

Synthesis and Characterization of Silver Oxide Nanoparticles by a Novel Method

Ng Law Yong, Akil Ahmad, Abdul Wahab Mohammad

Abstract— Silver oxide (Ag_2O) nanoparticles were synthesized through a simple chemical method using silver nitrate (AgNO_3) in the presence of polyethylene glycol (PEG) as reducing agent. In current study, synthesis process by maintaining the solution pH using sodium hydroxide (NaOH) solution was investigated and compared. Zetasizer Nano series, X-ray diffraction (XRD) analysis, and Fourier transform infrared spectroscopy (FT-IR) studies were performed to confirm the size distribution measurement, chemical compound composition, crystallographic structure and functional group corresponding to Ag-O. Silver nanoparticles synthesized in the form of silver oxide were confirmed by the XRD study.

Index Terms— Fourier transform infrared spectroscopy, Nanoparticles, Polyethylene glycol, Silver oxide, Size distribution, Crystallographic study, X-ray diffraction analysis

1 INTRODUCTION

IN recent years, nano-sized metal particles have got much attention due to its unique optical, electrical and magnetic properties, which depends on the size and shape of the particle. Silver oxide (Ag_2O) nanoparticles is a well-known material having vast applications in the field of oxidation catalysis [1, 2], sensors [3], fuel cells [4], photovoltaic cells [5], all-optical switching devices, optical data storage systems [6] and as a diagnostic biological probes [7].

Many physical and chemical properties including luminescence, conductivity, and catalytic activity depend upon the size of nanoscale materials. The most common and simplest bulk-solution synthetic method that has been used for the preparation of metal nanoparticles is chemical reduction of metal salts. For the synthesis of nanoparticles we generally use the soluble metal salt, a reducing agent, and a stabilizing agent (caps the particle and prevents further growth of the particle or aggregation).

It was, therefore, thought worthwhile to prepare Ag_2O nanoparticles for its various applications through simple chemical modification using polyethylene glycol (PEG), sodium hydroxide (NaOH) and silver nitrate (AgNO_3).

2 MATERIALS AND METHODS

The silver oxide (Ag_2O) particles were produced using starting materials such as polyethylene glycol (PEG) of molecular weight 6000 (supplied by Sigma-Aldrich), silver nitrate (AgNO_3) salt (supplied by Sigma-Aldrich) and sodium hy-

periment for solution preparation, cleaning purposes or other usages were produced using Millipore RiOs 3 water purification system.

2.1 Synthesis of Silver Oxide Particles

In current study, wet chemical route was employed for the silver oxide particle synthesis. This study had conducted and compared two different routes of silver oxide particle synthesis. In the Route 1, the pH of the solution mixture was not controlled or adjusted. The pH of the solution mixture was allowed to change accordingly after the reaction took place. In the Route 2, the pH of the solution mixture was fixed at the range of pH 9.8 to pH 10. Addition of the NaOH solution was employed to maintain the solution pH until the completion of the reaction. Some of the details of the Route 1 and Route 2 were further discussed in the following sections.

2.1.1 Route 1

20 g of the PEG was dissolved in 1 liter of the RO water before being heated up to 50 degree Celsius. The solution was allowed to be stirred for another 1 hour to ensure all the PEG was completely dissolved to form a homogeneous solution. Aqueous PEG solution obtained was then filtered to remove impurities, if any. Silver nitrate solution prepared using 0.5 g of silver nitrate salt was added into the PEG solution prepared under a constant stirring rate and at constant temperature of 50 degree Celsius. pH of the solution was not controlled. The solution was continuously stirred for 1 hour to complete the chemical reactions. After the formation of the particles, the solution was filtered through the filter paper to separate the particles from the mother solution. Particles obtained were rinsed with RO water several times before it was rinsed again using ethanol. The particles were dried in oven at 60 degree Celsius for overnight.

2.1.2 Route 2

All the steps employed in the Route 2 were similar to that of Route 1, except where the addition of the silver nitrate solu-

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dioxide (NaOH) pellets (supplied by Merck). All of the chemicals were of analytical grades and used without further purification. Reverse osmosis (RO) water used throughout the ex-

tion was followed by the pH adjustment. The solution mixture pH was set at pH 9.8 to 10 throughout the reaction process. During the pH adjustment, NaOH solution of 0.1 M was slowly added into the solution mixture when the solution pH was reducing.

2.2 X-ray Diffraction Analysis

The particles formed from both Route 1 and Route 2 was characterized using X-ray diffraction analysis. BrukerD8 Advance X-ray diffractometer, Germany, was employed to provide X-ray diffraction patterns of the particles.

2.3 Size Distribution Analysis

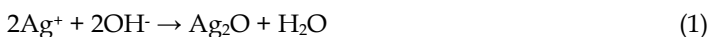
Zetasizer Nano series was used for the size distribution measurement. Water has been used as dilution medium and polar dispersant in the measurement of particle size distribution. Refractive index of water was set as 1.330 with the viscosity 0.8872 cP. Temperature of the measurement was conducted at 25 °C.

2.4 Fourier Transform Infrared (FTIR) Spectroscopy

Nicolet 6700 FTIR spectrometer (Thermo Fisher Scientific Inc., MA) with a DTGS KBr detector and a KBr beam splitter was used for FTIR analysis. All spectra were obtained from 32 scans at a 4.00 cm⁻¹ resolution with wave numbers ranging from 400 to 4000 cm⁻¹ and with an optical velocity of 0.6329.

3 RESULTS AND DISCUSSION

During the preparation of the silver oxide particles, the pH of the solution was monitored continuously. However, in the Route 1 method, no adjustment of the solution pH will be conducted. In the Route 2 method, NaOH solution will be introduced into the solution mixture to keep the solution pH at the range of pH 9.8 to pH 10. In both methods, the solution will change from yellowish to brownish colour to indicate that the chemical reaction took place in the solution mixture (Figure 1 (a) and Figure 1 (b)). After the completion of the chemical reaction which took place slowly, brownish black precipitates of silver oxide particles were observed to be formed in the solution mixture (Figure 1 (c)). The proposed chemical reaction according to reported study [8] was represented by the chemical equation below:



(a)



(b)



(c)

Figure 1. (a) Yellowish solution of the solution mixture at initial stage (b) brownish solution of the solution mixture at intermediate stage and (c) brownish-black silver oxide particles formed at the final stage of the chemical reaction.

3.1 X-ray Diffraction Analysis

According to the Figure 2, Route 1 method was less efficient to produce high purity of silver oxide. It consisted of other compound such as silver carbonate. This could be explained by the low concentration of hydroxide ions in the solution. The conversion of the silver ions into silver oxide was less effective in comparison to Route 2 method. However, Route 2 method was successful to produce high purity of silver oxide particles with high crystallinity. By referring to the reported case study [9], formation of metallic silver particles crystallized in the face centered cubic (fcc) structure would produce the XRD pattern which is similar to the XRD pattern produced through Route 2. Maintaining the solution pH at high alkalinity such as pH 9.8 to pH 10, high concentration of hydroxide ions can contribute to more silver oxide particles formation. Using the Scherrer formula $D = n\lambda/\beta\cos\theta$, where D is the crystallite size, n is a constant (=0.9 assuming that the particles are spherical), λ is the wavelength of the X-ray radiation, β is the line width (obtained after correction for the instrumental broadening) and θ is the angle of diffraction we have calculated the crystallite size of the Ag₂O particles. The average particle size obtained from XRD data is found to be about 37.90 nm.

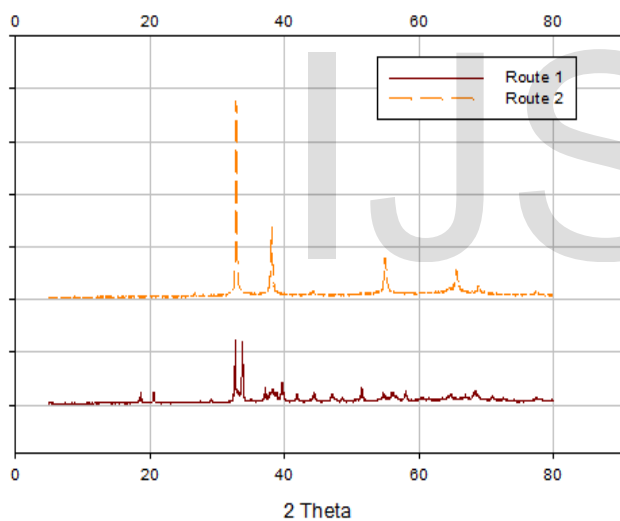


Figure 2. XRD patterns obtained for both Route 1 and Route 2 method.

3.2 Size Distribution Analysis

Size distribution analysis using silver oxide particles verified that the averaged size produced using Route 1 were 1411 nm. However, particles with smaller averaged size distribution were produced using Route 2, which was 319.6 nm. Particle sizes are normally affected by various factors such as solution pH, stirring speed, and the present of stabilizer. In Route 1, large particle sizes were obtained and these could be reasoned with its low pH after the addition of the reactant. Route 2 produced much smaller-sized particles as the pH was maintained around pH 9.8 to 10. However, the occurrence of particle agglomeration which can hardly avoided using mechanical stirring caused the final obtained particles to have sizes at around 319.6 nm. This issue has to be overcome in the near future by

controlling the stirring speed, temperature of the mixture, and the presence of stabilizer.

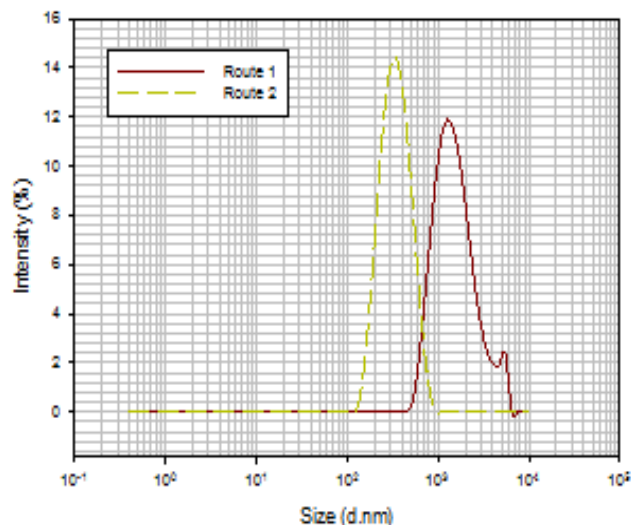


Figure 3. Particle size distribution for both particles produced using Route 1 and Route 2

3.3 Fourier Transform Infrared Spectroscopy (FT-IR) Study

As shown in Fig. 4, the intense peak appeared in the range of 513 cm⁻¹ which correspond to the stretching vibration of Ag-O group. This further verified the compound as silver oxide particles in addition to the XRD analysis conducted.

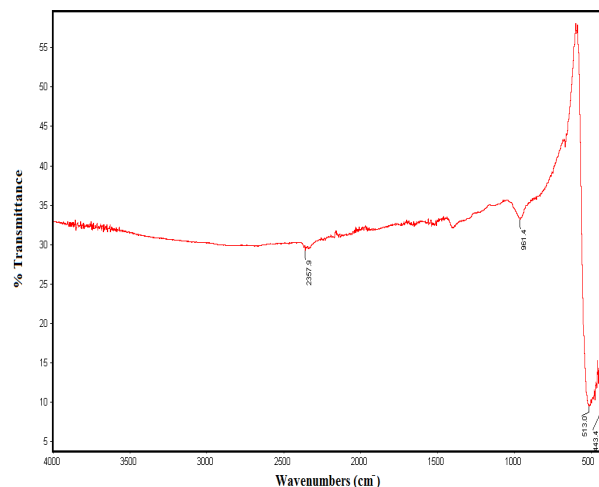


Figure 4. FT-IR spectra of Ag₂O nanoparticles.

4 CONCLUSION

In this article, we have successfully synthesized the silver oxide nanoparticles through a simple chemical route. X-ray diffraction analysis reveals that the crystallite size of the Ag₂O particles was found to be 37.90 nm. From the FT-IR Study the

stretching vibration of Ag-O group was found to be in range of 513 cm^{-1} which confirms the formation of Ag_2O particles.

REFERENCES

- [1] F. Derikvand, F. Bigi, R. Maggi, C.G. Piscopo, G. Sartori, *Journal of Catalysis* 271 (2010) 99.
- [2] W. Wang, Q. Zhao, J. Dong, J. Li, *International Journal of Hydrogen Energy* 36 (2011) 7374.
- [3] V.V. Petrov, T.N. Nazarova, A.N. Korolev, N.F. Kopilova, *Sensors and Actuators B: Chemical* 133 (2008) 291.
- [4] E. Sanli, B.Z. Uysal, M.L. Aksu, *International Journal of Hydrogen Energy* 33 (2008) 2097.
- [5] Y. Ida, S. Watase, T. Shinagawa, M. Watanabe, M. Chigane, M. Inaba, A. Tasaka, M. Izaki, *Chemistry of Materials* 20 (2008) 1254.
- [6] W.-X. Li, C. Stampfl, M. Scheffler, *Physical Review B* 68 (2003) p.165412.
- [7] Y.-H.W.a.H.-Y. Gu, *Microchimica Acta* 164 (2009) 41.
- [8] P.K. Sahoo, S.S. Kalyan Kamal, T. Jagadeesh Kumar, B. Sreedhar, A.K. Singh, S.K. Srivastava, *Def. Sci. J.* 59 (2009) 447.
- [9] M. Popa, T. Pradell, D. Crespo, J.M. Calderón-Moreno, *Colloids and Surfaces A: Physicochemical and Engineering Aspects* 303 (2007) 184.

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