

Enhanced Growth Control of Silver Nanowires Through Silver Nitrate Concentration for the Optical, Thermal, Crystal Structure, and Morphology Properties

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Abstract

Silver nanowires were successfully synthesized using the polyol method with silver nitrate concentrations of 0.3 M and 0.5 M. The synthesized silver nanowires were characterized using UV-Vis, FTIR, DTA/TGA, XRD, TEM, SEM-EDX, and SAED to investigate their optical, chemical, thermal, structural, and morphological properties. UV-Vis spectra exhibited characteristic surface plasmon resonance absorption bands at 350–390 nm, with peak positions influenced by changes in nanowire dimensions and aspect ratio as a function of silver nitrate concentration. FTIR analysis confirmed the interaction of polyvinylpyrrolidone and ethylene glycol with the silver surface, indicating effective surface stabilization during synthesis. Thermal analysis showed that silver nanowires synthesized at 0.5 M possessed higher thermal stability, as evidenced by higher decomposition temperatures and greater residual mass. XRD patterns revealed the characteristic face-centered cubic crystal structure of silver with dominant diffraction planes of (111), (200), (220), (311), and (222). TEM and SAED analyses confirmed the formation of one-dimensional polycrystalline silver nanowires, while SEM observations showed that increasing silver nitrate concentration produced nanowires with smaller diameters (185.79 ± 3 nm) and longer lengths (16.98 ± 2 μ m). EDX analysis further verified the high purity of silver in the synthesized samples. These results demonstrate that silver nitrate concentration plays a crucial role in controlling the optical, thermal, structural, and morphological characteristics of silver nanowires synthesized via the polyol method. The improved properties obtained at higher precursor concentration highlight the potential of these silver nanowires as conductive nanomaterials for transparent conductive films, flexible electronics, optoelectronic devices, and sensor applications.

Keywords

Silver Nanowires, Silver Nitrate, Nanomaterials, Characterization, Polyol

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1. INTRODUCTION

Recent advances in optoelectronics, flexible electronics, transparent conductive electrodes, and wearable sensors have significantly increased the demand for conductive materials that simultaneously exhibit high electrical conductivity, excellent optical transparency, and outstanding mechanical flexibility (Zhang et al., 2020; Kwon et al., 2018). Conventional transparent conductive materials such as indium tin oxide (ITO) exhibit excellent electrical and optical performance; however, their inherent brittleness, limited flexibility, and rising indium prices limit their use in next-generation flexible devices (Yang et al., 2020). Consequently, one-dimensional metallic nanostructures, particularly silver nanowires (AgNWs), have emerged as one of the most promising alternatives because they can form highly conductive percolation networks while maintaining high

optical transparency (Zhang et al., 2017).

Among various metallic nanomaterials, AgNWs exhibit exceptional electrical conductivity, high optical transmittance, low sheet resistance, and excellent plasmonic properties resulting from surface plasmon resonance (SPR). Their high aspect ratio enables efficient electron transport through interconnected nanowire networks while minimizing optical scattering, making them highly attractive for applications in transparent electrodes, touch screens, flexible displays, photovoltaic devices, and wearable electronics (Wu et al., 2020; Hu et al., 2010). These properties strongly depend on nanowire morphology, including diameter, length, aspect ratio, crystallinity, and structural uniformity, all of which are determined during synthesis.

The polyol process remains the most widely adopted synthesis route for AgNWs because it offers simple operation,

relatively low processing temperatures, excellent reproducibility, and compatibility with large-scale production (Dzido et al., 2023; Shi and Fang, 2022). In this method, ethylene glycol serves as both a solvent and a reducing agent, while polyvinyl-pyrrolidone (PVP) selectively adsorbs onto specific Ag crystal facets to promote anisotropic crystal growth. Meanwhile, chloride ions from iron (III) chloride (FeCl_3) regulate silver ion release via transient silver-chloride (AgCl) formation and facilitate the oxidative etching of multiply twinned particles, thereby favoring one-dimensional nanowire growth (Ha et al., 2022).

Previous studies have demonstrated that synthesis parameters, including reaction temperature, PVP molecular weight, silver nitrate (AgNO_3) feeding rate, reaction time, and precursor concentration, strongly influence AgNW morphology and performance (Choi et al., 2023; Basarir et al., 2022). Among these parameters, AgNO_3 concentration directly determines the supersaturation level of Ag atoms, thereby governing nucleation density, crystal growth kinetics, and anisotropic growth during the polyol process. Increasing precursor concentration generally accelerates Ag atom generation, but excessive concentrations may also promote secondary nucleation, particle aggregation, and nanowire thickening, resulting in reduced aspect ratio and deteriorated optical performance (Liu et al., 2017, 2019).

Although numerous investigations have examined the influence of precursor concentration on AgNWs synthesis, most studies focus primarily on morphological evolution or electrical conductivity, while comprehensive correlations among precursor concentration, crystal structure, thermal stability, optical response, and nanowire morphology remain limited. Furthermore, previous reports generally investigate a broad concentration range or optimize synthesis conditions, without systematically explaining how moderate precursor concentrations simultaneously influence nucleation behavior and anisotropic growth. Consequently, the fundamental relationship between precursor concentration and the coupled evolution of optical, thermal, crystallographic, and morphological properties remains insufficiently understood.

Another unresolved issue concerns the optimum precursor concentration required to balance nucleation and crystal growth. At relatively low AgNO_3 concentrations, insufficient supersaturation limits nucleation and slows nanowire growth, resulting in lower AgNW yield. Conversely, excessive precursor concentrations increase the reduction rate of Ag^+ ions, producing abundant nuclei that compete for available silver atoms, thereby promoting thicker nanowires, shorter lengths, and increased nanoparticle formation. Therefore, identifying an intermediate precursor concentration that maximizes anisotropic growth while suppressing isotropic particle formation remains an important challenge for scalable AgNWs production.

Based on these considerations, this work systematically investigates the influence of two moderate AgNO_3 concentrations (0.3 M and 0.5 M) on the nucleation and anisotropic growth behavior of AgNWs synthesized via the polyol method. These

concentrations were deliberately selected because they represent two different supersaturation regimes while maintaining identical synthesis conditions, allowing the isolated effect of precursor concentration on crystal growth kinetics to be evaluated. Unlike previous studies that mainly report morphological observations, this work correlates UV-Vis spectroscopy, FTIR spectroscopy, X-ray diffraction (XRD), differential thermal analysis/thermogravimetric analysis (DTA/TGA), and electron microscopy to establish a comprehensive relationship between precursor concentration, structural evolution, thermal behavior, and optical performance.

This study hypothesizes that increasing AgNO_3 concentration from 0.3 M to 0.5 M increases the availability of Ag atoms during reduction, thereby altering nucleation density and crystal growth kinetics. This change is expected to enhance anisotropic growth, increase nanowire crystallinity and thermal stability, and alter surface plasmon resonance characteristics by altering nanowire dimensions and aspect ratio. Understanding these relationships will provide new scientific insight into precursor-controlled growth mechanisms and help optimize scalable AgNWs synthesis for transparent, conductive, and flexible electronic applications.

2. EXPERIMENTAL SECTION

2.1 Materials

In this research, the materials used included silver nitrate (AgNO_3) 99% (Sigma-Aldrich Pte. Ltd., Science Park Drive, Singapore), polyvinylpyrrolidone (PVP Mw. 30,000 g/mol), ethylene glycol (EG 99.5%, Merck kGaA, Darmstadt, Germany), ethanol 96% (Merck kGaA, Darmstadt, Germany), iron (III) chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$), silicon oil, and deionized water.

2.2 Methods

Synthesis of AgNWs: Before AgNWs synthesis was carried out, first make a solution of AgNO_3 with a concentration of 0.3 M and 0.5 M in EG solvent and a solution of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ with a concentration of 0.04 M in EG solvent until dissolved at room temperature (Junaidi et al., 2020, 2021). The solution was stored in a container for the next process. Synthesis of AgNWs by the polyol method as shown in Figure 1.

During the AgNWs synthesis process, all glassware was thoroughly cleaned with deionized water and ethanol, then dried before use to minimize contamination. The synthesis of AgNWs was carried out using the polyol method under ambient conditions, without the use of an inert gas. Ethylene glycol served simultaneously as the solvent and reducing agent, while polyvinylpyrrolidone (PVP, Mw $\approx$ 30,000) acted as the capping agent. The silicone oil bath was heated to $(140 \pm 1)^\circ\text{C}$ and maintained at this temperature throughout the reaction using a digital temperature controller. The temperature stability was continuously monitored using a calibrated thermocouple immersed in the oil bath. Before precursor addition, 4.995 g of PVP was dissolved in 100 mL of EG under continuous

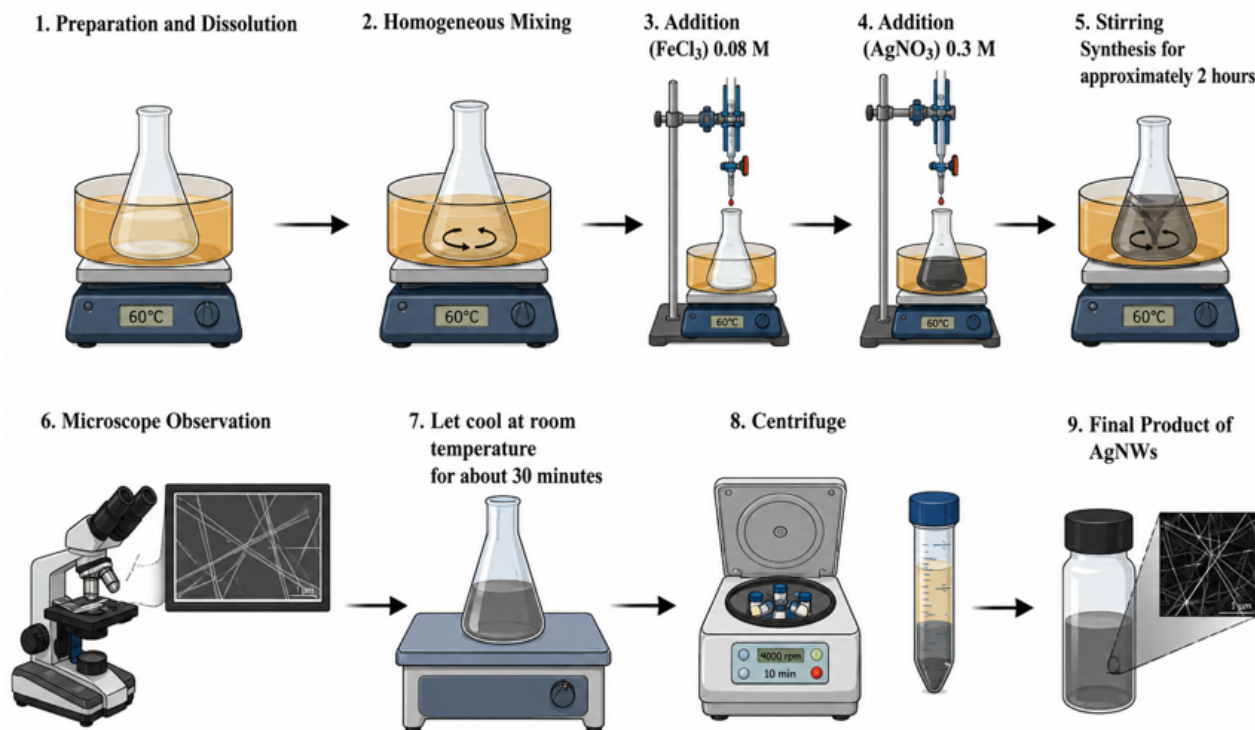


Figure 1. Synthesis of Silver Nanowires by the Polyol Method

magnetic stirring at 350 rpm for 20 min, until a clear, homogeneous solution was obtained. Subsequently, 0.25 mL of 0.04 M $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ /EG solution was injected into the PVP solution using a microsyringe over approximately 5 min while maintaining the stirring speed at 350 rpm. The $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ solution acted as a growth mediator by regulating oxidative etching and promoting anisotropic crystal growth.

After complete mixing, the AgNO_3 /EG precursor solution (0.3 M or 0.5 M) was introduced dropwise using a burette. The addition rate was carefully controlled at approximately 0.30 mL min^{-1} , corresponding to a total addition time of approximately 30 min, to ensure a gradual reduction of Ag^+ ions and to avoid excessive homogeneous nucleation. During precursor addition, the reaction temperature was maintained at $(140 \pm 1)^\circ\text{C}$, and the stirring speed was kept constant at 350 rpm to ensure homogeneous mixing and uniform heat transfer throughout the reaction mixture. After the complete addition of the AgNO_3 solution, the reaction mixture was continuously stirred at 350 rpm for an additional 2 h at 140°C until the solution gradually changed from transparent to silver-gray, indicating the formation of AgNWs (Shi and Fang, 2022; Ha et al., 2022). The reaction vessel remained open to the ambient atmosphere throughout the synthesis, and no external pressure or inert atmosphere was applied. After completion of the reaction, the AgNW colloidal suspension was naturally cooled to room temperature with approximately $(25 \pm 2)^\circ\text{C}$

before purification. Ethanol was added to the colloidal solution to precipitate the AgNWs, followed by centrifugation at 3,000 rpm for 15 min. The washing procedure was repeated three times using ethanol to remove excess EG, residual PVP, and unreacted precursors. The purified AgNWs were finally dispersed and stored in ethanol at room temperature for subsequent characterization (Liu et al., 2017).

Characterization of AgNWs Solution: To determine the optical absorption peak of the AgNWs solution, UV-Vis spectroscopy (Shimadzu, UV-1700) was used at wavelengths of 300 to 800 nm. The optical absorption spectra of the AgNWs colloids are presented as a graph of absorbance versus wavelength. The chemical structure of the AgNWs was examined using FTIR (Cary-630) to analyze the functional groups of compounds in the AgNWs colloids in the wavelength range of 500 to $4,000 \text{ cm}^{-1}$. The thermal properties of AgNWs were characterized using DTA/TGA (TGD7300) in the range of 24 to 800°C . The DTA/TGA results were used to determine the thermal stability of AgNWs over a specific temperature range until they undergo decomposition or other structural changes.

To identify the crystal structure of AgNWs, XRD characterization was performed. The XRD testing was conducted using a Shimadzu 6000 XRD with a Cu tube source ($1.5406 \text{ \AA}$) over a 20° - 90° range at a scan speed of $5^\circ/\text{min}$. The diffraction pattern data were analyzed using Match 2 software and refined

with Rietica to identify the diffraction peaks, thereby determining the sample's elemental content. The diffraction pattern was used to determine the Miller indices, lattice parameters, and the interplanar spacing. The surface structure characterization was performed using a JEOL TEM (JEM-1400) to determine the surface and crystal structures. In addition, TEM characterization will yield a diffraction ring pattern from AgNWs, which aims to determine their crystal planes. The diffraction ring pattern is then analyzed to determine the distance of each ring from the center of the circle. The diffraction data will then be compared with the XRD test results to strengthen the results obtained.

The morphology and size of the AgNWs were characterized using a SEM/EDX (JEOL JSM-6510 LA) at 10 kV. Next, the diameter and length of each AgNW were measured to determine the relationship between their length and frequency. The bar graph was then fitted using a Gaussian curve approach to determine the average diameter and length of the AgNWs.

3. RESULT AND DISCUSSION

3.1 Optical Properties of AgNWs

During the synthesis of AgNWs, upon dissolving PVP/EG and adding the $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ /EG solution, a clear solution was obtained that did not exhibit any color change. After the AgNO_3 /EG solution was injected into the PVP/EG and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ /EG solutions, the solutions slowly turned brownish. This indicates the reduction process of Ag^+ ions to Ag atoms. After ± 1 hour, the solution turned pale yellow, indicating the formation of silver nanoparticles (AgNPs). After ± 2 hours of synthesis, the solution turns gray, approaching white, indicating that AgNWs have formed. Optical absorption tests of AgNWs colloids and layers were performed using a UV-Vis spectrophotometer over the wavelength range of 300 to 800 nm. The UV-Vis spectra for the colloidal samples and the AgNWs layers are shown in Figure 2.

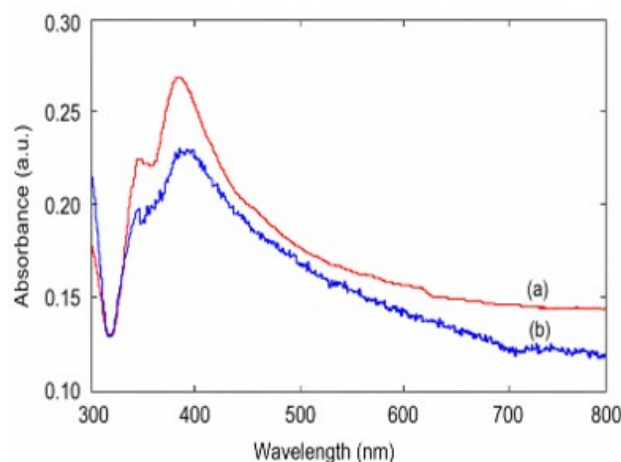


Figure 2. UV-Vis Spectra of Silver Nanowires for 0.3 M and 0.5 M

Figure 2 presents the UV-Vis absorption spectra of AgNWs colloids synthesized using AgNO_3 precursor concentrations of 0.3 M and 0.5 M. The sample synthesized at 0.3 M exhibits a weak absorption band centered at approximately 350 nm and a dominant transverse plasmon resonance peak at 387 nm, which is characteristic of silver nanowires synthesized by the polyol method (Lin et al., 2020). The weak band corresponds to the longitudinal surface plasmon resonance (LSPR). In contrast, the strong absorption at approximately 387 nm originates from the transverse surface plasmon resonance (TSPR), confirming the formation of one-dimensional Ag nanostructures (Larciprete et al., 2012; Xu et al., 2018).

Compared with the 0.3 M sample, the AgNWs synthesized at 0.5 M exhibit a slight redshift of the transverse plasmon peak, accompanied by peak broadening, indicating changes in nanowire dimensions and size distribution. The shift in the peak position (λ_{max}) reflects an increase in the average nanowire diameter, while the broader absorption band (larger FWHM) suggests a wider diameter distribution and increased structural heterogeneity (Xue et al., 2019; Naz et al., 2021). In plasmonic nanostructures, the full width at half maximum (FWHM) is directly related to electron scattering and particle size distribution. Narrow absorption bands generally indicate highly uniform nanowires, whereas broader bands imply greater variation in diameter, surface roughness, or partial aggregation (Kelly et al., 2003; Amendola and Meneghetti, 2009).

The observed plasmonic behavior can be explained by the effect of AgNO_3 concentration on nucleation and anisotropic crystal growth. At a precursor concentration of 0.3 M, the Ag^+ reduction rate is relatively slow, resulting in fewer nuclei and enabling controlled crystal growth. This condition favors the formation of AgNWs with relatively smaller diameters and higher aspect ratios, resulting in sharper SPR peaks with smaller spectral bandwidths. In contrast, increasing the precursor concentration to 0.5 M increases supersaturation of Ag atoms, accelerates nucleation, and promotes faster radial and longitudinal crystal growth (Devaraj et al., 2013). Consequently, the average nanowire diameter increases, shifting the transverse SPR peak toward longer wavelengths.

The plasmon resonance of AgNWs is strongly dependent on the aspect ratio (length/diameter). Although both length and diameter increase with increasing precursor concentration, previous studies have demonstrated that the TSPR is predominantly governed by nanowire diameter, whereas the LSPR is more sensitive to the aspect ratio (Kwon et al., 2018; Yang et al., 2020). Therefore, the observed red shift of the transverse plasmon peak indicates that the increase in diameter is more dominant than the increase in length. In addition, a broader longitudinal resonance suggests a wider aspect-ratio distribution among the synthesized nanowires.

Furthermore, increasing the AgNO_3 concentration increases the probability of nanowire overlap and partial aggregation because the amount of PVP available per Ag atom becomes relatively lower. Such interparticle plasmonic coupling modifies the local electromagnetic field around adjacent nanowires,

resulting in additional peak broadening and a slight red shift of the SPR bands (Zhang et al., 2017; Amendola and Meneghetti, 2009). These spectral changes demonstrate that UV-Vis spectroscopy provides quantitative evidence of morphological evolution during AgNW growth, consistent with the crystal growth mechanism expected at increasing precursor concentrations.

Overall, the UV-Vis results indicate that increasing the AgNO_3 concentration shifts the peak position (λ_{max}) and broadens the FWHM, reflecting changes in nanowire diameter, size distribution, and aspect ratio. These findings support the hypothesis that precursor concentration controls the nucleation and anisotropic growth kinetics of AgNWs, thereby directly influencing their plasmonic properties and potential performance in transparent conductive and optoelectronic applications.

3.2 Chemical Structure of AgNWs

FTIR analysis of AgNWs revealed functional groups in the AgNW colloids. Observations were made in the wavelength range of 4,000 to 500 cm^{-1} as shown in Figure 3.

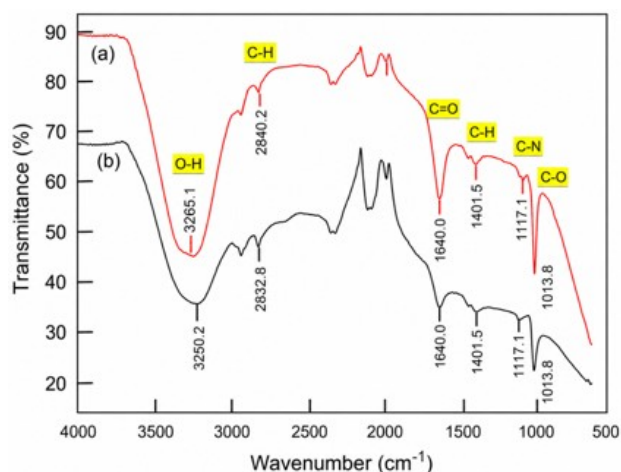


Figure 3. FTIR Spectrum of Silver Nanowires for 0.3 M and 0.5 M

Figure 3 presents the FTIR spectra of AgNWs synthesized using AgNO_3 precursor concentrations of 0.3 M and 0.5 M. Both spectra exhibit similar characteristic absorption bands corresponding to polyvinylpyrrolidone and residual ethylene glycol, confirming that these compounds remain adsorbed on the AgNWs surface after synthesis. However, slight shifts in peak position and changes in intensity are observed as the AgNO_3 concentration increases, indicating that the precursor concentration influences the chemical environment surrounding the Ag nanowires and the interaction between the capping agent and the Ag surface.

A broad absorption band appears at 3265.1 cm^{-1} for the 0.3 M sample and shifts to 3250.2 cm^{-1} for the 0.5 M sample. This band is assigned to the O-H stretching vibration of adsorbed water molecules and residual EG. The shift toward lower wavenumbers suggests stronger hydrogen bonding

or increased interaction between hydroxyl-containing species and the AgNWs surface at the higher precursor concentration (Zhang et al., 2016; Abdullah et al., 2019). The absorption bands located at 2952.1 to 2832.8 cm^{-1} correspond to the C-H stretching vibration of PVP and EG, indicating that a thin organic layer remains on the AgNWs surface after purification.

The most prominent feature of both spectra is the absorption band at approximately 1640 cm^{-1} , which is attributed to the C=O stretching vibration of the pyrrolidone ring in PVP. This band confirms that PVP is successfully adsorbed onto the AgNWs surface through coordination between the carbonyl oxygen and silver atoms, thereby suppressing lateral crystal growth and promoting anisotropic nanowire formation (Jia et al., 2021). Additional bands observed at approximately 1401 cm^{-1} , 1117 cm^{-1} , and 1014 cm^{-1} are assigned to C-H bending, C-N stretching, and C-O stretching vibrations of PVP and EG, respectively, further confirming the presence of residual stabilizing molecules after washing.

Weak absorption features are observed in the 1997 to 2355 cm^{-1} region. Because these bands are generally weak, inconsistent, and frequently associated with atmospheric CO_2 absorption or instrumental artifacts during FTIR measurements, they are not discussed further and are not considered evidence of specific chemical bonding in the AgNWs (Smith, 2011; Stuart, 2004). Therefore, the discussion focuses on the characteristic functional groups directly involved in AgNWs formation.

Overall, the slight shifts observed in the O-H and C=O absorption bands demonstrate that increasing the AgNO_3 concentration modifies the coordination environment between PVP molecules and the Ag surface. A higher precursor concentration produces more Ag atoms during reduction, increasing the available surface for PVP adsorption and slightly altering the vibrational energy of coordinated functional groups. These spectral changes are consistent with the proposed growth mechanism in which precursor concentration influences nucleation, anisotropic crystal growth, and ultimately the morphology of AgNWs, as confirmed by the corresponding SEM and XRD analyses (Bi et al., 2019; Cao et al., 2020; Kurniawidi et al., 2025).

3.3 Thermal Properties of AgNWs

The thermal analysis was carried out at a heating rate of 10 $^{\circ}\text{C}/\text{min}$ over a temperature range of 24 $^{\circ}\text{C}$ to 800 $^{\circ}\text{C}$. In Figure 4, the blue curve shows the sample's mass change (TG), the green curve shows the thermal change (DTA), and the red curve shows the mass loss rate (DTG). The analysis was conducted at temperatures up to approximately 800 $^{\circ}\text{C}$ to determine the evaporation, decomposition, and thermal stability of AgNWs. Based on the thermal stability of AgNWs, the temperature range at which the material undergoes decomposition or other structural changes is analyzed. The DTA/TGA results are shown in Figure 4.

Figure 4(a) shows that the AgNWs 0.3 M sample mass loss occurred at temperatures around 123.2 to 132.4 $^{\circ}\text{C}$, with a mass loss of approximately -44.53%. In Figure 4(b), the 0.5

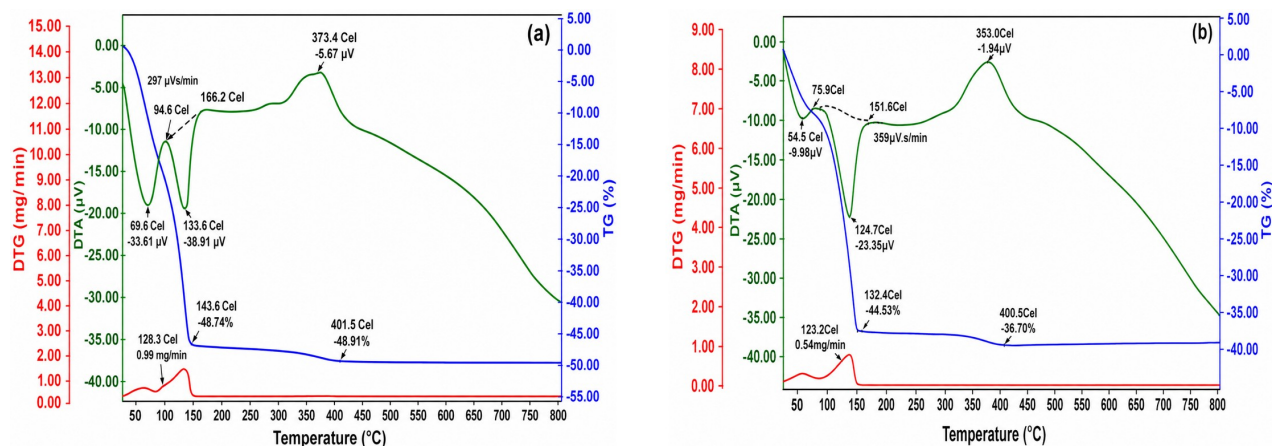


Figure 4. DTA/TGA Graph of AgNWs for Concentration of (a) 0.3 M and (b) 0.5 M

M AgNWs sample showed a significant mass loss at temperatures of 128.3 to 143.6 °C, corresponding to approximately -48.74%. The greater mass loss at the 0.5 M AgNWs concentration indicates that the sample contains more organic residues or adsorbed molecules than the 0.3 M AgNWs sample. This decrease indicates solvent evaporation and the initial decomposition of organic compounds, such as EG and PVP, that were still adsorbed on the AgNW surface (Gaynor et al., 2010).

The endothermic DTA peaks at approximately 69.6 °C and 133.6 °C for the AgNWs 0.5 M sample indicate heat absorption due to the evaporation of water and solvent, as well as the initial degradation of organic matter. DTA values of -33.61 μV and -38.91 μV indicate the intensity of the heat energy changes during these processes. In the AgNWs 0.3 M sample, the endothermic peaks occurred at 54.5 °C and 124.7 °C, with values of approximately -9.98 μV and -23.35 μV , respectively. The lower DTA value at the AgNWs 0.3 M concentration indicates that less organic material decomposed than in the AgNWs 0.5 M sample.

Furthermore, both samples exhibited exothermic peaks at high temperatures, at 353.0 °C for the AgNWs at 0.3 M concentration and 373.4 °C for the AgNWs at 0.5 M concentration. These peaks are associated with crystal structure reorganization, oxidation of organic carbon residues, or increased crystallinity of the AgNWs during heating. The shift of the exothermic peak to higher temperatures at the 0.5 M concentration indicates that the sample has better thermal stability and requires more energy to undergo thermal structural changes.

The DTG curves show the maximum mass loss rate. In the AgNWs 0.5 M sample, the maximum mass loss rate reached approximately 0.99 mg/min at 128.3 °C, while in the AgNWs 0.3 M sample, the maximum mass loss rate was approximately 0.54 mg/min at 123.2 °C. This indicates that material decomposition of 0.5 M AgNWs occurs more rapidly and more intensely due to the higher volatile and organic compound content. At around 400 °C, both samples begin to exhibit mass stability (Ha et al., 2022; Liu et al., 2017, 2019). The difference in

thermal characteristics between the two AgNW concentrations occurs because variations in the amount of Ag^+ ions during synthesis affect the morphology, size, and number of AgNWs formed. At a concentration of AgNWs 0.5 M, the number of reduced Ag atoms is greater, resulting in increased interaction between Ag and PVP (Basarir et al., 2022; Lestariana et al., 2025). Consequently, a greater amount of organic residue coats the nanowire surface, and the decomposition process requires higher energy. Furthermore, increasing the AgNO_3 concentration also increased the crystallinity and size of the AgNWs, resulting in improved thermal stability compared to the AgNWs 0.3 M sample.

Overall, the DTA/TGA results indicate that increasing the silver nitrate concentration from 0.3 M to 0.5 M improves the thermal stability of the AgNWs and increases the Ag metal content in the sample. This is indicated by the higher decomposition temperature, larger final mass residue, and greater thermal energy changes at the 0.5 M AgNWs concentration. Increasing the AgNO_3 concentration tends to improve the thermal stability of AgNWs because the silver metal content and crystallinity increase, while the proportion of organic residues decreases relatively. This is demonstrated by decreased mass loss in TGA and changes in the thermal peak characteristics in DTA (Ha et al., 2022; Liu et al., 2019). DTA/TGA analysis is crucial for assessing the thermal stability of AgNWs before their application in optoelectronic devices, sensors, or high-temperature conductive coatings.

3.4 Crystal Structure of AgNWs

The structure of AgNWs samples was evaluated using XRD and TEM to characterize the crystal structure. This XRD characterization used a Bruker AXS D8 Advance ECO with Cu radiation (1.54 Å) and a scattering angle (2θ) of 10-90°. The obtained XRD data were analyzed using QualX. The XRD spectrum patterns of AgNWs colloids with concentrations of 0.3 M and 0.5 M are shown in Figure 5.

Figure 5 shows the X-ray diffraction patterns of AgNWs samples synthesized using the polyol method with varying

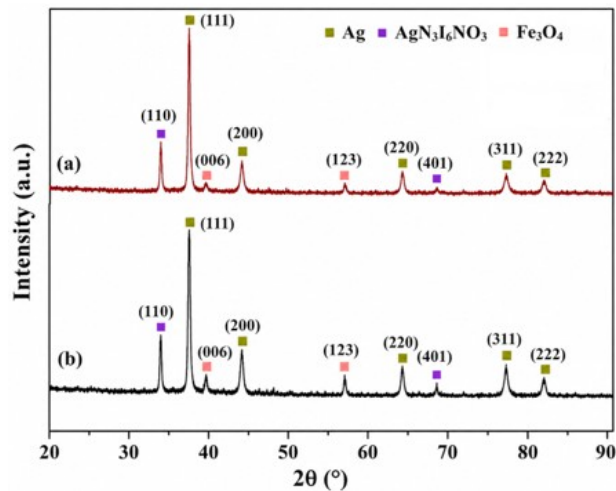


Figure 5. XRD Pattern of AgNWs for Concentration of (A) 0.3 M and (B) 0.5 M

concentrations of AgNO_3 precursor. The diffraction patterns of both samples exhibit characteristic peaks corresponding to the face-centered cubic (FCC) crystal structure of Ag. The main diffraction peaks appear at approximately 2θ angles of 38.1° , 44.3° , 64.4° , 77.4° , and 81.5° , corresponding to the (111), (200), (220), (311), and (222) crystal planes, respectively. The presence of these peaks indicates that the synthesized products are predominantly Ag metal with high crystallinity. These results are in accordance with the JCPDS standard data No. 04-0783 for crystalline silver and are consistent with previous studies on AgNWs synthesis using the polyol method (Sun et al., 2003; Zhang et al., 2017).

The most intense diffraction peak was found on the (111) plane at around 38° , indicating that AgNWs crystal growth occurs predominantly in the (111) direction. The significantly higher intensity of the (111) peak compared to other peaks indicates a preferential orientation along the nanowire axis. This phenomenon is common in AgNWs because the (111) plane has the lowest surface energy, making it more stable during anisotropic growth. The dominance of the (111) plane also indicates the successful formation of one-dimensional structures in the form of high-aspect-ratio nanowires (Coskun et al., 2011; Shi and Fang, 2022).

In addition to the Ag peak, a weak peak at approximately $2\theta = 32^\circ$ was identified, associated with the (310) plane of residual AgNO_3 , and a small peak at approximately 39° , associated with the (006) plane of Fe_2O_3 . The presence of Fe_2O_3 is due to the metal precursor used, namely $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$. The appearance of these peaks indicates the presence of residual AgNO_3 precursor and traces of iron compounds from the addition of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ during synthesis. However, the intensity of these two peaks is relatively low compared to that of the Ag peak, suggesting that the impurities formed are very small and do not affect the dominant AgNWs phase in the sample. These results indicate that the ethanol washing and centrifugation processes

successfully removed most of the remaining reactants (?).

Comparison of the XRD patterns between samples 0.3 M and 0.5 M shows that increasing the AgNO_3 concentration causes an increase in the intensity of the Ag diffraction peak, particularly on the (111) plane. This indicates an increase in the number of Ag crystals formed and an increase in the sample's crystallinity. Higher AgNO_3 concentrations result in greater reduction of Ag^+ ions to Ag atoms during the polyol process, increasing the nanowire growth rate and the number of crystals formed. Consequently, the diffraction intensity is higher, and the diffraction peaks appear sharper.

The relatively narrow diffraction peak widths in both samples indicate a relatively large crystallite size and a good degree of crystal order. In general, the narrower the FWHM, the larger the crystallite size. This indicates that the Ag^+ reduction by ethylene glycol is effective, yielding AgNWs with a well-defined crystal structure. High crystallinity is crucial for improving electrical conductivity because it reduces electron scattering at grain boundaries (Wang et al., 2009).

The successful formation of a crystalline Ag phase with a dominant (111) orientation significantly impacts the optical and electrical properties of AgNWs. A well-ordered crystal structure allows for higher electron mobility, thus enhancing electrical conductivity. Furthermore, high crystallinity also supports SPR, thereby enhancing the optical properties of AgNWs for applications in sensors, optoelectronic devices, transparent conductive films, and flexible electrodes (Kwon et al., 2018; Yang et al., 2022).

Table 1. The Ag phase refinement results of AgNWs.

Parameters	COD	Samples of AgNWs	
	Database	0.3 M	0.5 M
χ^2	-	0.773	1.522
R_{wp} (%)	-	4.81	8.16
R_p (%)	-	3.51	6.16
R_{exp} (%)	-	5.47	6.62
Lattice constant			
a (Å)	4.0790	4.08097	4.065517
b (Å)	0	0.000003	0.000008
c (Å)	0	0.000074	0.000425
Cell volume (Å ³)	0	0.00000001	0.00000014

Overall, the XRD results demonstrate that the polyol method successfully produces AgNWs with a good FCC crystal structure, a dominant (111) plane, high crystallinity, and very low impurity content. Increasing the AgNO_3 concentration has been shown to increase the diffraction peak intensity and the degree of crystallinity of the AgNWs, potentially enhancing the material's performance in various optoelectronic and flexible electronics applications. The refinement process involves fitting the calculated and measured diffraction patterns with the COD database data. The output information includes, among other things, the match index of the calculated diffraction pattern with the measured diffraction pattern, as shown in Figure

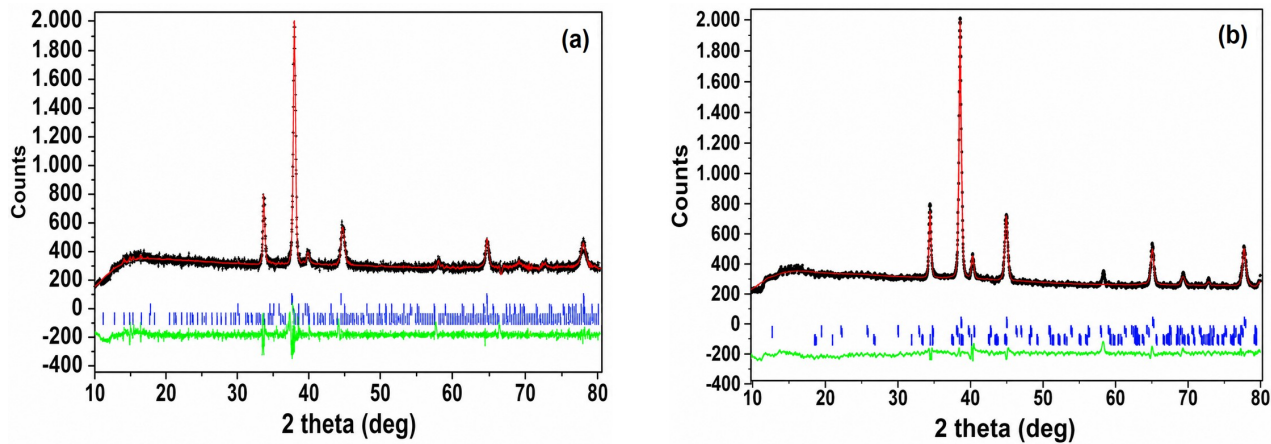


Figure 6. Refinement Results of AgNWs Colloidal Samples For (a) 0.3 M and (b) 0.5 M

Table 2. Calculation of Crystallite Size of AgNWs 0.3 M and AgNWs 0.5 M

Sample of AgNWs	K	2θ (°)	λ (nm)	FWHM (°)	FWHM (rad)	L (nm)
AgNWs 0.3 M	0.9	38.21	0.15406	0.29412	0.00513	28.58
AgNWs 0.5 M	0.9	38.50	0.15406	0.34482	0.00602	24.40

6, and results from Reitica refinement, which provide crystal structure parameter information, as shown in Table 1.

Figure 6 shows the refinement results of the AgNWs. The calculated diffraction pattern is shown by the red line, derived from the COD database data. The measured diffraction pattern, shown by the dashed black line, represents the XRD graph of the AgNWs sample, derived from the XRD data. The red line represents the graph from the Reitica database, after first entering the parameters from the COD database that correspond to the emerging phases. The green line shows the intensity difference between the XRD graph and the Reitica database, while the blue line shows the positions of the matched peaks. This will affect the match index for the refinement result. This match can be declared complete if the difference between the calculated and measured patterns shows relatively small fluctuations, as evidenced by a goodness-of-fit (GoF) value <4%.

Table 1 indicates that the sample contains a significant amount of silver. This is supported by the refinement match data such as GOF (χ^2), weighted profile residual (R_{wp}), profile residual (R_p), and expectation residual (R_{exp}). Each value of the residue (R_{wp} , R_p , and R_{exp}) must be smaller than 20. The χ^2 value indicates the GoF; the smaller the χ^2 value, the more accurate the matching process will be. The χ^2 value is obtained from the comparison of R_{wp} and R_{exp} . The R_p value will be smaller when the difference in intensity between the observed and calculated data is smaller (Olewnik-Kruszkowska et al., 2019; Zhao et al., 2022). The results of the XRD analysis can also be used to determine the crystal size of AgNWs by analyzing the highest peak in OriginLab. In the software, the

FWHM values and crystal sizes of AgNWs are shown in Table 2.

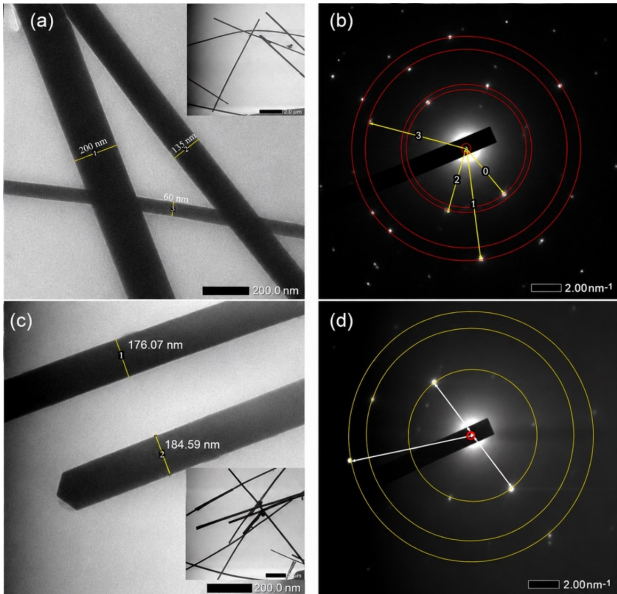


Figure 7. TEM and SAED Images of AgNWs For (a-b) 0.3 M and (c-d) 0.5 M

Table 2 shows the value of half of the maximum peak height or FWHM of the XRD and refinement result for the AgNWs 0.3 M sample, calculated from the OriginLab software, from the peak that has the highest intensity at an angle of 38.21° of 0.00513 radians with a crystal size of 28.58 nm. At the

Table 3. SAED data analysis for AgNWs 0.3 M

R (nm)	λ	L (nm)	d (nm)	a (nm)	$h^2 + k^2 + l^2$	Miller Index (hkl)
3.89	3.35	284.29	0.245	0.4142	2.850	111
4.29	3.35	284.29	0.222	0.4142	3.490	200
6.55	3.35	284.29	0.145	0.4142	8.143	220
7.47	3.35	284.29	0.128	0.4142	10.688	311

same time, AgNWs 0.5 M obtained an FWHM value at an angle of 38.50° of 0.00602 radians with a crystal size of 24.40 nm. Both samples were derived from the XRD results and calculated using the Scherrer equation. Table 2 shows that the AgNWs 0.3 M have a larger crystal size than the AgNWs 0.5 M sample. The FWHM value is used to calculate the crystal size; the smaller the FWHM, the larger the crystal. The results of the calculation indicate that AgNWs are in the nanometer range, confirming that they are nanomaterials (Aldakheel et al., 2023).

TEM characterization was performed to analyze particle diameter, yielding brighter, clearer images. This analysis was also used to strengthen the average diameter distribution obtained from SEM characterization. TEM characterization with SAED was used not only to analyze TEM images but also to analyze diffraction patterns that later identified the crystal phase. The TEM and SAED results are shown in Figure 7. The results of measurements and calculations on the diffraction rings are presented in Tables 3 and 4.

from the diffraction ring pattern analysis of the AgNWs colloid sample shown in Figure 7(b), four crystal planes were identified: (111), (200), (220), and (311), as shown in Table 3. From the AgNWs diffraction ring pattern, no diffraction ring pattern was formed for the (222) crystal plane, as obtained from the results of the X-ray diffractogram of the AgNWs colloid. The X-ray diffractogram of the AgNWs colloid shows that the intensity of the (222) plane is lower than that of the other planes, which explains why the diffraction rings for the two planes are not clearly visible.

Figure 7(c) shows the TEM results of AgNWs 0.5 M analyzed using ImageJ software, which obtained AgNWs diameters of 176.07 nm and 184.59 nm. The diffraction pattern shown in Figure 7(d) indicates that the sample is polycrystalline. Analysis of the distance between the planes was performed using ImageJ. As is well known, decahedral particles, often called multi-twin particles (MTPs), have five planes of symmetry and a surface consisting of (111) planes. These planes act as growth points for AgNWs. When decahedral AgNPs form, other reduced Ag atoms crystallize on the (111) planes due to their high surface energy. This results in the growth of decahedral AgNPs into AgNWs. At the beginning of formation, PVP interacts more strongly with the (100) planes than with the (111) planes, resulting in continued growth at both ends of the AgNWs. Because the double planes of decahedral particles do not bend during growth, AgNWs with a pentagonal cross-section, possessing five planes of symmetry at the ends and surfaces, are formed (Garnett et al., 2019).

The Miller indices in Table 4 were obtained based on the adjustment of the interplane distance results with the XRD characterization results (JCPDS File 04-0783). This diffraction pattern shows three crystal planes: (111), (220), and (311). These three crystal plane patterns are included in the FCC crystal structure. These patterns are also found in the XRD analysis (Chan et al., 2016; Bai et al., 2020).

3.5 Morphology and Size of AgNWs

SEM-EDX characterization was performed using a ZEISS Evo MA10. SEM was used to identify the length and diameter of AgNWs particles for 100 samples of AgNWs. EDX was used to identify the elemental composition of the AgNWs. The SEM results are shown in Figure 8.

The SEM image results were then analyzed for diameter and length distributions using ImageJ software. Figure 8(a-

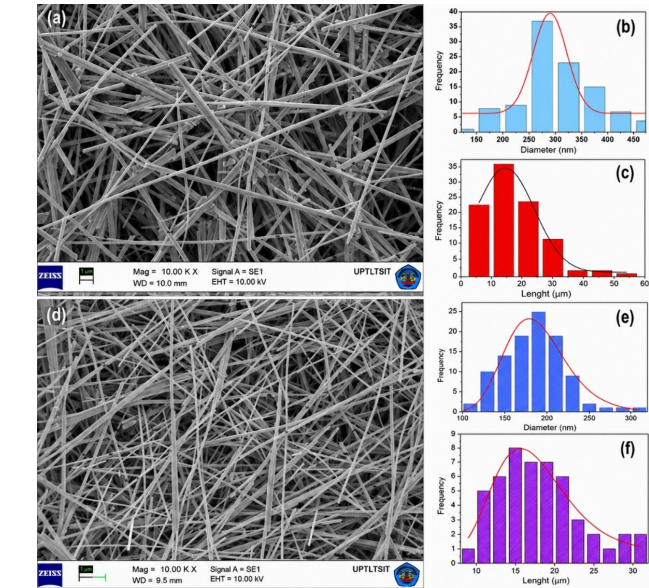


Figure 8. (a-c) SEM Images of AgNWs 0.3 M, and (D-F) SEM Images of AgNWs 0.5 M

Figure 7(a) shows the TEM results of AgNWs 0.3 M, which obtained an AgNWs diameter of 135 to 200 nm. Meanwhile,

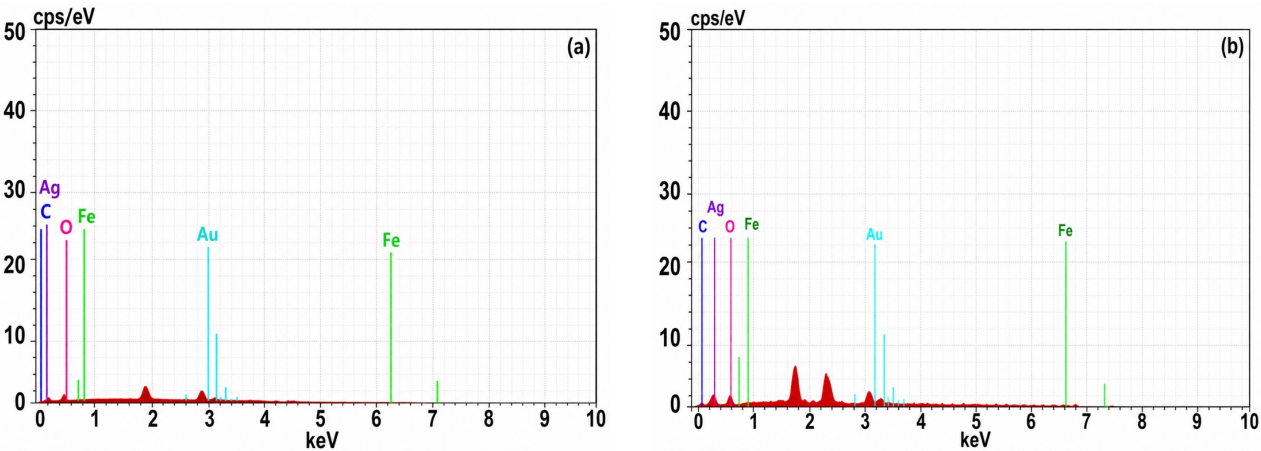


Figure 9. EDX Spectroscopic Characteristics of AgNWs (a) 0.3 M and (b) 0.5 M

Table 4. SAED Data Analysis for AgNWs 0.5 M

<i>R</i> (nm)	λ ($\times 10^{-3}$) nm	<i>L</i> (nm)	<i>d</i> (nm)	<i>a</i> (nm)	$h^2 + k^2 + l^2$	Miller Index (<i>hkl</i>)
4.29	3.35	284.29	0.234	0.4142	2.330	111
6.93	3.35	284.29	0.143	0.4142	8.582	220
8.18	3.35	284.29	0.126	0.4142	12.243	311

c) shows that this synthesis process can produce AgNWs at 0.3 M, with a very homogeneous diameter of (290 ± 7) nm and a length of around (15 ± 1) μ m. For AgNWs 0.5 M, the diameter distribution of the AgNWs colloid obtained was around (185.79 ± 3) nm, and the length distribution obtained was around (16.98 ± 1) μ m, as shown in Figure 8(d-f). These results show that the AgNWs formed are much longer than those reported in previous studies Fitzgerald et al. (2021); Junaidi et al. (2017). The elemental composition of AgNWs can be determined from the EDX results shown in Figure 9.

The elemental content using EDX of AgNO₃ powder in Figure 9(a) shows that there are several elements other than Ag and O, namely carbon (C) and iron (Fe). AgNWs 0.3 M colloid contains Ag = 96.87%; O = 1.37%; and C = 1.76%. The EDX results show that the AgNWs colloid sample still contains the Fe element originating from FeCl₃.6H₂O, which functions as a controller of the size and morphology of the AgNWs colloid. The results of the AgNWs 0.5 M spectrum in Figure 9(b) show several elements, along with their weight percentages: Ag (71.24%), Fe (3.25%), C (7.91%), and O (17.60%). Based on the total AgNWs sample, the Ag element has the highest weight percentage of the total sample weight among the other elements. This indicates the high purity of AgNWs.

In the polyol method, AgNO₃ serves as a source of Ag⁺ ions, which are reduced to Ag atoms by a solvent such as ethylene glycol. Increasing the AgNO₃ concentration increases the number of Ag⁺ ions in the solution, thus increasing the number

of reduced Ag atoms. These Ag atoms then aggregate to form crystal nuclei and subsequently grow into one-dimensional nanowire structures. At higher AgNO₃ concentrations, the crystal growth rate tends to be faster than the rate of new nucleus formation. This causes more Ag atoms to attach to the existing nuclei, increasing the length of the nanowires. Consequently, SEM results show longer AgNWs compared to samples with low AgNO₃ concentrations (Hu et al., 2010).

Furthermore, increasing the AgNO₃ concentration increases the supersaturation of Ag⁺ ions in the solution. High supersaturation accelerates the diffusion of Ag atoms toward the crystal surface, leading to a predominance of anisotropic (one-way) growth. This anisotropic growth causes the nanowires to elongate and have a larger aspect ratio. The concentration of AgNO₃ also affects the effectiveness of capping agents, such as PVP, in controlling crystal growth by coating specific crystal planes with silver. If the amount of AgNO₃ is increased while the amount of PVP remains constant, the PVP's ability to control the entire particle surface is reduced. As a result, crystal growth is less inhibited, resulting in a decrease in nanowire diameter (Zhang et al., 2008).

4. CONCLUSIONS

Silver nanowires were successfully synthesized by the polyol method through controlling the AgNO₃ precursor concentration, demonstrating that precursor concentration is a key parameter governing the nucleation and anisotropic growth

of AgNWs. The combined UV-Vis, FTIR, XRD, DTA/TGA, TEM, and SEM analyses consistently show that variations in AgNO₃ concentration influence not only the structural and optical characteristics but also the nanowire growth kinetics and surface chemistry. The higher precursor concentration promoted more efficient anisotropic crystal growth, resulting in AgNWs with improved crystallinity, higher aspect ratios, and enhanced thermal stability. These findings provide experimental evidence that the balance between Ag⁺ reduction, PVP surface coordination, and crystal growth kinetics determines the final morphology and physicochemical properties of AgNWs synthesized by the polyol method. The observed relationship between precursor concentration and nanowire evolution contributes to a better understanding of the growth mechanism of one-dimensional silver nanostructures, which remains an important issue for reproducible large-scale synthesis. From an application perspective, AgNWs synthesized at higher AgNO₃ concentrations exhibit characteristics desirable for transparent conductive networks, including high aspect ratios, good crystallinity, and improved thermal stability. These properties are expected to enhance electrical percolation while maintaining optical transparency, making the synthesized AgNWs promising candidates for transparent electrodes, flexible electronics, optoelectronic devices, sensors, and next-generation wearable technologies. Future work should investigate the electrical conductivity, optical transmittance, and device-level performance of the synthesized AgNWs to establish direct structure, property, and performance relationships.

5. ACKNOWLEDGEMENT

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