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The Effect of Microwave Power in the Green Synthesis of Silver Nanoparticles Using *Citrus sinensis* Peels Extract

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Abstract

There are numerous green methods for synthesizing silver nanoparticles using plant extracts such as leaves, flowers, stems, and fruit extracts. However, most of those synthesized have weaknesses such as slow reduction and inefficient time. This study used a microwave to accelerate the reduction process of Ag^+ ions into Ag^0 nanoparticles using an aqueous extract of *Citrus sinensis* peels. A heating time of 5 minutes produces silver nanoparticles in optimal condition with a color change from yellow to reddish brown. According to UV-Vis, silver nanoparticles at power 100 W and 300 W show peaks at 404 nm and 406 nm. FTIR indicates that phytochemical compounds are involved in the reduction of nanoparticles. XRD shows silver nanoparticles are FCC crystalline. TEM reveals that power 100 W yields an average diameter of 12 nm while 300 W shows a smaller diameter of 5 nm.

1. Introduction

Nanoparticles are currently being developed for the needs of human life. Researchers explore various materials to determine their advantages at the nanoscale. Silver is one of the materials that is currently being explored as a nanomaterial because it has many potentials such as biocatalyst [1, 2, 3], antibacterial [4, 5, 6], antioxidant [7, 8, 9], good X-ray fluorescence [10], drug delivery [11, 12], and cancer imaging [13, 14, 15]. Many methods of synthesizing silver nanoparticles have been used, one of which is the green synthesis method. Green synthesis utilizes phytochemical components in plants that donate electrons to bind with silver ions, resulting in a reduction process. Researchers consider it less effective because the natural reduction process with the aid of plants takes many hours [16].

A new synthesis technique called microwave irradiation has just been developed to accelerate the reduction process. The presence of a microwave is thought to speed up the reduction process by directly heating the material without using a container (conduction or convection) [17]. This study used microwaves to synthesize silver nanoparticles using *Citrus sinensis* peel extract. The influence of microwave power and irradiation time on the synthesis of silver nanoparticles was assessed. UV-Vis, FTIR, XRD, and TEM techniques were employed to facilitate identification.

2. Experimental

2.1. Materials and Instruments

The materials used were silver nitrate powder (Merck, 99.8%), *Citrus sinensis* peel, distilled water, deionized water (DI water), sodium hydroxide (NaOH), hotplate, and Whatman filter paper no. 42, Samsung ME731K (2.45 GHz) microwave, sonicator, UV-Vis, FTIR, XRD, and TEM.

2.2. Preparation of *Citrus sinensis* Peel Extract

Peels from *Citrus sinensis* were soaked in deionized water for an hour and then allowed to dry. The peel was sliced into small pieces, weighed, and combined with 100 mL of distilled water in a glass, as per Kahrilas *et al.* [18]'s instructions. Next, the *Citrus sinensis* peel solution was heated for 10 minutes at 90°C. The Whatman paper was used to filter the extract after it was left to stand until it arrived at room temperature.

2.3. Synthesis of Silver Nanoparticles

A solution of 1 mM silver nitrate ($AgNO_3$) was combined with a ratio of 5:1 with *Citrus sinensis* peel extract, referred to as the sample. The addition of 0.1 mL of a 1 M NaOH solution modified the pH of the sample to 10. Next, the samples were exposed to microwave irradiation for 1, 3, and 5 minutes at a power level of 100 W to determine the optimal irradiation time. As a result, the optimal time was employed as an independent variable to control the microwave power at 100 W and 300 W. The samples were further characterized using UV-Vis spectroscopy, Fourier-Transform Infrared Spectroscopy (FTIR), X-ray diffraction (XRD), and Transmission Electron Microscopy (TEM) techniques to determine their properties.

3. Results and Discussion

3.1. UV-Vis Analysis

UV-Vis spectrophotometer was used to identify the UV or visible light absorption bands of silver nanoparticles. The SPR peak between 380 and 450 nm determined the quality of the synthesized silver nanoparticles [19, 20, 21]. Figure 1 shows colloidal silver nanoparticles of varied microwave irradiation times. After one minute of irradiation, the figure shows no production of silver nanoparticles. This demonstrates that the reduction process is unable to occur with low power (100 W) for a brief period of time.

Figure 1 additionally reveals the presence of SPR peaks at 3 and 5 minutes, at wavelengths of 404 nm and 406 nm. As shown in Figure 1, the peak reaches its maximum at the 5-minute irradiation. Longer heating times result in an increase in absorbance intensity, indicating the generation of more nanoparticles in the colloid [22]. Next, a duration of 5 minutes was employed as a control variable to determine the attributes of silver nanoparticles under varying microwave power.

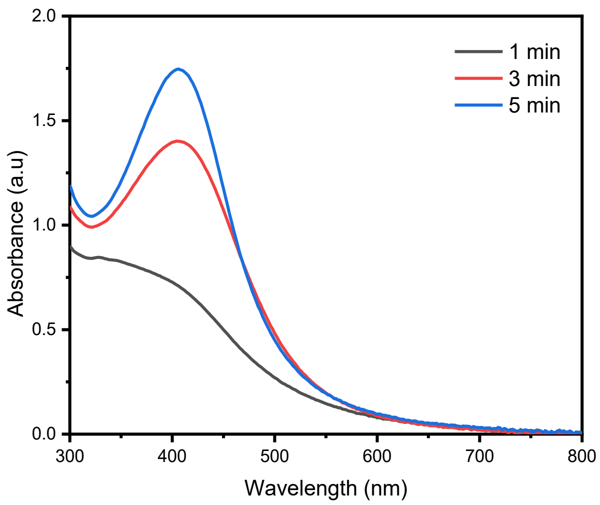

Figure 1. UV-Vis spectrum of colloidal silver nanoparticle synthesis with *Citrus sinensis* peel extract at varying microwave times

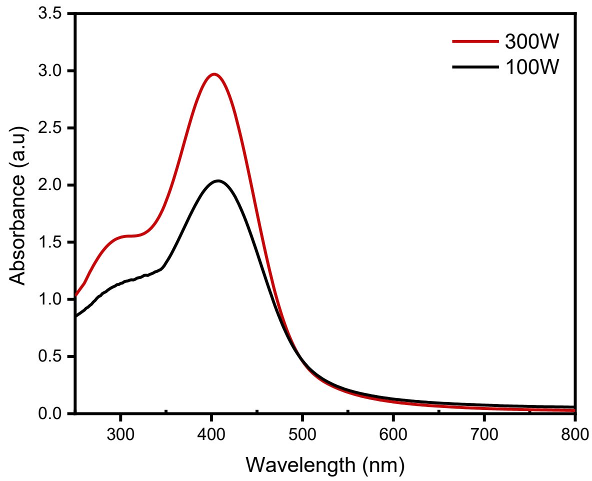

Figure 2. UV-Vis spectrum of colloidal silver nanoparticles with *Citrus sinensis* peel extract at 100 W and 300 W microwave power

The UV-Vis spectrum in Figure 2 illustrates how power affects the properties of silver nanoparticles. Figure 2 shows that the SPR of colloids of silver nanoparticles is 404 nm with an absorbance of 1.4 at 100 W power and 406 nm with an absorbance of 1.7 at 300 W power. These results support the findings of studies by Ali *et al.* [22] and Motitswe and Fayemi [23], which state that applying higher power results in producing more nanoparticles. This may arise because high temperatures will increase the components' kinetic motion, weakening the group bonds in *Citrus sinensis* peel extract and raising the probability of electron release [24]. More nanoparticles are certain to arise due to this increase in the chance of interaction between free electrons and silver ions.

3.2. FTIR Analysis

FTIR is a technique for identifying and analyzing functional groups involved in the reduction process. The FTIR spectra of silver nanoparticles at 100 W and 300 W power and the *Citrus sinensis* peel extract are displayed in Figure 3. The presence of an absorption peak at 3389 cm^{-1} suggests the existence of an O-H bond, specifically associated with a hydroxyl group. Peaks 1405 cm^{-1} and 2934 cm^{-1} show the existence of C-H bonds (alkanes), whereas peaks 2363 cm^{-1} and 1621 cm^{-1} show the presence of C=O (carbonyl) bonds, which are present in terpenoids and flavonoids [25]. The peak of a C-O-H at 1061 cm^{-1} suggests the presence of a C-O-H (carboxyl) bond. These groups' shift in transmittance peaks indicates that they are involved in the reduction process [23].

At a power level of 300 W, this compound demonstrates substantial changes in transmittance. This illustrates that the hydroxyl group donates additional electrons to the silver ion, indicating that more reduction reactions occur at higher power levels. The existence of peaks at 670 cm^{-1} and 679 cm^{-1} in silver nanoparticle colloids suggests that the carbonyl group arises from modifications in the carboxyl group, resulting in the release of hydroxyl (O-H) groups [26, 27].

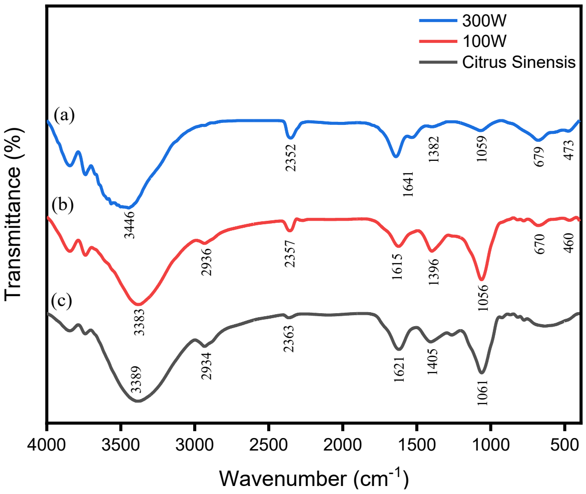

Figure 3. FTIR spectra of silver nanoparticles at (a) 300 W, (b) 100 W, (c) *Citrus sinensis* peel extract

Citrus sinensis peel extract contains phenolic, ketone, flavonoid, and terpenoid chemicals commonly observed to have hydroxyl, carbonyl, and carboxyl groups [28]. The phytochemical elements in *Citrus sinensis* peel extract have been crucial in converting silver ions into silver nanoparticles, as evidenced by the FTIR spectra [29]. Silver nanoparticles are present since their last absorption peaks are located at 460 cm^{-1} and 473 cm^{-1} [30, 31, 32, 33].

3.3. XRD Analysis

XRD is used to verify the crystalline structure of silver nanoparticles. The XRD diffractograms of silver nanoparticles at power 100 W and 300 W are displayed in Figure 4. Diffraction peaks at 38.14 (111), 38.27 (111), 44.21 (200), 44.5 (200), 64.57 (220), and 77.56 (311) indicate Face-Centered Cubic (FCC) crystal properties, as explained by ICDD No. 89-3722 [34, 35, 36]. The nanoparticles produced with a power of 100 W exhibited impurities at 2θ values of 33.04° and 61.74°. However, no additional impurity peaks were detected when the power was increased to 300 W. This is due to the fact that increased power results in the release of more electrons, which speeds up the creation of nanoparticles [37, 38].

3.4. Transmission Electron Microscopy (TEM) Analysis

TEM was utilized to examine the shape and size of the silver nanoparticles. Figures 5 in this work illustrate the investigation of the size and shape of silver nanoparticles at each power condition. Figure 5(a) displays the shape of silver nanoparticles produced at 100 W of power. The resulting nanoparticles have a spherical shape with an average diameter of 12 nm, as shown in Figure 5(c). In these circumstances, a size range of 5 to 25 nm is formed. The shape of silver nanoparticles produced under 300 W power settings is displayed in Figure 5(b). The nanoparticles exhibit a consistent spherical morphology, with an average diameter of 5 nm and a size distribution ranging from 1 to 7 nm (Figure 5(d)).

The size of the silver nanoparticles generated in this investigation is smaller than that of earlier investigations, one of which was conducted by Niluxsshun *et al.* [39]. The process of synthesis required producing 5-80 nm nanoparticles by allowing the material to remain at room temperature in the absence of light for five hours. Further research by Zayed *et al.* [34] using traditional heating yielded 35–45 nm silver nanoparticles. According to these two techniques, creating smaller-sized nanoparticles is more successful when using silver nanoparticles. This aligns with the viewpoint presented by Jahan *et al.* [17] in the introduction.

Increasing the power of microwave irradiation will result in a higher energy level, leading to an increased transfer of electrons that interact with silver ions. The heightened level of interaction leads to an increased nucleation rate in nanoparticles, resulting in a more comprehensive crystallization process [38, 40]. As a result, the produced nanoparticles will have a smaller and more consistent size. Furthermore, increasing the reaction rate results in a larger quantity of silver nanoparticles, as illustrated in Figure 2.

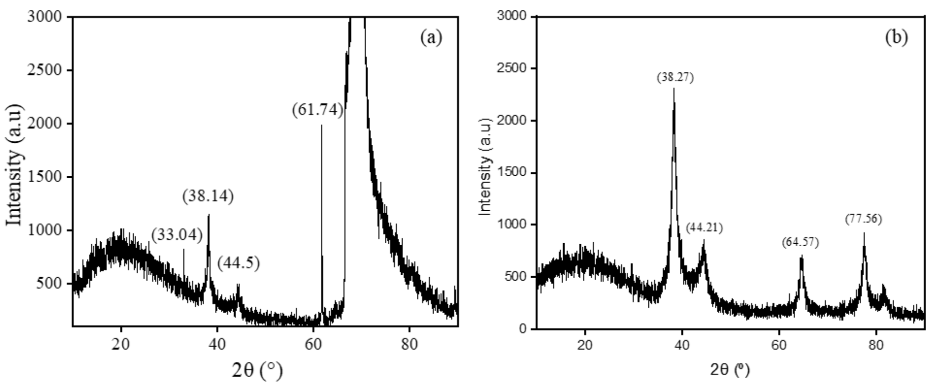

Figure 4. XRD diffractograms of synthesized silver nanoparticles using *Citrus sinensis* peel extract at microwave powers of (a) 100 W, (b) 300 W

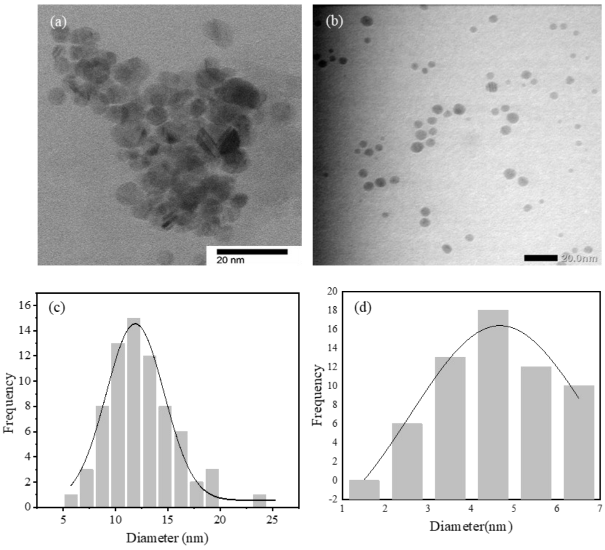

Figure 5. TEM images of silver nanoparticles synthesized with *Citrus sinensis* peel extract at microwave powers of (a) 100 W and (b) 300 W. Size distributions of synthesized silver nanoparticles at (c) 100 W and (d) 300 W

4. Conclusion

Silver nanoparticles have been effectively synthesized using *Citrus sinensis* peel extract and microwave assistance. UV-Vis, FTIR, XRD, and TEM were used to examine the properties of the nanoparticles. UV- Vis measurements indicate that more nanoparticles are produced with longer heating times. The phytochemical components of *Citrus sinensis* peel extract were found to be crucial to the reduction of silver ions into silver nanoparticles, according to FTIR analysis. Furthermore, Low-power (100 W) nanoparticle synthesis has yielded crystals with an FCC structure. By reducing the power from 100 W to 300 W, the diameter of the nanoparticles drops from 12 nm to 5 nm, as demonstrated by TEM, which also reveals that the particles have a spherical geometry. The results of this study validate that smaller nanoparticle sizes are obtained at increased microwave power. Moreover, the production of silver nanoparticles can be accelerated by using microwaves.

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