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(Research Article)

From Ions to Aggregates: Green Synthesis and Characterization of Silver Microparticles Using Endemic *Origanum sipyleum* L. Extract

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Abstract

Most research on plant-mediated synthesis aims to produce nanoparticles. However, nature does not always follow this nanoscale path; factors like reaction kinetics and phytochemical saturation can sometimes push the system toward micro-scale structures. In this study, we report the biosynthesis of silver microparticles (AgMPs) using the aqueous extract of *Origanum sipyleum* L., a plant endemic to the Kazdağı region of Türkiye. Our characterization using UV–Vis, FTIR, and SEM revealed an interesting outcome: while the extract effectively reduced silver ions via its phenolic –OH groups, the specific reaction environment promoted the growth of irregular, micrometer-sized aggregates instead of discrete nanoparticles. We observed a hypsochromic shift in the UV-Vis spectra (λmax ~350 nm) rather than the typical nanoscale Surface Plasmon Resonance (SPR) peak. This data, combined with SEM imagery, suggests a growth mechanism driven by Ostwald ripening and phytochemical bridging. Ultimately, these findings distinguish the plant's reducing power from its stabilizing capability, offering a new perspective on the thermodynamic boundaries between nano- and micro-particle formation in green chemistry.

Keywords: Silver microparticles; *Origanum sipyleum* L.; Green synthesis; Ostwald ripening; Endemic plants

1. Introduction

Green synthesis is widely recognized as a sustainable pillar of materials science because it eliminates the need for hazardous chemicals in reduction processes (Ahmed et al., 2016; Iravani, 2011). In this field, plant-mediated protocols are particularly popular for generating metallic structures. Researchers predominantly focus on silver nanoparticles (AgNPs) because of their well-documented antimicrobial and catalytic properties (Balasubramanian et al., 2019; Dolai et al., 2021). Yet, the scope of green chemistry is not limited to the nanoscale. Depending on variables such as reaction kinetics, supersaturation levels, and nucleation dynamics, plant extracts can also yield micro-structured materials.

Despite the abundance of literature on AgNPs, the green synthesis of silver microparticles (AgMPs) is rarely documented. As a result, we still lack a complete understanding of how these larger structures form in biological systems. This gap is significant. To define the true capabilities of specific plant extracts, we must understand the boundary conditions where nucleation ends and micro-scale growth begins. While most scientists aim for monodispersity at the nanoscale, the fundamental thermodynamic mechanisms that trigger aggregation—specifically Ostwald ripening and phytochemical bridging—remain underexplored in plant-based studies.

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There are species of medicinal and economic value in the Lamiaceae family (Paşa et al., 2019; Paşa, 2019). The genus *Origanum* is an excellent candidate for metal ion reduction due to its rich phenolic profile (Sankar et al., 2013; Zahran et al., 2018). *Origanum sipyleum* L., which is endemic to the Kazdağı region, is known for its antioxidant and pharmacological potential (Ferrante et al., 2019; Kayabasi et al., 2022; Nakiboglu et al., 2007; Özer et al. 2018). However, to date, no one has explored its ability to synthesize metallic structures, whether at the micro- or nano-scale.

In this study, we address this gap by using *O. sipyleum* extract to synthesize silver structures. Rather than discarding our results for failing to meet "nano" expectations, we aim to characterize these silver microparticles. By doing so, we seek to elucidate the thermodynamic conditions—such as Ostwald ripening—that favor micro-scale formation over nanoscale stabilization.

2. Materials and Methods

2.1. Plant Collection and Identification

We collected aerial parts of *O. sipyleum* L. from the Kazdağı region (Balıkesir, Türkiye) between May and July 2024. A specialist taxonomically verified the plant material to ensure consistency with previous botanical studies in the region (Durmuskahya et al., 2016).

2.2. Extract Preparation

To prepare the aqueous extract, we modified the procedure described by Ambika and Sundrarajan (2015). We infused 25 g of dried plant material in 250 mL of distilled water at 70–80 °C. After cooling the mixture, we filtered it to remove solid residues and stored the liquid at 4 °C for later use.

2.3. Synthesis of Silver Structures

For the synthesis process, we mixed 125 mL of the prepared plant extract with 500 mL of 1 mM aqueous $AgNO_3$ solution (Jain et al., 2011; Nithya & Ragunathan, 2009). We monitored the reaction mixture for a color change, which serves as the primary indicator of silver ion reduction. Once the reaction was complete, we centrifuged the suspension at 10,000 rpm to isolate the particles, then washed and dried them for characterization.

2.4. Characterization Techniques

We analyzed the morphological and optical properties of the synthesized particles using Scanning Electron Microscopy (SEM; EVO 40 LEQ), Fourier Transform Infrared Spectroscopy (FTIR; Perkin Elmer Spectrum One, 4000–600 cm^{-1}), and UV–Vis Spectroscopy (Lambda 25, 200–900 nm) (Balaz et al., 2017; Beydoun et al., 1999).

3. Results

3.1. FTIR Analysis

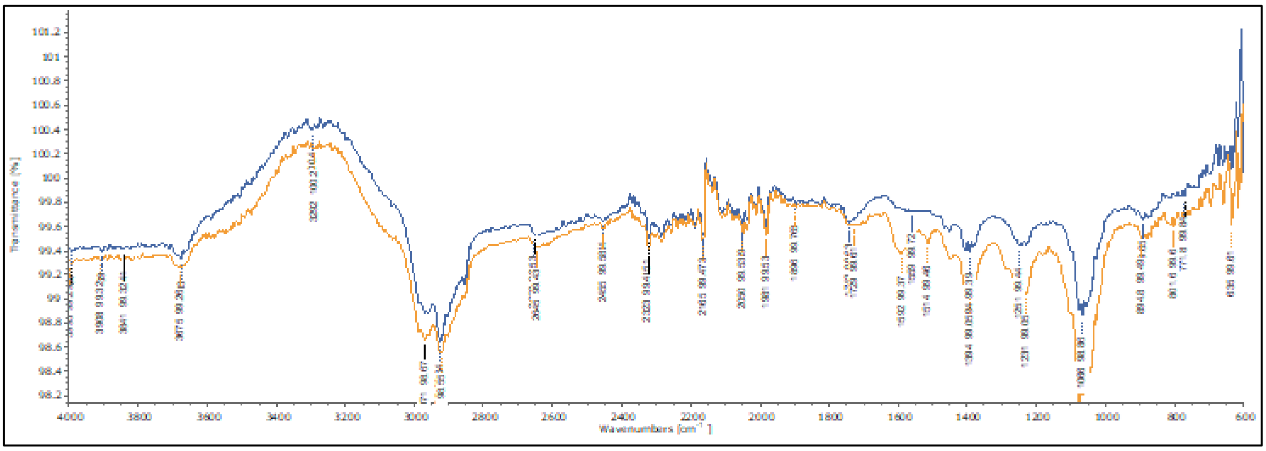

Figure 1 IR spectrum of *O. sipyleum* L. extract (top) and silver particle centrifuge filtrate (bottom)

We employed FTIR analysis to identify which functional groups were responsible for reducing and capping the silver ions. When we compared the spectrum of the synthesized particles to the crude extract, we observed significant shifts in the phenolic –OH stretching region (3000–3600 cm^{-1}) (Figure 1). This shift suggests that phenolic groups oxidized during the reduction of Ag^{+} to Ag^{0}, a mechanism that aligns with other plant-mediated synthesis studies (Ambika & Sundrarajan, 2015; Sankar et al., 2013). Since there were no major shifts in the carbonyl or aliphatic regions, we can infer that phenolic moieties were the primary reducing agents in this system.

3.2. UV–Vis Spectroscopy

Optical characterization gave us the first clue regarding particle size. The reaction mixture showed a maximum absorbance (λmax) at 350.26 nm. This represents a hypsochromic shift from the extract's baseline of 341.83 nm (Figure 2). It is important to note that spherical AgNPs typically display a Surface Plasmon Resonance (SPR) band between 400 and 450 nm (Ahmed et al., 2016; Zahran et al., 2018). In our case, the absence of this characteristic SPR peak, combined with absorbance in the UV region, points to particles that exceed the nanometer limit. This is consistent with Mie theory, which predicts that SPR bands will dampen and broaden as larger aggregates form.

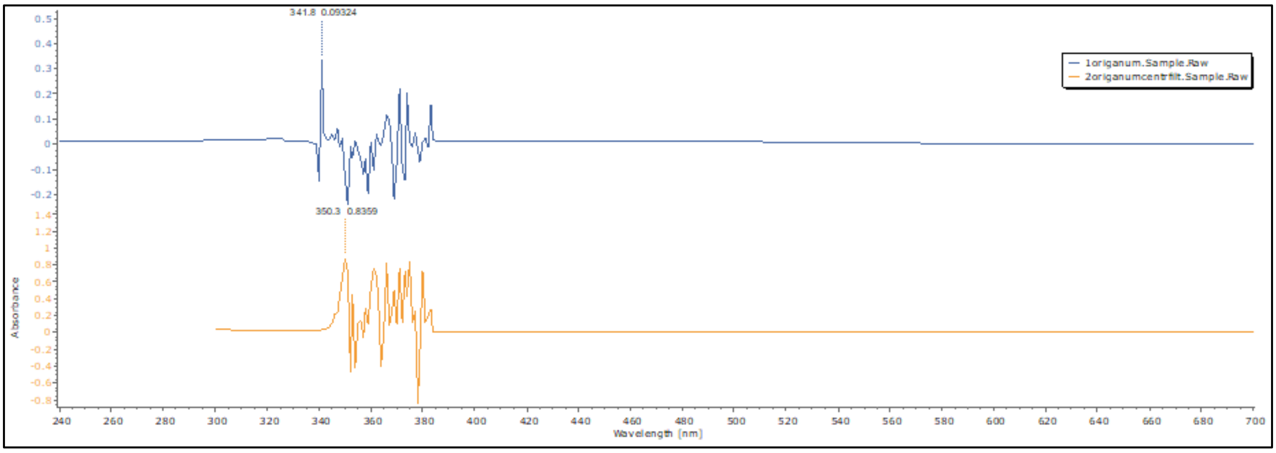

Figure 2 UV-Vis spectrum of *O. sipyleum* L. extract (1) and centrifugation filtrate (2)

3.3. SEM Analysis

Microscopic imaging (Figure 3) confirmed that we had synthesized micro-scale structures rather than discrete nanoparticles. The particles exhibited an irregular, heterogeneous morphology, with sizes falling predominantly in the micrometer range. We observed significant aggregation and boundaries where particles had fused, finding no evidence of discrete nanoscale structures. These morphological features imply a rapid growth phase where capping agents failed to effectively arrest nucleation (Mittal et al., 2013).

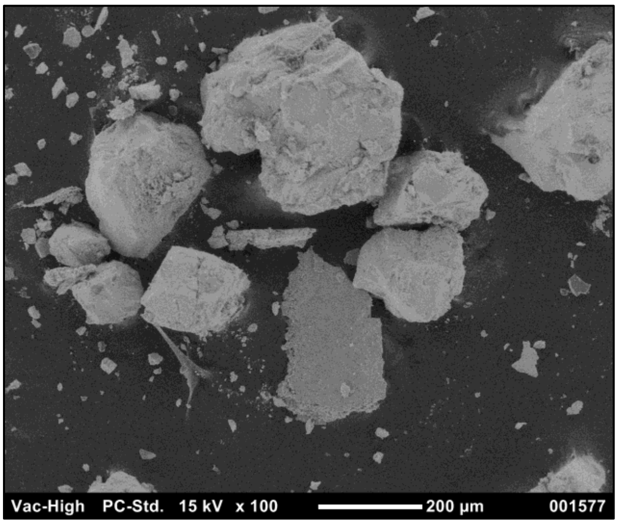

Figure 3 SEM image of silver particles

4. Discussion

The synthesis of silver microparticles using *O. sipyleum* sheds light on the distinct thermodynamic behavior of this endemic extract. Although the reduction of silver ions was clearly successful—evidenced by both the color change and FTIR data—the particle growth mechanism favored the formation of larger aggregates.

We attribute the formation of microparticles to two main factors: Ostwald ripening and uncontrolled nucleation. In systems where precursor concentrations are high or capping efficiency is insufficient, smaller nuclei are thermodynamically unstable. Consequently, they tend to dissolve and redeposit onto larger particles to minimize surface energy. The irregular morphology in our SEM images supports this theory, suggesting an initial burst of nucleation followed by a rapid growth phase where particles fused into micro-structures.

In addition, the high concentration of phenolic compounds in the *O. sipyleum* extract likely created a "bridging effect." Phenolics act as reducing agents, but at high saturation levels, they can physically bridge multiple silver nuclei together. This promotes flocculation rather than keeping individual particles isolated. This behavior contrasts with optimized synthesis protocols, where researchers adjust pH and temperature to increase repulsive forces in the electrical double layer. In our study, the unoptimized high ionic strength likely compressed this layer, which facilitated the aggregation we observed (Mittal et al., 2013).

Crucially, these findings challenge the assumption that green synthesis is only scientifically valuable if it yields nanoparticles. Instead, this work defines the boundary conditions of the process. It demonstrates that while *O. sipyleum* has a high reducing potential, it lacks the stabilizing efficiency required for nanoscale arrest under these specific extraction conditions. This distinction is vital for future applications where micro-scale silver might be preferred, such as in filtration-based catalysis or systems requiring slower ion release.

5. Conclusion

This study documents the first green synthesis of silver microparticles using the endemic *Origanum sipyleum* L. extract. Our data confirms that the extract effectively reduces Ag^+ ions. However, the reaction kinetics favored the formation of thermodynamically stable, micrometer-scale aggregates rather than nanoparticles. This transition from nucleation to microscale aggregation appears to be driven by Ostwald ripening and phytochemical bridging. Therefore, this research provides a baseline for understanding the kinetic limitations of *O. sipyleum* extracts. It suggests that future efforts aiming for nanoscale materials must prioritize the optimization of stabilizing parameters, such as pH and dilution factors.

Compliance with ethical standards

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