IOSR Journal of Engineering (IOSRJEN) **www.iosrjen.org**
ISSN (e): 2250-3021, ISSN (p): 2278-8719
Vol. 11, Issue 6, June 2021, ||Series -II|| PP 01-06

Synthesis of silver nanoparticles capped by Oleylamine, application for conductive film production

My Kieu Thi Truong , Dung My Thi Dang, Mai Quynh Thi Tran, Chien Mau Dang

Institute for Nanotechnology (INT), Vietnam National University - Ho Chi Minh City
Corresponding author: My Kieu Thi Truong
Received 10 June 2021; Accepted 24 June 2021

Abstract: This article presented the preparation of silver nanoparticles from $AgNO_3$ salts with reducing agents ascorbic acid and capping agent Oleylamine by wet chemical method at 60^0C. The reduction of Ag^+ into Ag was monitored by UV-Vis absorption spectroscopy method. The morphology and particle size were analyzed by transmission electron microscopy (TEM) and Dyamic Light Scattering (DLS). The operability of capping agent Oleylamine was confirmed by infrared absorption spectroscopy (FT - IR). The results showed that silver nanoparticles were prepared with uniform particle size, 6 – 7 nm, at 410 nm.
Keywords: silver nanoparticles, wet chemical method

I. INTRODUCTION

In recent years, nano-silver has received the attention of many researchers, because silver is a metal with high electrical conductivity, anti-oxidation and good corrosion resistance [1]. In addition, nano-size metal has more special properties than block metal, such as conductivity, melting temperature and light absorption ability [2], [3]. One of the important applications of nano-silver is the nano-silver ink applied to inkjet technology of electronic circuits [4]. Therefore, electrical conductivity, stability and antioxidant are important factors to assess the quality of ink as well as silver nanoparticles. Hence, the requirement is that synthetic nano-silver has to have high electrical conductivity, small particle size, and stable distribution in solvent and good oxidation resistance [5].

There have been many methods to synthesize nano-silver. In particular, wet chemical method using surfactants (or capping agents) have shown many outstanding advantages [6]. This method is quite simple, does not require sophisticated equipment, easy to control the reaction conditions to obtain the desired particle size, and high yields.

The reducing agent for this method is quite diverse and is a factor that determines the size and shape of the particle. Ascorbic acid is a reducing agent, which is not too strong, helps to avoid too quick reaction which results in particle agglomeration, short time, low temperature and small particle size [7]. Moreover, the presence of capping agent is a key role to adjust the size of silver particles by controlling the growth of silver particles and creating a protective layer that prevents their coagulation. The capping agents are often studied and used in preparation nano silver such as: Polyvinylpyrrolidon (PVP) [8], [9], Polyethyleneglycol (PEG) [10], Polyvinylalcohol (PVA) [8]. However, the nano particles were synthesized with the bulk polymer protective layer, were limited the conductive capacity. Furthermore, polymers with hydrophilic heads, increasing the risk of oxidation of silver nanoparticles. Thence, the capping agents with amine tails have been noticed with many advantages: good oxidation resistance, weak bonding with the other nano-metal, so it is easy to separate and increase the conductivity.

II. EXPERIMENTAL

Materials and methods

$AgNO_3$ (M_W=169,87 g/mol, Merck), L-Ascorbic acid 99% $C_6H_8O_6$ (M_W=176,12 g/mol, Sigma Aldrich), Toluene C_7H_8 (M_W=92,14 g/mol, d=0,87 g/ml, Fisher), Oleylamine 70% $C_{18}H_{37}N$ (M_W=267,49 g/mol, d=0,813 g/ml, Sigma Aldrich), Acetone 99,5% C_3H_6O (M_W=58,08, d=0,791 g/ml, Xilong).

The equipment used for preparation of silver nano-particles: hot plate stirrer, centrifuge Hermle Z326, ultrasonic tank EM 30HC (Germany).

Analyze equipment: UV-Vis Spectrometer (Cary 100 Varian, USA), DLS instrument (LB-550, Horiba, Japan), Transmission electron microscopy (JEOL JEM – 1010, USA), X-ray diffractometer (SU8010, Hitachi, Japan), FT-IR Spectrometer (TensorTM 37, Brucker, USA).

Procedure

Synthesis silver nano-particles capped by Oleylamine in Toluene solution was developed, based on report of K. Shirevas et al,. [11]. Therein, ascorbic acid played as the reducing agent. The reaction of nano particles formation could occur at low temperature (60^{o}C) and short reaction time (8h). First, 15ml Toluene and 4.4 mL Oleylamine were prepared in Schott flask, stirred well and heated to 60^{o}C. Then, as 300 mg $AgNO_3$ was added slowly in the flask, mixture color turned from light yellow, to orange, brown and dark brown in 2 minutes. This mixture was stirred in 8h at 60^{o}C. During the reaction, the mirror coating phenomena could appear on the wall of flask. After the end of reaction, added 20 ml acetone in the nano-silver mixture. The product were washed with acetone by centrifugation, and then dried at ambient temperature. Finally, obtained powder was smooth and navy blue.

III. RESULTS AND DISCUSSION

Each type of metal nano-particles has the characteristic oscillation of valence electrons when absorbs rays with different wave length in UV-Vis zone [12]. This phenomenon called the surface plasmon resonance. Based on this theory, the presence of metal nano-particles is determined by UV-Vis spectrometer. The surface plasmon resonance peak changes when the particle size changes, because the scattering of their valence electrons changes. For silver nano-particles, the characteristic peak responses at 390 nm to 420 nm. The closer the peak to 420 nm, the bigger the size of silver nano-particles .

The silver nano-particles were prepared in toluene solution with the concentration 0.02 mg/ml for UV-Vis analysis. The result was shown in figure 1, the synthesized silver nano-particles absorbed at wave length of 410 nm, this is the characteristic absorption peak of silver nano-particles size at the range 10 – 40 nm [13].

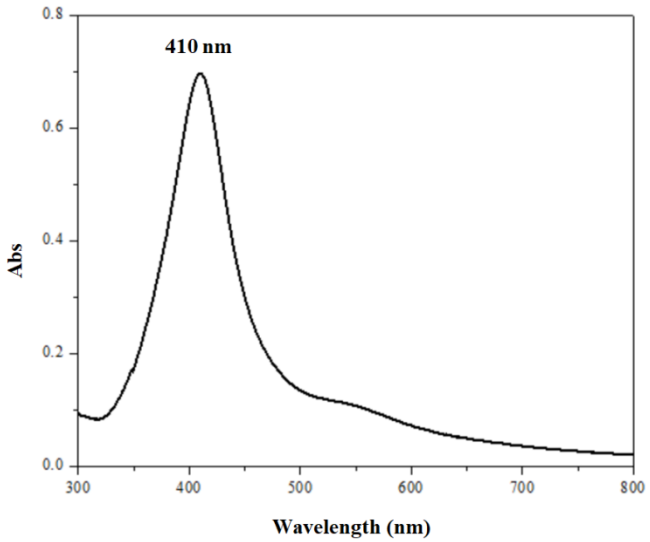

Figure 1: UV – Vis spectrum of nano-silver solution.

The nano-particle size was determined by DLS instrument, and the result was presented in figure 2. The average size of silver nano-particles was 11±3 nm, accordance completely with UV-Vis result. Moreover, the size difference was relative small; it indicated that the synthesis silver nano-particle size was evenly distributed. However, DLS method just gave the preliminary results, because this result was taken from many conversion formulas, causing high error.

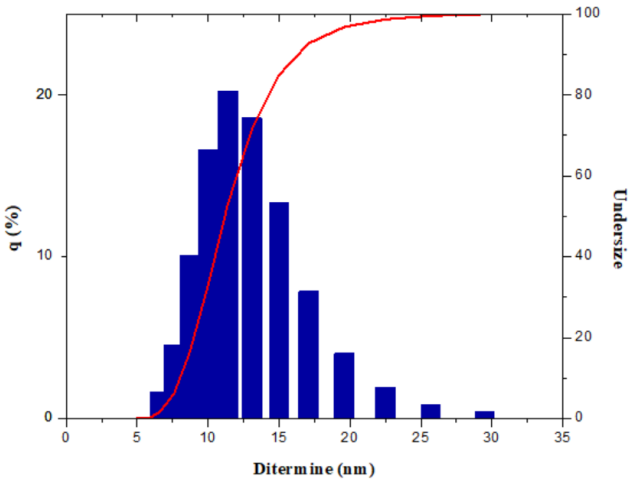

Figure 2: DLS result.

For observation the form and precise size, the silver nano-particles were studied by TEM, the result was demonstrated in Figure 3. ImageJ software was used to determine the size distribution of nano-particles based on the TEM images; the distributed chart was presented in Figure 4. This results shown that the synthesis nano-silver had the sphere form, small size, separation and congruence. The average size was 6.5±0.5 nm.

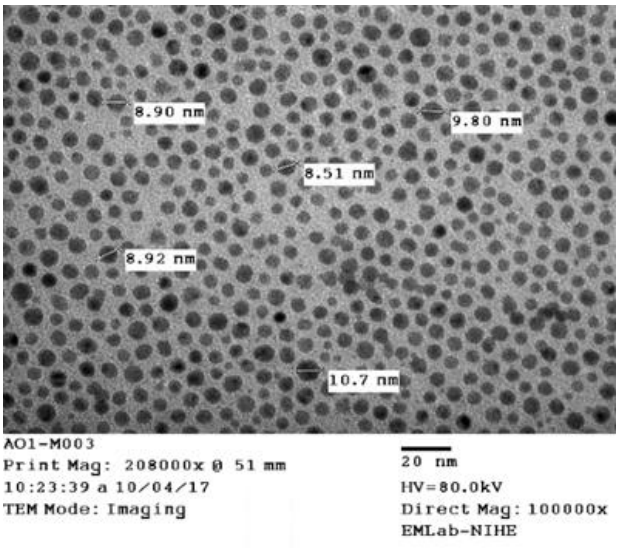

Figure 3: TEM image of the silver nano-particles with capping agent Oleylamine.

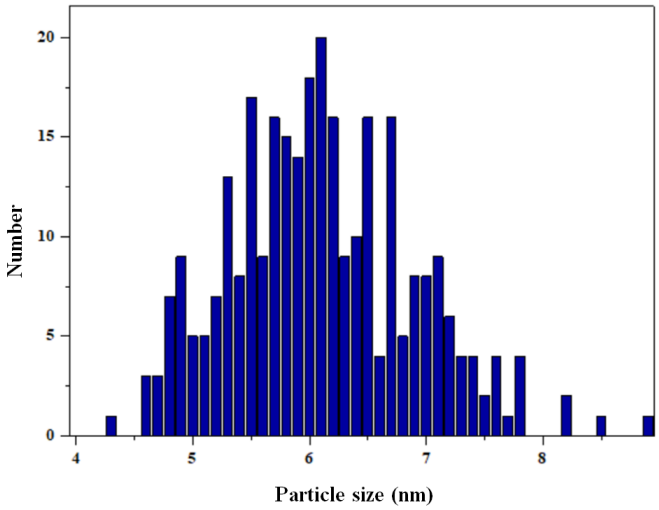

Figure 4: The distribution chart of nano-particle size.

EDS was used also to determine the relative quantity of every element in the nano-silver sample (figure 5). There was the appearance of the characteristic peaks of Ag, C and N elements. Therein, Ag and C were two elements which had the highest concentration. C and N were belong to the capping agents, in which C was the principal element. Furthermore, the EDS result indicated that it did not have the existence of O element, so there was not Ag_2O in the sample. Thus, the reducing reaction of silver nano-particles formation was occurred completely.

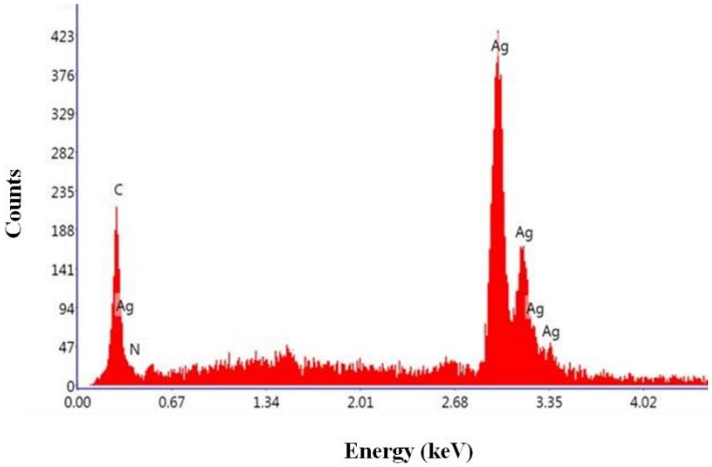

Figure 5: EDS result.

To confirm the presence of the protection layer Oleylamine on the silver nano-particle surface, the silver sample was analyzed by FT-IR. The spectrum (figure 6) demonstrated that there were the characteristic peaks of amine group (-NH_2) at 3300 cm^{-1} and 1590 cm^{-1}. The strong absorption peaks at 2954 cm^{-1} and 2927 cm^{-1} were the symmetric and asymmetric vibration of –CH_2 group. The peak at 1650 cm^{-1} of the C=C vibration could not observe obviously because of the baseline noisy [11].

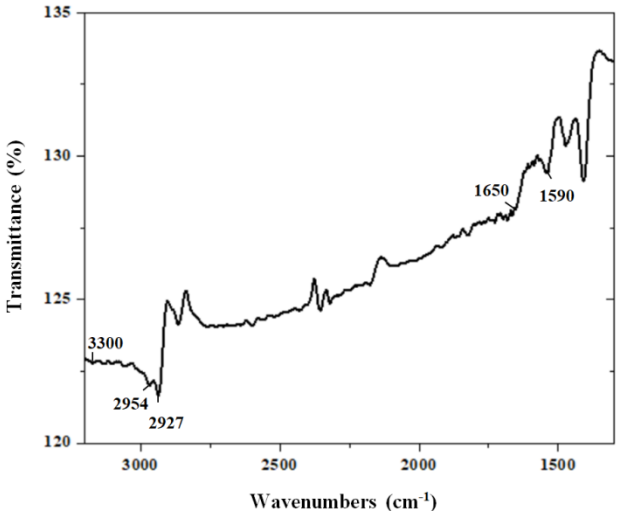

Figure 6: FT-IR spectrum of nano-silver sample.

The conductive capacity of silver nano-particles was determined. The synthesis nano-particles were dispersed in Toluene solution with the concentration 100 mg/ml, shaken well and put in the ultrasonic in 30 minutes. The solution was dripped on the glass substrate, and heated at 200^0C in 30 minutes. The obtained membrane was thin, polished, fastened well on the substrate. The electric resistance was 0.5-1 Ω.

Figure 7: Silver membrane.

IV. CONCLUSIONS

The silver nano-particles were synthezied successfully by wet chemical method with ascorbic acid as the reducing agent, and Oleylamine as the capping agent, in Toluene solution. The synthesis nano-particles had the sphere form and congruence. The average particle size was 6.5±0.5 nm. There was not presence of Ag_2O, so the reaction was conducted completely at 60^oC, in 8h. The conductive result test shown good conductive capacity, with electric resistence 0.5-1 Ω. With many advantages, the synthesis silver nano-particles were suitable to apply in the conductive membrane. In addition, product was in powder and easy to conserve and adjust quantity of silver in ink.

ACKNOWLEDGEMENTS

The authors highly appreciate the financial support by FIRST Central Project Management Unit Grant Agreement No.: 09/FIRST/2a/INT.

REFERENCES

[1]. L. Huang, Y. Huang, J. Liang, X. Wan, Y. Chen (2011), "Graphene - Based conducting Inks for direct inkjet printing of flexible conductive patterns and their applications in electric circuits and chemical sensors", Nano Research, 4, pp.675-684.

[2]. V. Volkman, S.K. Pei, Y. Redinger, D. Yin, S. and Subramanian (2004), "Ink-jetted Silver/Copper conductors for printed RFID applications", Materials Research Society Symposium Proceedings, 814, pp. I7.8.1-6.

[3]. D. Kim, S. Jeong,S. Lee, B.K. Park, and J. Moon (2007), "Organic thin film transistor using silver electrodes by the ink-jet printing technology", Thin Solid Films, 515, pp.7692-7696.

[4]. H.H. Lee, K.S. Chou (2005), Nanotechnology 16, pp.2436

[5]. A. Kosmala, R. Wright, Q. Zhang, P. Kirby (2011), "Synthesis of silver nanoparticles and fabrication of aqueous Ag ink for inkjet printing", Materials Chemistry and Physics, 129, pp.1075-1080.

[6]. R. Janardhanan, M. Karuppaiah, N. Hebalkar, T.N. Rao (2009), "Synthesis and surface chemistry of nano silver particles", Polyhedron, 28, pp.2522-2530.
[7]. P.K. Sahoo, S.S.K. Kamal, T.J. Kumar, B. Sreedhar, A.K. Singh, S.K. Srivastava (2009), "Synthesis of silver nanoparticles using facile wet chemical route", Defence Science Journal, 59(4), pp.8469-8474.
[8]. B.A. Zielinska, E. Skwarek, A. Zaleska, M. Gazda, J. Hupka (2009), "Preparation of silver nanoparticles with controlled particle size", Procedia Chemistry, 1, pp.1560-1566.
[9]. D. Malina, A.S. Kupiec, Z. Wzorek, Z. Kowalski (2012), "Silver nanoparticles synthesis with different concentration of polyvinylpyrrolidone", Digest Journal of Nanomaterials and Biostructures, 7(4), pp.1527-1534.
[10]. K. Shameli, M.B. Ahmad, S.D. Javayeri, S. Sedaghat, P. Shabanzadeh, H. Jahangirian, M. Mahdavi, Y. Abdollahi (2012), "Synthesis and characterization of polyethylene glycol mediated silver nanoparticles by the green method", International Journal of Molecular Science, 13, pp.6639-6650.
[11]. A. Ghosale, R. Shankar, V. Ganesan, K. Shrivas (2016), "Direct – writing of paper based conductive track using silver nano - ink for electroanalytical application", Electrochimica Acta, 209, pp.511-520.
[12]. S. Link and M.A. El-Sayed (2000), "Shape and size dependence of radiative, non-radiative and photothermal properties of gold nanocrystals", International Reviews in Physical Chemistry, 19, pp.447-455.
[13]. D. Paramelle, A. Sadovoy, S. Gorelik, J. Hobley, D.G. Fernig (2014), "A rapid method to estimate the concentration of citrate capped silver nanoparticles from UV - Visible light spectra", Analyst, 139, pp.4855.

My Kieu Thi Truong, et. al. "Synthesis of silver nanoparticles capped by Oleylamine, application for conductive film production." *IOSR Journal of Engineering (IOSRJEN)*, 11(06), 2021, pp. 01-06.