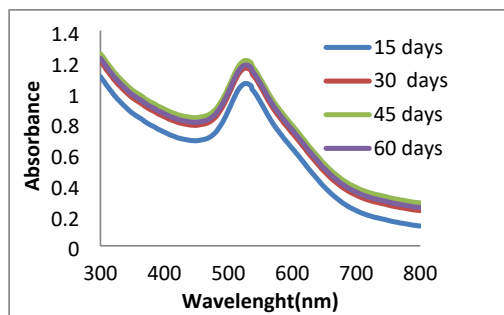


Fig. 6 UV-Vis spectral stability of *Rubus caesius* L.-mediated gold nanoparticles (RC-AuNPs) synthesized using 0.075 mM $\text{HAuCl}_4 \cdot 0.3 \text{H}_2\text{O}$, 0.4 mL aqueous fruit extract, and 180 W microwave irradiation during storage

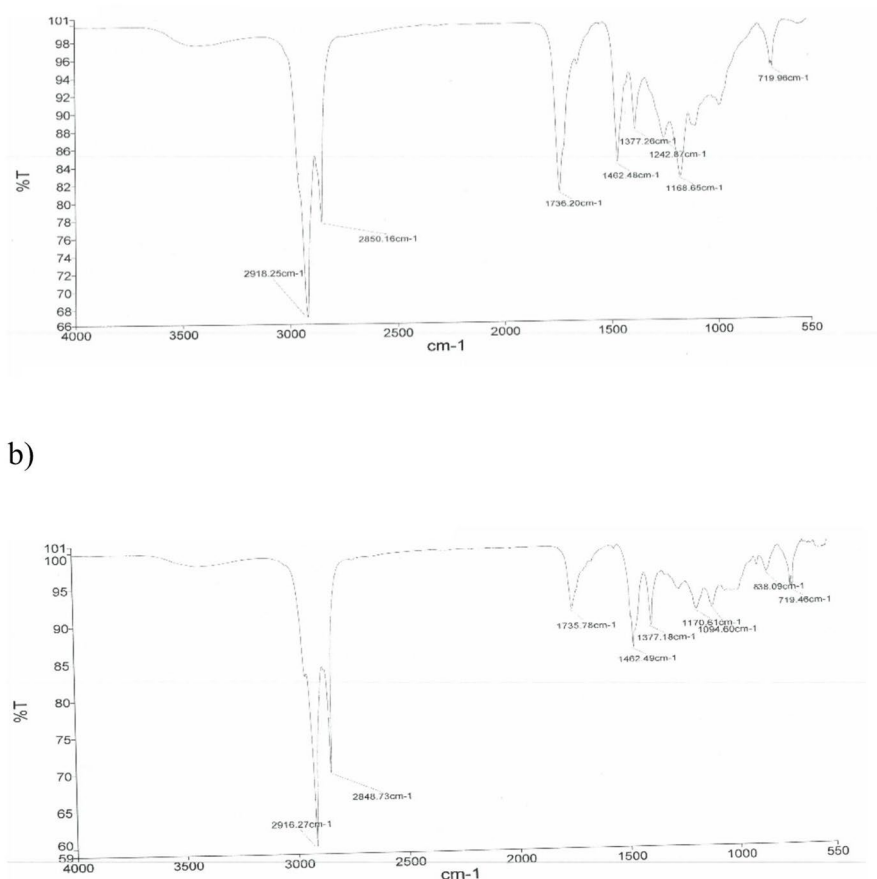


Fig. 7 Fourier transform infrared (FT-IR) spectra of (a) aqueous *Rubus caesius* L. fruit extract and (b) *Rubus caesius* L.-mediated gold nanoparticles (RC-AuNPs) synthesized using 0.075 mM $\text{HAuCl}_4 \cdot 0.3 \text{H}_2\text{O}$, 0.4 mL extract, and 180 W microwave irradiation

the FT-IR spectra of the aqueous *Rubus caesius* L. fruit extract and the synthesized RC-AuNPs, respectively. Comparison of the spectra revealed slight peak shifts and changes in band intensities following AuNP formation. The major absorption bands observed in the fruit extract at 2918.25, 2850.16, 1736.20, 1462.48, 1377.26, 1242.87, 1168.65, and 719.96 cm^{-1} appeared at 2916.27, 2848.73, 1735.78, 1462.49, 1377.18, 1170.61, and 719.48 cm^{-1} in the RC-AuNP spectrum, while the band at 1242.87 cm^{-1} disappeared or markedly weakened. In addition, weak bands at 1094.60 and 838.09 cm^{-1} were observed

in the RC-AuNP spectrum. These spectral changes, particularly those associated with C=O and C–O/C–O–C vibrations, indicate possible interactions between oxygen-containing constituents of the fruit extract and the AuNP surface. These functional groups may have contributed to Au³⁺ reduction and the surface stabilization of the synthesized AuNPs.

Figures 8(a, b) and 9 summarize the morphological and colloidal characteristics of RC-AuNPs synthesized using aqueous *Rubus caesius* L. fruit extract. The TEM micrographs shown in Fig. 8(a) revealed that the nanoparticles were generally well dispersed, although limited aggregation was observed in some regions. Morphological examination showed a heterogeneous particle population composed of spherical, triangular, and hexagonal structures. This mixed morphology may be related to the differential adsorption of phytochemical components from the fruit extract onto specific crystalline surfaces during microwave-assisted rapid reduction and nanoparticle growth. It has been reported that microwave irradiation affects nucleation and growth kinetics and can contribute to anisotropic nanoparticle formation under certain conditions [7, 16]. However, since TEM characterization focused on the selected condition rather than on samples synthesized at different microwave powers, the direct effect of microwave power on the observed morphology cannot be conclusively determined in the present study.

As presented in the particle size distribution histogram in Fig. 8(b), the RC-AuNPs had an average core diameter of 11.615 ± 3.156 nm, with particle sizes ranging from 8.155 to 21.998 nm. The particle-size distribution was determined from 40 nanoparticles ($n = 40$) measured in the TEM image. These results support the formation of nanosized gold particles, while also indicating a certain degree of shape heterogeneity. In contrast, DLS analysis (Fig. 9) yielded a considerably larger average hydrodynamic diameter of 71.92 ± 4.12 nm, together with a PDI value of 0.425, indicating a moderately polydisperse distribution in suspension. This apparent discrepancy can be explained by the different measurement principles of the two techniques. TEM provides the dried metallic core size of individual nanoparticles, whereas DLS measures the hydrodynamic diameter of particles dispersed in aqueous medium, including the solvation layer, surface-bound phytochemicals from the fruit extract, and possible loose particle associations. Moreover, DLS is intensity-weighted and is therefore highly sensitive to a small number of larger particles or aggregates. Thus, although the TEM histogram showed a relatively narrow core size

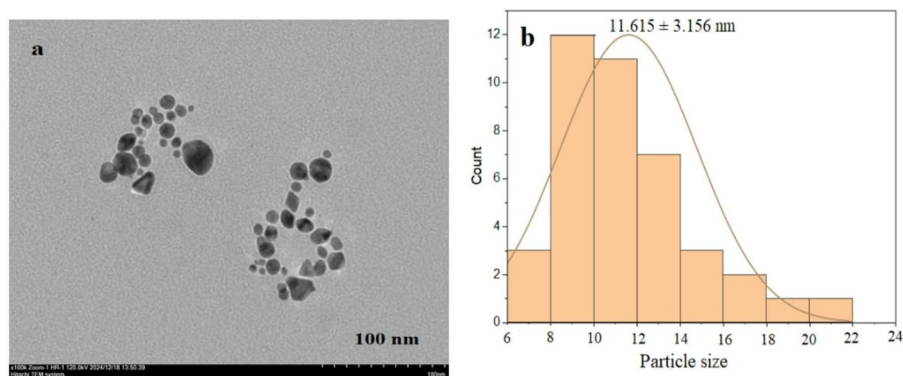


Fig. 8 Transmission electron microscopy (TEM) image (a) and particle-size distribution histogram (b) of *Rubus caesius* L.-mediated gold nanoparticles (RC-AuNPs) synthesized using 0.075 mM HAuCl₄·0.3 H₂O, 0.4 mL extract, and 180 W microwave irradiation

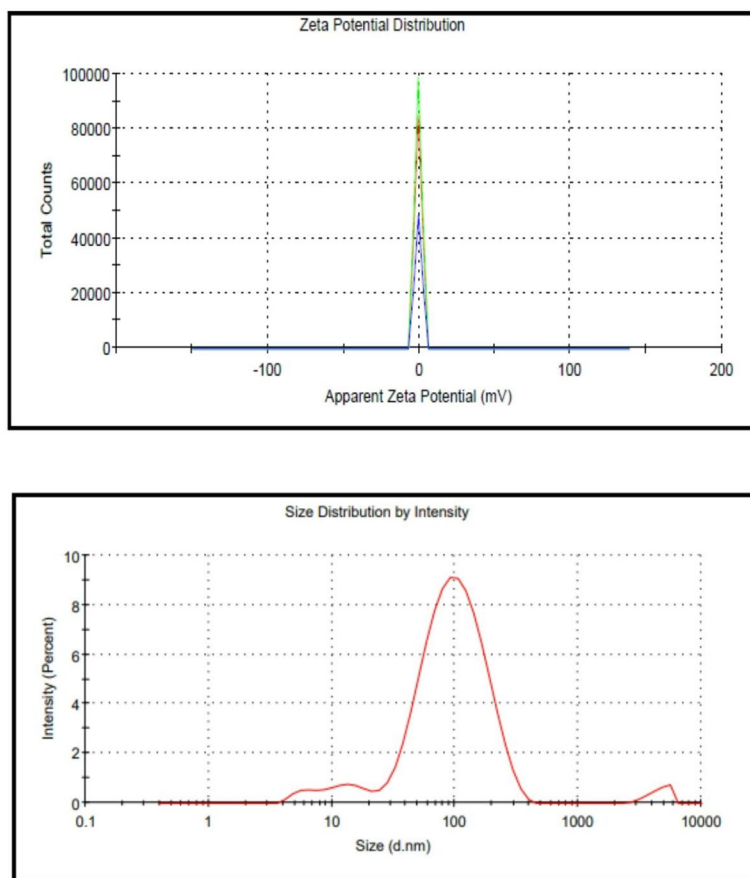


Fig. 9 Zeta potential and dynamic light scattering (DLS) intensity-weighted size distribution of *Rubus caesius* L.-mediated gold nanoparticles (RC-AuNPs) synthesized using 0.075 mM $\text{HAuCl}_4 \cdot 0.3 \text{ H}_2\text{O}$, 0.4 mL extract, and 180 W microwave irradiation

range of 8.155–21.998 nm, the higher DLS-derived hydrodynamic size and PDI value likely reflect the contribution of phytochemical surface layers, irregular particle shapes, and/or small amounts of larger aggregates in suspension. Similar differences between microscopic core size and colloidal hydrodynamic size have also been discussed in previous green and microwave-assisted nanoparticle synthesis studies using plant-derived extracts [5–7]. This interpretation is consistent with the measured zeta potential value of $-0.287 \pm 2.28 \text{ mV}$, which is close to neutral and indicates weak electrostatic repulsion between particles. Therefore, the colloidal system cannot be considered electrostatically stable, and the zeta potential data suggest limited electrostatic colloidal stability.

As shown in Table 2, the RC-AuNPs obtained in the present study exhibited nanoscale core dimensions comparable to those previously reported for other *Rubus*-mediated AuNPs. However, the near-neutral zeta potential and PDI value of 0.425 indicated limited colloidal stability. Unlike the previous studies, the present work combined an MAE-derived aqueous *R. caesius* L. fruit extract with microwave-assisted AuNP synthesis and systematically evaluated precursor concentration, extract volume, and microwave power. As shown in Table 2, the RC-AuNPs obtained in the present study exhibited nanoscale core dimensions comparable to those previously reported for other *Rubus*-mediated

Table 2 Comparison of *Rubus*-mediated AuNP synthesis studies with the present work

Plant source	Particle size	Zeta potential (mV)	PDI	Reaction / monitoring time	Microwave conditions	Stability	Reference
<i>Rubus idaeus</i>	18 ± 3 nm (TEM) ¹	NR	NR	SPR at ~ 10 min	900 W for 2 min during extract preparation; none during synthesis	Long-term colloidal stability; resistance to aggregation in up to 400 mM NaCl ²	Demirbaş et al., (2019)
<i>Rubus fruticosus</i>	18 ± 3 nm (TEM) ¹	NR	NR	SPR at ~ 40 min	900 W for 2 min during extract preparation; none during synthesis	Long-term colloidal stability; resistance to aggregation in up to 400 mM NaCl ²	Demirbaş et al., (2019)
<i>Rubus palmeri</i>	~ 13 nm (SEM/TEM); predominantly spherical at 30 °C and triangular at 20 °C	NR	NR	Monitored for 10 h at 30 °C; SPR peak at 540 nm	NR	Long-term stability: NR	Gurrola-Rios et al., [13]
<i>Rubus caesius</i> L.	11.6 ± 3.2 nm (TEM); 71.92 ± 4.12 nm (DLS)	−0.287 ± 2.28	0.425	SPR within 10–15 min ³	180 W during synthesis; 900 W for 8 min during MAE	UV–Vis spectral stability up to 45 days	Present study

¹ General TEM particle size reported for the berry-mediated AuNPs investigated by Demirbaş et al., (2019)

² Long-term colloidal stability and resistance to salt-induced aggregation were reported for berry-mediated AuNPs by Demirbaş et al., (2019)

³ The 10–15 min period represents the time required to obtain a well-defined SPR response and does not indicate complete reaction time

NR, not reported; PDI, polydispersity index; SPR, surface plasmon resonance; TEM, transmission electron microscopy; SEM, scanning electron microscopy; DLS, dynamic light scattering; MAE, microwave-assisted extraction.

AuNPs. Demirbaş et al., (2019) reported predominantly spherical and relatively uniform AuNPs synthesized using *R. idaeus* and *R. fruticosus* fruit extracts, with an average particle size of approximately 18 ± 3 nm. Gurrola-Rios et al., [13] reported *R. palmeri*-mediated AuNPs with an average diameter of approximately 13 nm. In comparison, the RC-AuNPs synthesized using aqueous *R. caesius* L. fruit extract had an average TEM core size of 11.6 ± 3.2 nm and exhibited a more heterogeneous morphology, including spherical, triangular, and hexagonal particles. However, the near-neutral zeta potential and PDI value of 0.425 indicated limited colloidal stability. Unlike the previous *Rubus*-based studies, the present work combined an MAE-derived aqueous *R. caesius* L. fruit extract with microwave-assisted AuNP synthesis and systematically evaluated precursor concentration, extract volume, and microwave power, thereby providing a process-oriented comparative evaluation framework.

4 Conclusion

In this study, AuNPs were synthesized using aqueous *Rubus caesius* L. fruit extract under microwave-assisted conditions, and their formation was supported by visual observation and spectroscopic analyses. The color change from yellow to purple-pink provided preliminary evidence of nanoparticle formation, which was further supported by the appearance of surface plasmon resonance (SPR) bands in the UV–Vis spectra. The results showed that both gold precursor concentration and extract volume had a

marked influence on nanoparticle formation, spectral characteristics, and dispersion behavior. Among the tested conditions, 0.075 mM $\text{HAuCl}_4 \cdot 0.3 \text{ H}_2\text{O}$ combined with 0.4 mL aqueous *Rubus caesius* L. fruit extract under 180 W microwave irradiation was selected as the most favorable synthesis condition, producing a well-defined and reproducible SPR band around 527 nm. Therefore, subsequent FT-IR, TEM, DLS, zeta potential, and UV–Vis spectral stability analyses were performed using RC-AuNPs synthesized under this selected condition. Likewise, the use of a higher extract volume led to less distinct spectral profiles, indicating that excess fruit extract may adversely affect the formation process under the selected conditions. In addition, the use of 180 W microwave power accelerated nanoparticle formation compared with 90 W, particularly in the presence of 0.4 mL extract, demonstrating the importance of microwave power in controlling reaction kinetics. UV–Vis spectral monitoring indicated that the synthesized RC-AuNPs largely maintained their spectral characteristics for up to 45 days, while changes in absorbance intensity became apparent by day 60. TEM analysis showed that the nanoparticles exhibited heterogeneous morphologies, including mainly spherical particles together with triangular and hexagonal forms, with an average core size of 11.6 ± 3.2 nm. In contrast, DLS analysis revealed a substantially larger average hydrodynamic diameter of 71.92 ± 4.12 nm and a PDI value of 0.425, indicating a moderately polydisperse distribution in suspension. The difference between TEM and DLS values is attributed to the fact that TEM reflects the metallic core size, whereas DLS measures the hydrodynamic size of particles in dispersion, including the surrounding solvation layer, surface-bound phytochemicals from the fruit extract, and possible loose particle associations. FT-IR analysis further suggested that oxygen-containing phytochemicals and related functional groups in the fruit extract may have participated in both the reduction of Au^{3+} ions and the surface stabilization of the resulting nanoparticles. However, the measured zeta potential value (-0.287 mV) was close to neutral, suggesting weak electrostatic repulsion between the particles. Therefore, the colloidal system cannot be considered electrostatically stable, and the zeta potential data suggest limited electrostatic colloidal stability.

In summary, the present study demonstrates that aqueous *Rubus caesius* L. fruit extract can be effectively employed in the microwave-assisted green synthesis of AuNPs, yielding nanoparticles with nanoscale core dimensions and condition-dependent optical and colloidal properties. The findings highlight that careful adjustment of precursor concentration, extract volume, and microwave power is essential for achieving favorable nanoparticle formation. Although the RC-AuNPs exhibited UV–Vis spectral stability for up to 45 days, they cannot be considered colloidally stable because of their near-neutral zeta potential and moderately polydisperse size distribution. Thus, the main contribution of this work lies in providing a comparative, species-specific evaluation of *Rubus caesius* L. fruit extract as a reducing and capping source within the broader context of plant-mediated AuNP synthesis. One of the important strengths of this study is the systematic evaluation of precursor concentration, fruit extract volume, and microwave power. This approach provides a process-oriented basis for RC-AuNP synthesis. Since the primary aim of the study was to evaluate nanoparticle production conditions and the physicochemical properties of the resulting nanoparticles, detailed phytochemical profiling was beyond the scope of the present work. Therefore, interpretations concerning the individual roles of specific phytochemical constituents in the reduction and

stabilization processes were kept limited. From a sustainability perspective, the combined use of an aqueous plant extract and microwave-assisted processing is particularly relevant to SDG 12 (Responsible Consumption and Production), as it supports a greener synthesis approach with reduced reliance on conventional chemical reducing agents and prolonged processing steps. Future studies should focus on evaluations incorporating different and complementary characterization techniques. Such studies may contribute to the development of green nanoparticle synthesis protocols.

Acknowledgements

The authors gratefully acknowledge the support provided by the Central Research Laboratories of Atatürk University and Karadeniz Technical University.

Author contributions

Gönül Serdar, Vildan Serdaroğlu, and Melek Koç Keşir contributed to the study design, experimental work, data visualization, formal analysis, and manuscript preparation. Memnune Kokoç performed TEM data visualization and statistical analyses and assisted with manuscript preparation. Salih Terzioğlu contributed to plant identification and sample collection.

Funding

This study received no external funding.

Data availability

The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

Declarations

Ethical approval

Not applicable. This study did not involve human participants, animals, or clinical samples.

Consent to participate

Not applicable. This study did not involve human participants.

Consent to publish

Not applicable. This manuscript does not contain any identifiable personal data.

Plant material statement

The plant material used in this study consisted of wild *Rubus caesius* L. specimens collected in August 2024 from Düzköy, Trabzon, Türkiye. The specimens were collected from a non-private, publicly accessible natural area; therefore, no landowner permission was required. The collection was conducted in accordance with relevant institutional, local, and national requirements for plant material sampling. Taxonomic identification of the specimens was performed by Prof. Dr. Salih Terzioğlu. A voucher specimen was deposited in the Herbarium of the Department of Forest Engineering, Faculty of Forestry, Karadeniz Technical University, Trabzon, Türkiye, under the voucher number KATO 24461.

Competing interests

The authors declare no competing interests.

Received: 20 April 2026 / Accepted: 31 August 2026

Published online: 04 September 2026

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