Green Synthesis and Characterization of Gold Nanoparticles **Using Aqueous Extract of Menthapolegium**

Pouran Pourhakkak^{*}, Shokoufeh Omidi, Mahdi YarAhmadi

Department of Chemistry, Payame Noor University, 19395-4697, Tehran, Iran

Received: 14 Nonember 2023 Accepted: 12March 2024 DOI: 10.30473/ijac.2024.69719.1280

Abstract

In this research, the results of two existing methods for synthesizing gold nanoparticles, i.e., the green and environment-friendly method and the usual chemical method called Turkovich, were compared and analyzed. In the green method, the extract of the aerial parts of the medicinal plant Mentha pulegium was used as a regenerating agent, and both methods, SEM, TEM, UV-VIS, EDX, and DLS, investigated the properties of the synthesized gold nanoparticles. The results indicated that the green synthesis of gold nanoparticles by extracting the aerial parts of the Mentha pulegium plant was smaller and more stable than the Turkevich method. SEM and TEM results show that the morphology of all nanoparticles is spherical. The size of nanoparticles synthesized by sodium citrate is 10.3 nm, and the size of nanoparticles synthesized by Mentha pulegium plant extract is 9.6 nm. The UV-VIS results show almost absorption in the region of 526 nm for gold nanoparticles synthesized by sodium citrate and absorption in the region of 421 nm for gold nanoparticles synthesized by Mentha pulegium plant extract. The synthesized gold nanoparticles had antibacterial activity against different bacteria.

organic-mineral

Keywords

Green synthesis; Mentha pulegium; Gold; Nanoparticle.

1.INTRODUCTION

Metal nanoparticles are more stable in plants than in other organisms in the body. Plants (especially plant extracts) can reduce metal ions faster than fungi and bacteria. In addition, to use an easy and safe green method in the scale and industrial production of dispersed metal nanoparticles, plant extracts are certainly better than plant biomass or live plants [1]. Today, many methods are used in the synthesis of nanoparticles, which depend on the shape and size of the particles. Torkovic described the first and most popular method. In recent years, the biosynthesis method using plant extracts has received more attention than physical and chemical methods [2]. Medicinal plants contain effective compounds such as flavonoids, terpenes, alkaloids, and antioxidants, which are part of the main reducing agents and cause the transformation of metal particles into nanoparticles [3]. In general, gold nanoparticles are produced in a liquid, reduced by sodium citrate by the Torkovitch method by reduction (oxidation-reduction) of chloroauric acid. Stabilizers are added to prevent particles from clumping. Citrate acts as both a reducing agent and a colloid stabilizer. They can be used with different organic ligands to create

hybrids

with

advanced

capabilities [4]. Considering the optical, electronic, molecular cognitive properties, gold and nanoparticles are the subject of significant research, many of which are used in fields such as electron microscopy, electronics, nanotechnology, materials science, and biomedicine [5-8]. Gold nanoparticles play an important role in nanotechnology and biomedicine due to their suitable biomass in biomolecular probes and significant optical parameters of resonant plasmon [9]. The crystalline nature of the synthesized gold nanoparticles was analyzed using the X-ray diffraction method [10]. Plasmonic nanoparticles are particles whose electron density can couple with electromagnetic radiation of much larger wavelengths than the particle due to the nature of the dielectric-metal interface between the medium and the particles: unlike pure metal, which has a maximum limit of how many wavelengths can effectively couple based on the size of the material [11]. What distinguishes these particles from natural surface plasmons is that plasmonic nanoparticles also exhibit interesting scattering, absorption, and binding properties based on their geometry and relative positions [11, 12]. These

^{*} Corresponding author: p. pourhakkak; E-mail: <u>p_pourhakkak@pnu.ac.ir</u>

unique properties have attracted attention in many applications, including solar cells, spectroscopy, signal amplification for imaging, and cancer treatment [13, 14]. At the end of medicine, efforts are being made to diagnose diseases, prevent them, and improve patients by using nanotechnology [15, 16]. It covers the use of nanomaterials in medical affairs and the use of nanoelectronics in the design of biosensors. One of the most critical medical problems is understanding nanotechnology's type and degree of impact on the environment and knowing whether or not nanoscale materials are toxic [17]. Many researchers have shown that gold nanoparticles can fight cancer cells. Gold nanoparticles synthesized using Gymnema Sylvestre, commonly known as cow plant, showed cytotoxic effects on Hep2 cells. Morphological changes were observed in Hep2 cells after treatment with synthesized gold nanoparticles. It showed the level of reactive oxygen species and nuclear changes was determined, and it was revealed that the death of Hep2 cells is caused by apoptosis [18]. The effective ingredients of medicinal plants play a unique role in controlling microbial agents, especially bacteria [19, 20].

In a study, Bouaichi et al. evaluated the antibacterial effect of some medicinal plants, including thyme, rosemary, and Mentha pulegium, against Pseudomonas savastanoi. Their study showed that thyme has the most antibacterial effect [21]. Elshae et al. studied the antimicrobial effect of some medicinal plants from the Mediterranean region against some plant pathogens, including Pseudomonas [22-23] investigated the antibacterial effect of 22 plant essential oils, including Mentha pulegium, against two plant Pseudomonas pathogenic bacteria, putidavarviniaherbicula. This study showed that Mentha pulegium essential oil has an excellent antibacterial effect compared to streptomycin and agromycin antibiotics. Mentha Pulegium from the mint family (Lamiaceae) contains volatile important or menthol oil (as the main ingredient), tannins, resinous substances, sugars, and vitamins. The extracts of Mentha pulegium leaves, owners, and stems can be a suitable alternative to many antibiotics due to their antimicrobial properties [24]. The compounds in Mentha pulegium are among the antioxidants that eliminate the effect of active and damaging molecules on DNA and proteins due to the property of trapping existing free radicals [25]. In the last several years, many studies have been conducted on the antioxidant activity of medicinal plants, including Mentha pulegium, which was found to be responsible for the plant's antioxidant activity [26]. Volatile compounds of the Mentha pulegium plant include menthone, menthol, iso menthone, piperine, neo menthone, 3-octanol, and polygon, which in Mentha pulegium plant extract; polygon compound can act as the most critical effective compound in antimicrobial activities and against some pathogenic bacteria [27-30].

2.EXPERIMENTAL

2.1. Materials

First, all the necessary laboratory utensils were washed with distilled water to perform the test. Then, we prepared the required standard materials. Mentha pulegium medicinal plant leaf extract was obtained using the Soxwell apparatus.

2.2. Standard preparation of required materials

To prepare 1% sodium citrate, we weigh 0.25 grams of sodium citrate with a digital scale, transfer it to a 25 ml ask, and make it up to volume with double distilled water. To prepare standard chloroauric, we take 1ml of chloroauric stock with a pipette, transfer it to a 100 ml ask, and make it up to volume with double distilled water.

2.3. Preparation of the extract of the aerial parts of the Mentha pulegium medicinal plant

The foxhole extraction method was used for extraction. Mix 5 grams of the aerial parts of the dried Mentha pulegium medicinal plant with methanol to make a paste. Then, the dough was poured into the desired container, and the container was placed in the machine. We add the solvent (methanol) gradually and boil it balanced. It usually takes about 8 hours to finish the work, but we have to wait for the end of the extraction until the solvent is entirely colorless at the top of the machine. After obtaining the extract, it was passed through Whatman filter paper by a vacuum pump. The extract was kept at 4°C for one week for further use.

3.RESULTS AND DISCUSSION

3.1. Scanning electron microscope (SEM) observations

A scanning electron microscope was used to examine the morphology and size of the synthesized nanoparticles. For this purpose, the samples are first coated with gold so that the resulting image has a higher resolution. The scanning electron microscope image of the synthesized sample is given in the g below. The image of the sample synthesized by sodium citrate reductant is presented. As shown in Fig 1, the size of the particles is 20.71 nanometers, and the particles have almost spherical morphology. The image related to the sample synthesized by the regenerator of Mentha pulegium extract is provided. In Fig 2, it can be seen that the size of the particles is 65.57 nanometers, and the particles have almost spherical morphology.

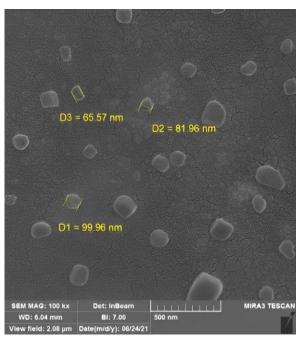


Fig. 1. SEM of gold nanoparticles synthesized by sodium citrate

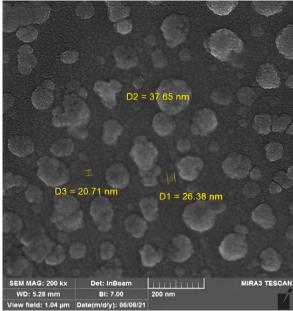


Fig. 2. SEM of gold nanoparticles synthesized by Mentha pulegium plant extract

3.2. Transmission electron microscope (TEM) observations

The TEM image and the size distribution of gold nanoparticles are shown in Fig 3. The synthesized particles have a spherical morphology and uniform size. As observed, gold nanoparticles have been synthesized by reducing sodium citrate with a size range of 10.3 nm. It is shown in Fig 4 that the synthesized particles have a spherical morphology. As observed, gold nanoparticles have been synthesized by reducing Mentha pulegium extract with a size range of 9.6 nm.

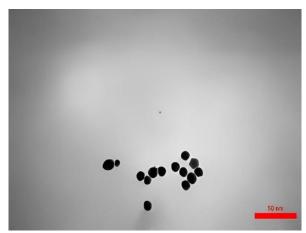


Fig. 3. TEM of gold nanoparticles synthesized by sodium citrate

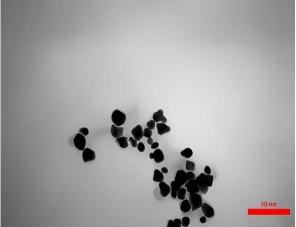


Fig. 4. TEM of gold nanoparticles synthesized by Mentha pulegium plant extract

3.3. Instrumental observations of UV-VIS spectrophotometry

UV/vis spectra of gold nanoparticles were prepared, considering that gold nanoparticles absorb light between 550 nm and 300 nm, and the absorption band of plasmon resonance at 526 nm for gold nanoparticles occurred by sodium citrate Fig 5. showed UV/vis spectra of gold nanoparticles. The absorption band of plasmon resonance at 421 nm for gold nanoparticles by Mentha pulegium extract occurred in Fig 6.

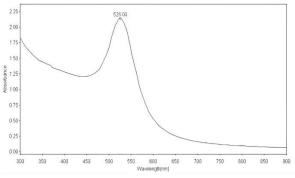


Fig. 5. UV-vis spectrum of synthesis of gold nanoparticles by sodium citrate

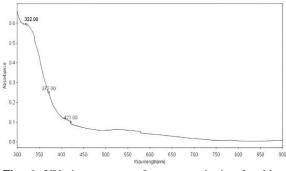


Fig. 6. UV-vis spectrum of green synthesis of gold nanoparticles by Mentha pulegium plant extract

3.4. X-ray Energy Diffraction Spectroscopy (EDX or EDS) analysis

EDX is the method used to determine the composition of the obtained product. Fig 7 shows the EDX spectrum of synthesized gold nanoparticles. Considering that the standards in this analysis were gold, the detected peak indicates the formation of gold nanoparticles, and the height of the peaks indicates the significant concentration of gold nanoparticles. The EDX spectrum of the synthesized nanoparticles shows a strong Au signal with a very weak Cl peak made of biomolecules. Chloride can also be caused by a minimal amount of plant extract remaining in the nanoparticles because chloride is an essential nutrient abundant in plants.

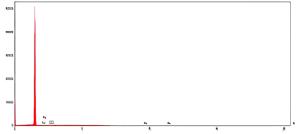


Fig. 7. Energy Diffraction X-ray EDX analysis of synthesized gold nanoparticles

3.5. Dynamic Light Scattering (DLS) Dynamic Light Scattering (DLS) method, sometimes called photon correlation spectroscopy, is used to determine the size distribution of particles in the liquid medium.

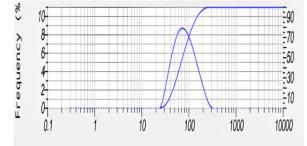


Fig. 8. Results of DLS analysis of green synthesis of gold nanoparticles by Mentha pulegium plant

The results of DLS show that the size of nanoparticles in the presence of the extract of the aerial parts of the Mentha pulegium plant is 69.9 nm, as shown in Fig 8.

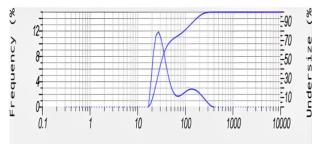


Fig. 9. Results of DLS analysis of nanoparticles synthesis by sodium citrate

In Fig 9, the results of DLS analysis of nanoparticle synthesis by sodium citrate show the size of nanoparticles in the presence of sodium citrate is 36.5 nm.

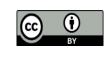
4.CONCLUSION

The results obtained in this research showed that the Mentha pulegium medicinal plant can synthesize gold nanoparticles. So far, this plant has not been reported for the biological reduction of gold ions. The green method synthesises gold nanoparticles using both the green method and the usual Torkovitch method, and by observing the analysis results, the green method provides nanoparticles with a smaller and more stable size than the Torkovitch chemical method. It is also more environmentally friendly than the Torkovitch method due to the absence of chemicals that are friendly to nature and the environment, and it will also require less cost. The production of gold nanoparticles using spectrometry) UV/Vis), transmission electron microscope (TEM). scanning electron microscope (SEM), X-ray energy diffraction (EDX), and dynamic light scattering (DLS) analysis. For UV/Vis analysis, the solution was poured into salt, considering that gold has an absorption spectrum of 300-550 nm. The plasmon resonance absorption band occurred at 421 nm for gold nanoparticles by leaf extract of Mentha pulegium medicinal plants, and the plasmon absorption band for gold nanoparticles by sodium citrate is 526 nm. The DLS pattern shows the suspension of Au nanoparticles using the extraction of the aerial parts of the Mentha pulegium plant and sodium citrate. Transmission Electron Microscope (TEM) was used to determine the place where gold nanoparticles were produced and the shape and size of nanoparticles. The particles with a spherical shape were shown on a scale of 9.6 nm. The spherical structure of gold nanoparticles with a size of 65.57 nm by scanning microscope (SEM) indicates the formation of gold nanoparticles. The results confirm that gold nanoparticles have been synthesized by leaf extract of Mentha pulegium medicinal plants.

REFERENCES

- [1]S. Iravani, Green synthesis of metal nanoparticles using plants, *Green Chem.* 13 (2011) 2638.
- [2]N. Ahmad, and S. Sharma, Green Synthesis of Silver Nanoparticles Using Extracts of Ananas comosus, *C.R.G.S.C.* 2 (2012) 141-147.
- [3]M. Shah, D. Fawcett, S. Sharma, S.K. Tripathy, and G.E. Poinern, Green synthesis of metallic nanoparticles via biological entities, *Materials* 8 (2015) 7278-7308.
- [4]V. R. Reddy Gold nanoparticles: Synthesis and applications. *Synlett* 11 (2006) 1791–1792.
- [5]X. Yang, M. Yang, B. Pang, M. Vara, and Y. Xia, Gold Nanomaterials at Work in Biomedicine, *Chemical Reviews* 115 (2015) 10410-10488.
- [6]D. Cabuzu, A. Cirja, R. Puiu, and A. Mihai Grumezescu, Biomedical applications of gold nanoparticles, *Current topics in medicinal chemistry* 15 (2015) 1605-1613.
- [7]C. N. Rao, G. U. Kulkarni, P. J. Thomas, and P. P. Edwards, Metal nanoparticles and their assemblies, *Chem. Soc. Rev.* 29 (2000) 27-35.
- [8]E. C. Dreaden, A. M. Alkilany, X. Huang, C. J. Murphy, and M. A. El-Sayed, The golden age: gold nanoparticles for biomedicine, *Chem. Soc. Rev.* 41 (2012) 2740-2779.
- [9]J. R. Peralta-Videa, Y. Huang, and J. G. Parsons, Plant-based green synthesis of metallic nanoparticles: scientific curiosity or a realistic alternative to chemical synthesis?, *Nanotechnol. Environ. Eng.* 2 (2016) 1, 4.
- [10]T. Bennur, Z. Khan, and R. Kshirsagar, Biogenic gold nanoparticles from the Actinomycete Gordoniaamarae: application in rapid sensing of copper ions, *Sens. Actuators. B chem B.* 233(2016) 684-690.
- [11]J. Ramsden, Essentials of Nanotechnology, *Publishing Aps* 13 (2009).
- [12]K. Eric Drexler, Nanotechnology: From Feynman to Funding, *Bull. Sc. Technol. & Soc.* 24 (2004).

- [13]P.G. Kik, and M. L. Brongersma, Surface Plasmon Nanophotonics, surface Plasmon Nanophotonics, *Springer* (2007) 1-9.
- [14]Y. Q. He, S. P. Liu, L. kong, and Z. F. Liu, A study on the sizes and concentrations of gold nanoparticles by spectra of absorption, resonance Rayleigh scattering and resonance non-linear scattering, *Spectrochim. Acta. A Mol Biomol Spectrosc*, 61 (2005) 2861-6.
- [15]M. Giersig, and P. Mulvaney, Preparation of Ordered Colloid Monolayers Electrophoretic Deposition, *Langmuir* 9 (1993) 3408-3413.
- [16]M. Faraday, Philosophical Transactions of the Royal, Society of London (1857) 145-181.
- [17]E. Shevchenko, D. Talapin, A. Kornowski, F. Wiekhorst, J. Kotzler, M. Haase, A. Rogach, and H. Weller, Colloidal Synthesis and Self-Assembly of CoPt3, *J. Adv. Mater. Technol.* 14 (2002).
- [18]S. S. Shankar, A. Ahmad, R. Pasricha, and M. Sastry, Bioreduction of chloroaurate ions by geranium leaves and its endophytic fungus yields gold nanoparticles of different shapes, *J. Mater. Chem.* 13 (2003) 1822–1826.
- [19]I. Rasooli, M. B. Rezaei, and A. Allameh, Growth inhibition and morphological alterations of Aspergillus niger by essential oils from thymus eriocalyx and thymus x-porlock, *Food control*. 17 (2006) 359-64.
- [20]K. Narasimha Murthy, K. Soumya, and C. Srinivas, Antibacterial Activity of Curcuma Longa (Turmeric) Plant Extracts Against Bacterial Wilt Of Tomato Cansed by Ralstonia Solanacearum, *Int. J. Sci. Res.* 4 (2015) 2136-41.
- [21]A. Bouaichi, R. Benkirane, K. Habbadi, A. Benbouazza, and E. H. Achbani, Antibacterial activities of the essential oils from medicinal plants against the growth of pseudomonas savastanoi PV. Savastanoi causal agent of olive knot, *J. Agric. Vet. Sci.* 8 (2015) 41-5.
- [22]H. S. Elshafie, S. Sakr, S. M. Mang, S. Belviso. V. De Feo, and I. Camele, Antimicrobial activity and chemical composition of three essential oils extracted from Mediterranean aromatic plant, *J. Med. Food.* 19 (2016) 1096-103.



COPYRIGHTS

© 2022 by the authors. Lisensee PNU, Tehran, Iran. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution 4.0 International (CC BY4.0) (http://creativecommons.org/licenses/by/4.0)

سنتز سبز و خصوصیات نانو ذرات طلا با استفاده از عصاره آبی گیاه دارویی پونه (Mentha pulegium)

پوران پورحکاک*، شکوفه امیدی،، مهدی یاراحمدی

۱- بخش شیمی، دانشگاه پیام نور، تهران، ایران
E-mail: <u>p pourhakkak@pnu.ac.ir</u>

تاریخ دریافت: ۲۳ آبان ۱۴۰۲ تاریخ پذیرش: ۲۲ اسفند ماه ۱۴۰۲

چکیدہ

در این تحقیق تنایج سنتز سبز نانوذرات طلا با عصاره آبی گیاه دارویی پونه با روش شیمیایی معمول به نام تورکوویچ مورد مقایسه و تجزیه و تحلیل قرار گرفت. در روش سبز، از عصاره اندام هوایی گیاه دارویی پونه (Mentha pulegium) به عنوان عامل احیا کننده و در روش شیمیایی از سدیم سیترات استفاده شد. در هر روش سبز، از عصاره اندام هوایی گیاه دارویی پونه (Mentha pulegium) به عنوان عامل احیا کننده و در روش شیمیایی از سدیم سیترات استفاده شد. در هر دو روش سبز، از عصاره اندام هوایی گیاه دارویی پونه (Mentha pulegium) به عنوان عامل احیا کننده و در روش شیمیایی از سدیم سیترات استفاده شد. در هر دو روش TEM SEM ندام هوایی گیاه دارویی پونه (DLS تلک UV-VIS و SEM در سنتز سبز نانوذرات طلا با استخراج اندامهای هوایی گیاه دارویی پونه اندازه ذرات، کوچکتر و پایدارتر از روش تورکویچ بوده و مورفولوژی تمام نانوذرات کروی و منظم است. اندازه نانو ذرات سنتز شده روش ترکویچ بوده و مورفولوژی تمام نانوذرات کروی و منظم است. اندازه نانو ذرات سنتز شده با عصاره گیاه پونه ۶/۹ نانومتر است. نتایج UV-VIS با UV-VIS با سنتز سبز نانوذرات طلا استخراج اندامهای هوایی گیاه دارویی پونه اندازه نانوذرات سنتز شده با عصاره گیاه پونه ۶/۶ نانومتر است. نتایج UV-VIS به و پایدارت انور ترای نانوذرات طلای سنتز شده با عصاره گیاه پونه ۶/۹ نانومتر است. نتایج UV-VIS با منور ان عامل در یا تورزات طلای سنتز شده با عصاره گیاه پونه را با کنری ترای داد. فعالیت ضد با کتریایی نانوذرات طلای سنتز شده در برابر باکتری و و در با در ناحیه ۲۲۲ نانومتر برای نانوذرات طلای سنتز شده در برابر باکتری در ای و در با کنری کانومتر برای نانوذرات طلای سنتز شده در برابر باکتری داد. فعالیت ضد با کترای دانون در بالای سنتز شده در برابر باکتری داد. فعالیت ضد با کنور در بالای سنتز شده در برابی در در باکتری ای در از کنونی در برای در در سری قرار گرفت.

کلید واژه ها سنتز سبز؛ گیاه، پونه؛ نانوذرات طلا.