Article

Microwave assisted green synthesis of silver nanoparticles using Mulberry leaves extract and silver nitrate solution

Liem Le 1,*, The Nguyen 2 and Dieu Nguyen 2

- ¹ Office of Academic Investigation, Duy Tan University, Vietnam
- ² Faculty of Natural Sciences, Duy Tan University, Vietnam; <u>nguyenphuocthe@dtu.edu.vn</u> (T.N.); <u>nguyentxuandieu@dtu.edu.vn</u> (D,N.)
- * <u>lengocliem@dtu.edu.vn</u>; Tel.: +84915995376

Abstract: In this work, silver nanoparticles (AgNPs) were synthesized rapidly and eco-friendlily using the extract of Mulberry leaves and aqueous solution of silver nitrate without any toxic chemical [1,2]. The Mulberry leaves extract acts as both reducing agent and stabilizing agent. The UV-Vis spectrum shows peak at 430 nm. The TEM image of synthesized AgNPs sample shows spherical shaped particles whose size range from 15 to 20 nm. TEM image of nano silver solution sample synthesized by microwave assisted method shows nearly spherical particles with an average particle size of 10 nm. The absorption UV-vis spectrum of silver nanoparticles synthesized by microwave assisted method (AgNPsmw) shows a sharp absorption band around 415 nm. After two month storage of AgNPsmw, the absorption spectrum of AgNPsmw was taken again. The UV-Vis spectrum shows negligible peak changes of silver nanoparticles have occurred after two months of storage. The synthesized AgNPs material could be used as an antimicrobial, used in the field of textile and in wastewater treatment.

Keywords: silver nanoparticles, Mulberry leaves extract.

I. Introduction

Nanostructured materials have many application in our daily life because of their special physico-chemical properties that could not be found in other material of larger scale. Among materials, nano silver has been most widely applied in our daily life. Because of its anti-bacteria properties, nano silver has been extensively used in the field of medical, agriculture and in wastewater treatment, etc[1-7]. Nanomaterial could be synthesized by many methods. However, synthesizing nano-silver using mulberry leaves extract as reducing agent not only offers many advantages but also use less chemical, thus reduce the pollution to the environment. In this work, mulberry leaves extract had been used as reduction agent in synthesizing nano silver (AgNpsmw) by microwave assisted method using as reducing agent which make up a highly effective and eco-friendly method.

II. Experimental

1. Preparation of mulberry leaves extract and silver nitrate solution

Mulberry leaves were collected from a garden house in Hoi An. Leaves collected must be intact, either too young or too old. Fresh leaves were then cleaned with water then laid out to dry in the air. Weight 10 g of fresh leaves, cut into thin strips, then put into a 200 ml glass heat-resistant flask.

Boil with distilled water in 5 minutes, let it cool and filter the mixture with Whatman filter paper using vacuum filter[4-8]. The mulberry leaves extract had light yellow color. Store the extracted solution in a fridge for further use. Dissolve silver nitrate solution made in China with distilled water to get 4.10^{-3} M aqueous AgNO₃ solution.



Figure 1. Picture of Mulberry leaves

2. Synthesis of nano silver material

2.1. Non-microwave asissted systhesis of nano silver

Table 1: Synthesized samples

AgNO ₃ solution (ml)	Mulberry leaves extract (ml)	Samples
0	10	\mathbf{M}_0
40	0	M 1
40	1	M ₂
40	3	M 3
40	5	M ₄
40	6	M 5
40	7	M 6
40	8	M 7

Visual observation and UV-vis spectral:

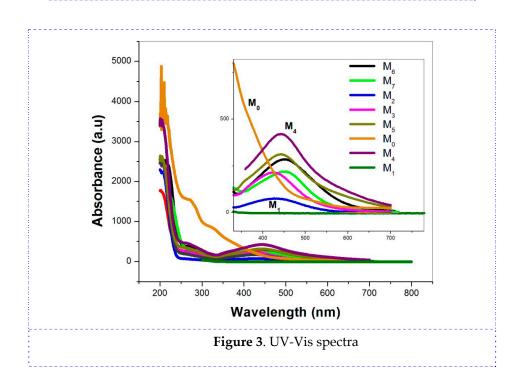
Mix AgNO $_3$ solution and mulberry leaves extract with the ratio shown in Table 1. Put each mixture on a shaker machine and stir for 30 minutes. It was noticed that except for samples M_0 and M_1 , the

color of all other mixtures changes from light brown to dark red. The color change in each sample varies according to the mixing ratio between AgNO₃ concentration and volume of mulberry leaves extract solution as shown in Figure 2. The observation showed that nano silver particles were formed.

Characterization Techniques: To investigate the optical properties and microstructure of nano silver material, we measured the UV-vis absorption spectra were used GE Ultrospec 7000 UV vis spectrophotometer, Transmission Electron Microscope (TEM) analysis of silver nanoparticles analysis was done using JEOL JEM 1010, The X-ray difraction spectra were measured by the diffractometer Bruker D8-Advance, FTIR Spectra for mulberry leaves extract was obtained in the range 400-4000 cm⁻¹ with IR-Prestige-21 Shimaduz FTIR spectrophotometer.



Figure 2. Photograph color change of colloids



Before taking UV-vis spectrum, each samples were dissolved 40 times. The UV-vis spectrum of the material shows a strong plasmon resonance band centered at 430 nm. From sample M2 to M4 the absorbance peak intensity increase as we increase the volume of mulberry leaf extract from 1 ml to 5 ml. For samples whose volume of mulberry leaves extract were more than 5 ml, the peak intensity decrease as the content of mulberry leaves extract increase. Combining spectral information with qualitative observation, it can be concluded that AgNps has been formed when mixing AgNO3 solution with mulberry leaves extract. Among our samples, the M4 not only showed the highest peak intensity value but also had the red darkest color. The observations indicate that it has the most AgNps particles formed. In can also be infered that, at high concentration of extract, the rate of AgNps production is so fast that it prevent the formation of protective layer between partices. Because of this, the aggregation phenomenon between particles occurs, increasing the particle size. The increased particle size reduced peak intensity and shift the peak to longer wavelength (as in M₅, M₆ and M₇ samples). Absorption spectrum of AgNO₃ aqueous solution, shown in olive colored line in Figure 3, has no peak in the measuring range. Absorption spectrum of mulberry leaves extract, shown in orange line in Figure 3, has absorption peak in the short wavelength region due to organics substances found in the extract solution and has no peak at wavelengths longer than 400 nm. The spectral information indicates that AgNps particles were formed only when AgNO3 is mixed with mulberry extract. The magnified from 380 to 700 nm of absorption spectrum (Figure 3), shows discrete lines.

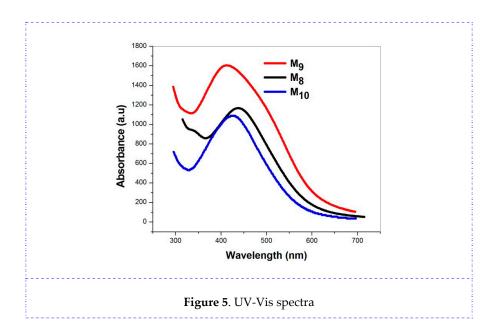
2.2. Microwave asissted systhesis of nano silver.

Visual observation and UV-vis spectral:



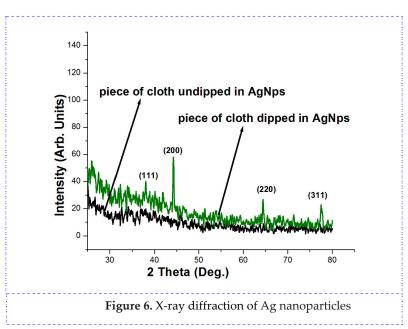
Figure 4. Ms: non-microwave assisted Ms: microwave assisted

Mix 50 ml of AgNO $_3$ solution with 6 ml of mulberry leaves extract, and divide into 2 equal portion. Put the first portion (M $_8$) on a shaker machine and stir for 30 minutes. The other portion (M $_9$) was heated in a microwave for one minute. The changed color of M $_9$ from light yellow to dark red within one minute in the microwave indicates that the efficiency of (AgNpsmw) synthesis by microwave assisted method was higher than that of non-microwave assisted method. The samples's color are shown in Figure 4.



The UV-vis spectra (Figure 5) shows that absorption intensity at around 415 nm of M₉ is higher than that of M₈. After being left alone for 2 months, M₉ sample were renamed M₁₀. Compared to spectrum of M₉, the UV-vis spetrum taken for M₁₀ (the blue line of Figure 5) shows a slight decrease in the peak intensity and the peak shift of 5 nm toward longer wavelength. The above observations indicated that the synthesized colloid solution is highly stable.

2.3. X-ray diffraction (XRD) Studies

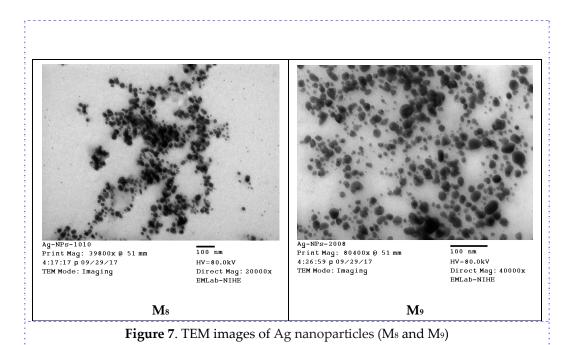


Cloth was cut into 25 cm² pieces and dipped in a colloids solution AgNps for 10 minutes, and another piece of cloth undipped in a colloids solution AgNps . The XRD pattern of the cloth dipped in a colloids solution AgNps and the cloth undipped in colloids solution AgNps are shown in Figure 6. The XRD pattern of the undipped cloth show no the characteristic peak of silver, while the XRD pattern of the cloth was dipped in the colloids AgNps shows the characteristic peak of silver. Four main characteristic diffraction peaks for Ag were observed at $2\theta = 38.2$, 44.1, 64.5, and 77.6° , which correspond to the (111), (200), (220) and (311) crystallographic planes of face-centered

cubic (fcc) Ag crystals, respectively (JCPDS 00-004-0783)[9]. Because the AgNps concentration was low and the cloth's internal structure had lots of empty spaces, not many AgNps particles were deposited onto the cloth surface, thus the peaks intensity are low.

2.4. Transmission electron microscope (TEM) analysis

The size and shape of the AgNPs was further confirmed by TEM analysis. The TEM image of M_8 , show relatively uniform spherical particle size, ranging from 15-20 nm, the TEM image of M_9 show particle size about 10 nm. The particle size was more uniform and no sign of nanoparticle clustering was observed.



2.5. FT-IR spectrum

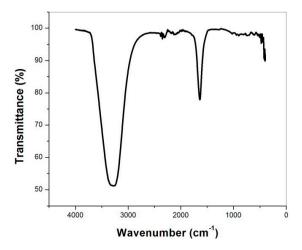


Figure 8. FTIR spectrum of mulberry leaves extract

Drop 0.5 ml of the AgNO₃ solution to 50 ml of mulberry leaves extract to prevent the extracted solution from rotting before taking spectrum for mulberry leaf extract. A FT-IR spectrum of synthesized silver nanoparticles by this green method is shown in Figure 8. A number of absorption peaks at 3261 cm⁻¹ and 1637 cm⁻¹. The peaks at 3261cm⁻¹ corresponds to O-H and N-H bond, the peak at 1637cm⁻¹ corresponds to C=O bond, indicating the biomaterial bind to the silver nanoparticles through amine and C=O of amide I and amid II of the protein[1,6,7]. Thus indicating mulberry leaves extract act as a reducing and stabilizing agent for silver particles.

III. Conclusion

In this work, silver nanoparticles (AgNPs) colloid solution has been successfully synthesized, using Green and eco-friendly method. This is a quick, highly effective and less chemical-consuming method. The Microwave assisted green synthesis method is more effective than non-microwave assisted method. The UV-Vis spectrum of AgNPs has absorbance peak ranging from 425 – 435 nm. Nano-silver particles are spherical shaped with size ranging from 15 to 20 nm. UV-vis spectrum of AgNpsmw shows peak at 415 nm, with an average particle size of 10 nm. The slight change of peak position after 2 month storage of AgNPsmw indicates that some nano-particles in the colloid solution had clumped together, but trivial. Synthesizing silver nanoparticles by mulberry leaves extract is eco-friendly and good antimicrobial efficiency against bacteria, viruses and other microorganisms[10] and used in wastewater treatment.

Funding: This research received no external funding

References

- [1] Yuet Ying Loo, Buong Woei Chieng, Mitsuaki Nishibuchi and Son Radu 2012 Synthesis of silver nanoparticles by using tea leaf extract from Camellia Sinensis. *International Journal of Nanomedicine*. 4263-4267.
- [2] N Krishnakuma, K Adavallan 2014 Mulberry leaf extract mediated synthesis of gold nano particles and its anti-bacterial activity against human pathogens. *Advances in Natural Sciences:* Nanoscience and Nanotechnology 5 025018.
- [3] Jaiswal S, Duffy B, Jaiswal AK, Stobie N, McHale P 2010 Enhancement of the antibacterial properties of silver nanoparticles using β-cyclodextrin as a capping agent. *Int J Antimicrob Agents*. 36(3) pp 280–283
- [4] Sun Q, Cai X, Li J, Zheng M, Chen Z, Yu C-P 2014 Green synthesis of silver nanoparticles using tea leaf extract and evaluation of their stability and antibacterial activity. *Colloids Surf Physicochem Eng Asp.* 5;444 pp 226–231.
- [5] Sajeshkumar N.K, Prem Jose Vazhacharickal, Jiby John Mathew and Anupa Sebastin 2015 Synthesis of silver nano particles from curry leaf (murraya koenigii) extract and its antibacterialactivity CIBTech Journal of Pharmaceutical Sciences. Vol.4 pp15-25.
- [6] Akl M. Awwad1 and Nidà M. Salem. Green synthesis of silver nanoparticles by Mulberry leavesextrac 2012 *Nanoscience and Nanotechnology*. pp 125-128.
- [7] Akl M. Awwad, Nidà M. Salem, Ammany O. Abdeen 2013 Biosynthesis of silver nanoparticles using Loquat leaf extract and its antibacterial activity. *Advanced materials letters*. 4(5) pp 338-342.

- [8] Jagpreet Singh, Navalpreet Singh, Aditi Rathi, Deepak Kukkar and Mohit Rawat 2017 Facile Approach to Synthesize and Characterization of Silver Nanoparticles by Using Mulberry Leaves Extract in Aqueous Medium and its Application in Antimicrobial Activity. *J Nanostruct* 7(2).pp134-140.
- [9] Siby Joseph and Beena Mathew 2014 Synthesis of Silver Nanoparticles by Microwave irradiation and investigation of their Catalytic activity. *Research Journal of Recent Sciences*. pp 185-191
- [10] G. Dhivya and M. Rajasimman 2015 Synthesis of silver nanoparticles using Momordica charantia and its applications. *Journal of Chemical and Pharmaceutical Research* 7(9):107-113