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Research Article

Synthesis and Growth Mechanism of Silver Nanowires through Different Mediated Agents ($CuCl_2$ and NaCl) Polyol Process

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Silver nanowires (AgNWs) have been synthesized by polyol process through different mediated agents ($CuCl_2$ and NaCl). The presence of cations and anions (Cu(II), Na^+, and Cl^-) has been shown to have a strong impact on the shape of silver nanostructures. The field emission scanning electron microscopy (FESEM) and transmission electron microscopy (TEM) show uniform nanowires. The UV-vis spectra show that plasmon peak indicated the formation of nanowires. The X-ray diffraction (XRD) pattern displayed that final product was highly crystallized and pure. The growth mechanism of AgNWs was proposed.

1. Introduction

One-dimensional (1-D) metal nanostructures such as nanowires have attracted extensive attention due to their unique magnetic, optical, and electronic properties compared to zero-dimensional (0-D) nanostructures [1–6]. Among these 1-D metal nanostructures, silver nanowires (AgNWs) are particularly of interest because the bulk Ag exhibits the highest electrical and thermal conductivity among all metals. There are many applications where nanowires could be exploited to greatly enhance the functionality of a material [7–9]. In these regards, the synthesis of nanowires has attracted attention from a broad range of researchers [10–14]. Over the last decade, various methods had been used to synthesize AgNWs such as polyol process [12–14], wet chemical synthesis [15, 16], hydrothermal method [17, 18], and ultraviolet irradiation photoreduction techniques [19, 20]. Among these methods, the polyol process is considered due to simple, effective, low cost, and high yield. In the polyol process, an exotic reagent leads to the formation of wire like structure. Xia and Sun [13] have modified the polyol process by generating AgNWs with diameters in the range of 30–50 nm. By controlling the parameters such as reaction time, molar ratio between capping agent and metallic precursor, temperature, and addition of control agent, a reasonable control growth of AgNWs may be achieved. In this work, AgNWs were synthesized through reducing silver nitrate ($AgNO_3$) with 1,2-propanediol (Sample 1) and ethylene glycol (EG) (Samples 2 and 3) in the presence of polyvinylpyrrolidone (PVP) as the surfactant which can direct the growth of AgNWs and protect them from aggregation. The mediated agents such as $CuCl_2$ (Sample 2) and NaCl (Sample 3) are added to facilitate the growth of AgNWs. We believe Cu(II), Na^+, and Cl^- ions are necessary for AgNWs production.

2. Experimental Method

Anhydrous 1,2-propandiol (99%), $AgNO_3$ (99%), and polyvinylpyrrolidone were purchased from Acros. All the chemicals were used as received without any further purification. The first sample (Sample 1) is without any mediated agent. 10 mL of 1,2-propanediol was added into a 50 mL three-necked flask at 170°C for 2 h. Then 0.5 mL of 1,2-propanediol solution of $AgNO_3$ (0.005 M) was injected into the 1,2-propanediol under vigorous magnetic stirring. Later, 3 mL of 1,2-propanediol solution of $AgNO_3$ (0.1 M) and 3 mL of 1,2-propanediol solution of PVP (0.45 M) were added dropwise

simultaneously over a period of 5 minutes. The solution immediately turned from colorless to light yellow. The reaction was continued for 1 h and heated at 170°C for 30 min. A grey suspension was obtained and allowed to cool at the room temperature. Then, the mixture was diluted by acetone, centrifuged, washed by deionized water, and dried in a vacuum for 24 hours at room temperature.

The second sample (Sample 2) is with $CuCl_2$ as the mediated agent. Firstly, 5 mL of ethylene glycol was added into a beaker and heated for 1 hour using silicon oil bath at 150°C. Then, 40 μL of a 4 mM $CuCl_2{\cdot}H_2O$/ethylene glycol was added into the solution, stirred, and allowed to heat for 15 minutes. After that, 1.5 mL of 114 mM PVP/ethylene glycol was added into the beaker, followed by 1.5 mL of 94 mM $AgNO_3$/ethylene glycol. The color changed to yellow and became brownish grey after $AgNO_3$/ethylene glycol was added. The solution was heated for another 1 hour. Then, the solution was taken out and let to cool at room temperature. The solution was centrifuged at 3000 rpm and 30 min each with acetone and deionized water. The final product was preserved in deionized water until characterization.

The third sample (Sample 3) is with NaCl as the mediated agent. 15 mL of 0.36 M of ethylene glycol (EG) solution of polyvinylpyrrolidone (PVP) was heated and stirred for 15 minutes. It was then followed by microwave heating at 170°C for an hour. Then, 20 μL of 0.1 M EG solution of NaCl and 20 μL of 0.06 M EG solution of $AgNO_3$ were injected into this EG solution of PVP to produce AgCl as seeds. After 15 minutes of injection, 15 mL of 0.06 M of EG solution of $AgNO_3$ was injected into mixture solution within 5 minutes. The mixture solution was stirred to obtain a homogeneous solution. The color of mixture solution slowly changes to light yellow. The mixture solution was then heated under microwave irradiation with temperature maintained at 170°C for approximately 30 minutes. It is allowed to cool naturally to room temperature and turn to gray color. The resulting solution was washed several times using acetone and ethanol and then dried in vacuum at 60°C.

The X-ray diffraction (XRD) patterns were recorded using a Siemens D5000 X-ray diffractometer (Cu-Kα radiation, $\lambda = 0.154$ nm). The transmission electron microscopy (TEM) images were taken on a Libra 120 model TEM using accelerating voltage of 400 kV. Field emission scanning electron microscopy (FE-SEM) images were taken using Zeiss Auriga. The UV-visible spectrum of the as-prepared products was recorded on a Varian Cary 50 UV-vis spectroscopy.

3. Results and Discussion

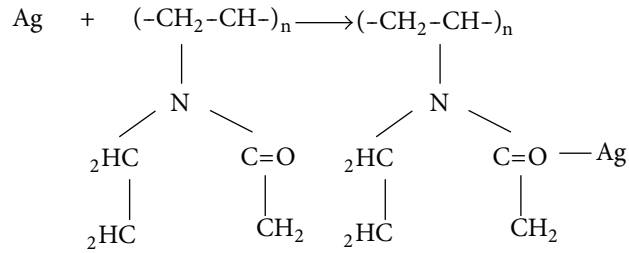
3.1. Mechanism for the Formation of AgNWs

3.1.1. Scheme 1 (*Sample 1 without any Mediated Agent*). The formation of anisotropic AgNWs involves two steps. In the first step, 1,2-propanediol was converted to propionaldehyde at high temperature (170°C) as shown in (1). Then it reduces Ag^+ ions to Ag atoms as shown in (2). Consider

$$HOCH_3CH_2CHOH \xrightarrow{170^\circ C} CH_3CH_2CHO + H_2O \quad (1)$$

$$2Ag^+ + 2CH_3CH_2CHO \longrightarrow CH_3CH_2COOH_3 + 2Ag + 4H^+ \quad (2)$$

In the second step, $AgNO_3$ and PVP were added dropwise to the reaction system allowing the nucleation and growth of AgNWs.



(3)

Ag atoms are nucleated through the homogeneous nucleation process. These AgNps were well dispersed because of the presence of a polymeric surfactant PVP that could be chemically adsorbed onto the surfaces of Ag through O–Ag bonding (3). PVP has an affinity toward many chemicals to form coordinative compounds due to the structure of polyvinyl skeleton with strong polar group (pyrrolidone ring). In this case, C=O polar groups were interacted with Ag^+ ions and form coordinating complex as shown in (3). When this dispersion of AgNps was continuously heated at 170°C, the small nanoparticles progressively disappeared to the benefit of larger ones via Ostwald ripening process [21]. The critical particle radius increased with temperature. As the reaction continued, the small AgNps were no longer stable in solution, and they started to dissolve and contribute to the growth of larger ones. With the assistance of PVP, some of the larger nanoparticles were able to grow into rod-shaped structures. The growth process would continue until all the AgNps were completely consumed and only nanowires survived.

3.1.2. Scheme 2 (*Sample 2 with $CuCl_2$ Mediated Agent*). In the initial step, high temperature is crucial for the conversion of ethylene glycol to glycolaldehyde as shown in

$$2HOCH_2CH_2OH + O_2 \xrightarrow{150^\circ C} 2CH_3CHO + 2H_2O \quad (4)$$

AgNps were formed by reducing Ag^+ ions with glycolaldehyde as shown in

$$2CH_3CHO + 2Ag^+ \longrightarrow CH_3CO{-}OCCH_3 + Ag + 2H^+ \quad (5)$$

It was found that a small amount of Cl^- must be added to a polyol synthesis to provide electrostatic stabilization for the initially formed Ag seeds [4]:

$$2HOCH_2CHO + CuCl_2 \longrightarrow 2CH_2COOH + Cu + 2HCl \quad (6)$$

$$Ag^+ + Cl^- \Longleftrightarrow AgCl \quad (7)$$

In addition to electrostatically stabilizing the initially formed Ag seeds, the high Cl^- concentrations during $CuCl_2$

mediated synthesis help reduce the concentration of free Ag^+ ions in the solution through the formation of AgCl. Subsequent, it will slowly release the Ag^+ and subsequent slow release of Ag^+ effectively. These facilitate the high-yield formation of the thermodynamically more stable multiply twinned Ag seeds that are required for wire length. Valency metal ions (Cu^{2+}) were reduced by EG to low valence (Cu^+) which in turn reacted with and scavenged adsorbed atomic oxygen from the surface of AgNps. Here, Cu^{2+} can remove oxygen from the solvent which prevents twinned seeds dissolved by oxidative etching and scavenging adsorbed atomic oxygen from the surface of the Ag seeds, facilitating multiply twinned seeds growth [12].

In the final step, $AgNO_3$ and PVP were added dropwise to the reaction system allowing the nucleation and growth of silver nanowires as shown in (3). AgNps were well dispersed because of the presence of PVP, a polymeric surfactant that could be chemically adsorbed onto the surfaces of Ag through O–Ag bonding. As the reaction continued, the small Ag nanoparticles were no longer stable in solution and they started to dissolve and contribute to the growth of larger ones. When multiple twinned form the nanoparticles during a nuclei period, the PVP was bounded preferently on {100} then {111} [22, 23]. This inhibited the growth along {111} direction. So the growth took place only in the {110} direction resulting in the fivefold twinned nanowires (Figure 1).

3.1.3. Scheme 3 (*Sample 3 with NaCl Mediated Agent*). Like in Scheme 2, the formation of anisotropic AgNWs involves a number of steps. The first two steps are similar to (4) and (5). Like in Scheme 2, chloride ions were added (8) to stabilize AgNps and prevented the growth of nanoparticles [10]. As a result, nanoparticles that can grow will dissolve via Oswald ripening:

$$2HOCH_2CHO + 2NaCl \longrightarrow 2CH_2COOH + 2Na^+ + 2HCl^- \quad (8)$$

$$Ag^+ + Cl^- \Longleftrightarrow AgCl \quad (9)$$

The high Cl^- concentration during NaCl mediated synthesis helps reduce the concentration of free Ag^+ in the solution through the formation of AgCl (9). Subsequent, it will slowly release the Ag^+ and subsequent slow release of Ag^+, effectively. This facilitates the high yield formation of the thermodynamically stable multiply twinned Ag seeds. So, AgCl precipitate that forms in the early stages of the reaction serves as a seed for multitwin particles. The formed AgCl nanoparticles can be reduced slowly and decreased reaction rate makes anisotropic growth of Ag nanowires favorable [24]. Meanwhile, Na^+ can remove oxygen from the solvent which prevents twinned seeds dissolved by oxidative etching and scavenging adsorbed atomic oxygen from the surface of the silver seeds, facilitating multiply twinned seeds growth [12]. In the final step, $AgNO_3$ and PVP were added dropwise to the recreation system allowing the nucleation and growth of AgNWs as shown in (3).

With passivation of some facets of particles by PVP, some nanoparticles can grow into multitwin particles. PVP is believed to passivate (100) faces of these multitwin particles and leave (111) planes achieve for anisotropic growth at [110] direction [13] (Figure 1). As the addition of Ag^+ continues, multitwin particles grow into Ag nanowires.

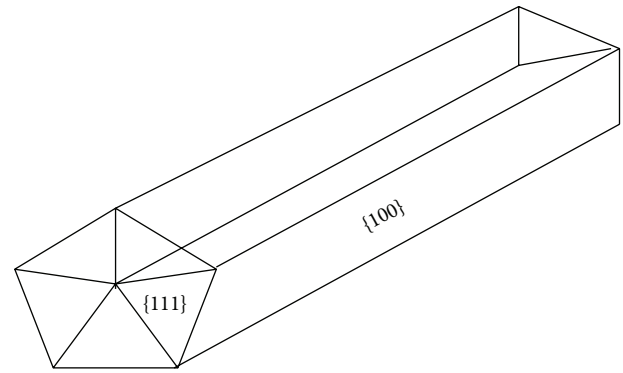


Figure 1: Schematic of 5-fold twinned pentagonal nanowires consisting of five elongated {100} facets and 10 {111} end facets.

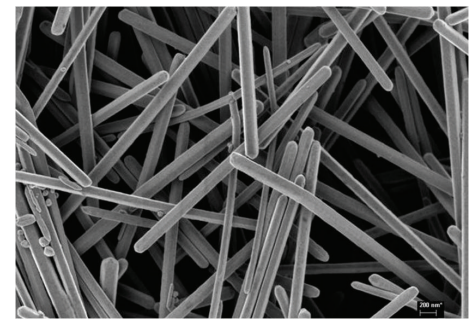


Figure 2: FESEM image of AgNWs (without any mediated agent).

3.2. FESEM Analysis. Figure 2 shows the FESEM image of AgNWs without any mediated agents. The image shows straightness along the longitudinal axis, the level of perfection, and the copiousness in quantity that we could routinely achieve using this synthetic approach. The presence of silver nanoparticles (AgNps) is also evident in the figure indicating that not all of AgNps are transformed into nanowires.

Figure 3 shows the FESEM image of AgNWs with $CuCl_2$ as the mediated agent. The image reveals that the product is entirely composed of a large quantity of uniform nanowires with a mean diameter of 65 nm. The high faces of PVP on all faces of the seeds lead to anisotropic growth mode.

Figure 4 shows the FESEM image of AgNWs with NaCl as the mediated agent. The image shows well-defined wires with mean diameter of 80 nm.

3.3. TEM Analysis. Figure 5 shows the TEM image of individual AgNWs without any mediated agents with 84 nm in diameter and 1119 nm in length.

Figure 6 shows a TEM image of an individual AgNW ($CuCl_2$ mediated agent) with 87 nm in diameter and 3 μm

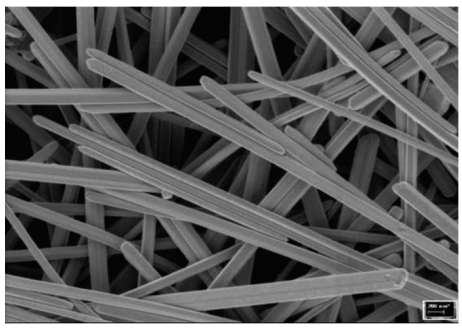


Figure 3: FESEM image of AgNWs (with $CuCl_2$ mediated agent).

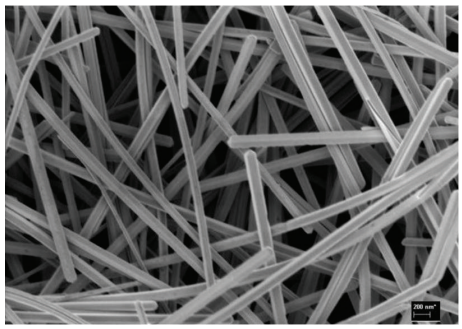


Figure 4: FESEM image of AgNWs (with NaCl mediated agent).

in length. The insert of Figure 6 reveals a pentagonal cross-section of AgNWs and indicates the multiple-twinned structure of the AgNWs [24].

Figure 7 shows the TEM image of AgNWs with NaCl mediated agent. The TEM image displayed a twin boundary in the middle of nanowires and pentagon shape cross-section at the end of nanowires.

3.4. XRD Analysis. Figure 8 shows the XRD pattern of AgNWs with and without mediated agent. The peaks at angles of $2\theta = 38.1^{\circ}$, 44.2°, 64.3°, and 77.5° correspond to the (111), (200), (220), and (311) crystal planes of the face center cubic (FCC) Ag, respectively. The lattice constant calculated from the diffraction pattern was 0.4086 nm, which is in agreement with the reported value of silver (JCPDS 04-0783). Furthermore, the intensity ratio of the (111) to (200) peaks is 2.0, in good agreement with the theoretical ratio, that is, 2.5 [14].

Figure 9 shows the XRD pattern of highly crystalline AgNWs with $CuCl_2$ mediated agent. It has a similar pattern with AgNWs without mediated agent (Figure 8). The XRD pattern reveals that the synthesis AgNWs through polyol process comprise pure phase. The lattice constant, a, was 4.07724 Å which is almost approaching the literature value of 4.086 Å. The ratio of intensity between (111) and (200) peaks reveals a relatively high value of 3.2 compared to the theoretical ratio value of 2.5. This high value of ratio indicates the enchancement of {111} crystalline planes in the AgNWs.

Figure 10 shows the XRD pattern of AgNWs with NaCl mediated agent. It exhibits well-defined peaks without any impurity element peaks detected. This indicated the success

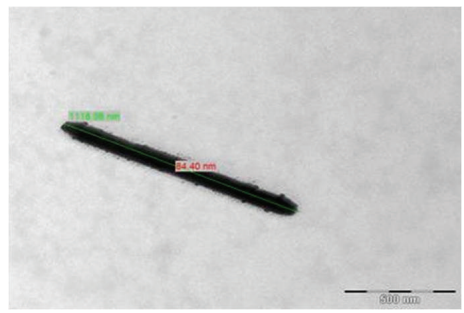


Figure 5: TEM image of AgNWs without any mediated agent.

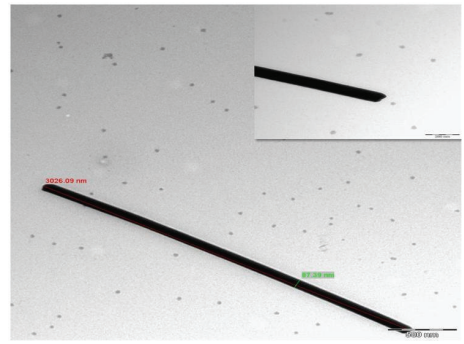


Figure 6: TEM image of AgNWs with $CuCl_2$ mediated agent (insert: pentagon cross-section of AgNWs).

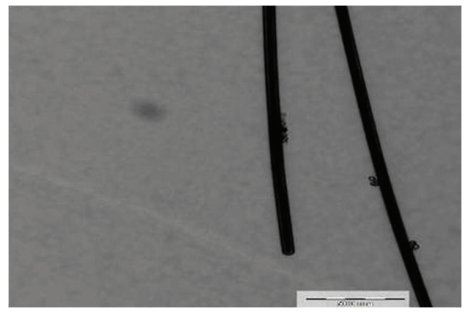


Figure 7: TEM image of AgNWs (with NaCl mediated agent).

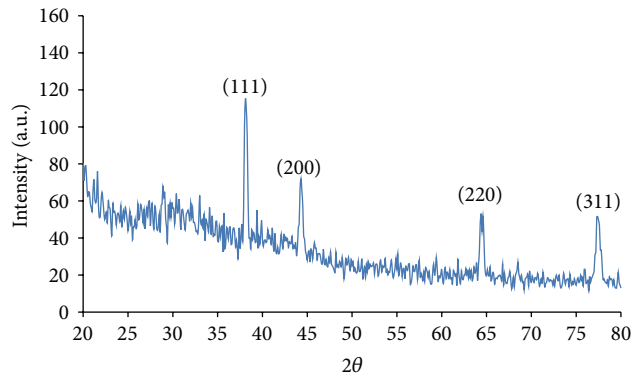


Figure 8: XRD pattern of AgNWs without any mediated agent.

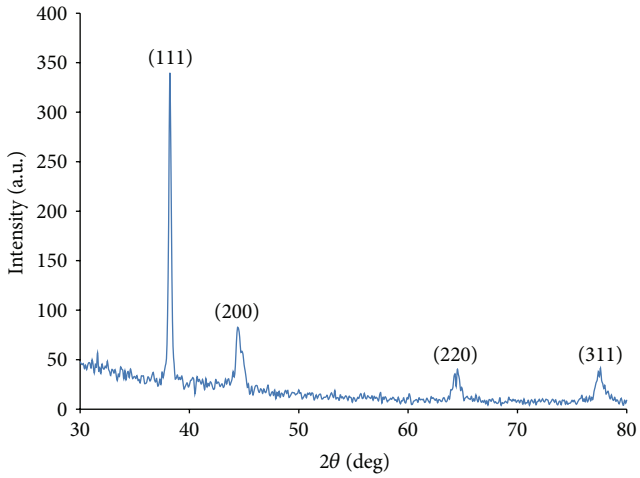


Figure 9: XRD pattern of AgNWs (with $CuCl_2$ mediated agent).

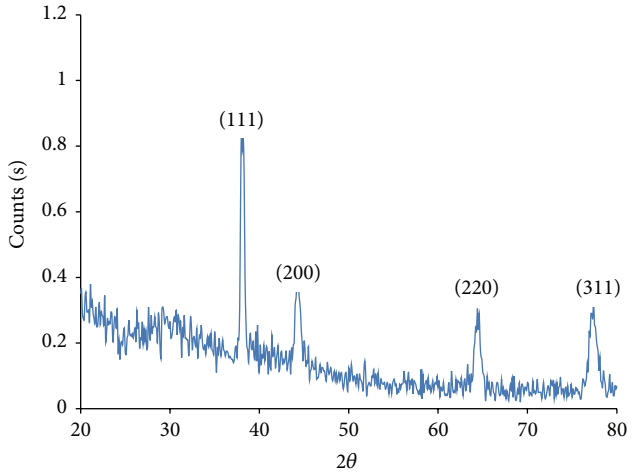


Figure 10: XRD patterns of the as-synthesized AgNWs (with NaCl mediated agent).

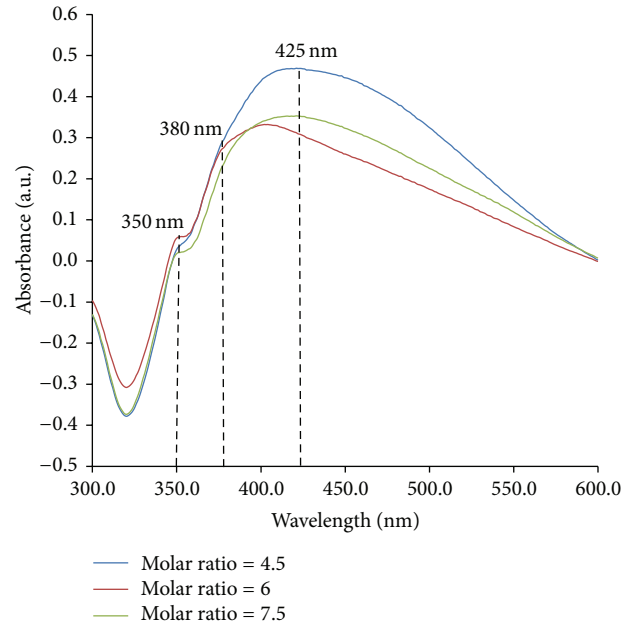


Figure 11: Absorption spectra of AgNWs (without any mediated agent) with different molar ratio of PVP and $AgNO_3$.

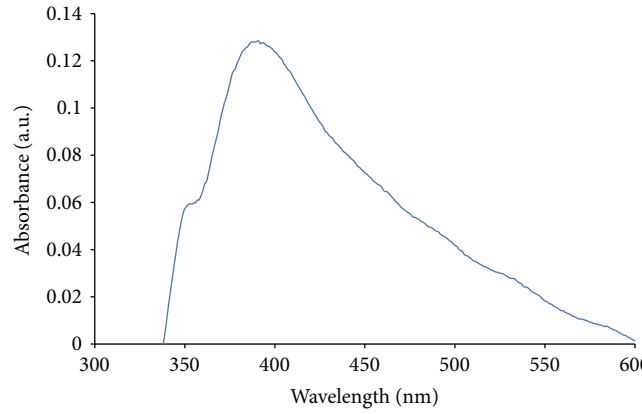


Figure 12: Absorption spectra of AgNWs (with $CuCl_2$ mediated agent).

in the formation of the crystalline silver nanowires. The four diffraction peaks obtained are similar to peaks in Figures 8 and 9. It is worth noting that the ratio of intensity between (111) and (200) peaks exhibits a relative value of 2.05 (the theoretical ratio is 2.5). It indicated that the sample becomes less crystalline with NaCl mediated agent.

3.5. UV-Vis Spectroscopy Analysis. Figure 11 shows the absorption spectra of AgNWs without mediated agent at different molar ratio of PVP to $AgNO_3$. The appearance of a weak surface plasmon resonance (SPR) at 425 nm indicated the formation of AgNps [17]. This implies that the final product synthesized under this particular condition was a mixture of AgNps and AgNWs. As the molar ratio increases from 4.5 to 7.5, the intensity of the SPR is slightly decreased. Furthermore, the SPR peak around 425 nm is blue-shifted to 404 nm. The shoulder peak at 380 nm could be considered as the optical signature of AgNWs. At this point, optical signatures similar to those of bulk Ag also began to appear as indicated by the shoulder peak around 350 nm. With increasing molar ratio, the intensity of absorption bands at 350 and 380 nm increased apparently due to increased density of AgNWs.

Figure 12 shows the UV-vis absorption spectra for AgNWs with $CuCl_2$ mediated agent. The peak positions at 391 nm could be considered as the optical signature of relatively long AgNWs. At this point, optical signatures similar to those of bulk silver also began to appear as indicated by the shoulder peak around 357 nm [25]. Compared to the previous absorption spectra (Figure 11), no peak for AgNps existed. This indicates that the sample is completely of AgNWs.

Figure 13 shows the absorption spectra of ANWs (with NaCl mediated agent) synthesized with different molar of $AgNO_3$. The appearance of a story peak at 384 nm could be considered as the transverse mode of relatively long AgNWs.

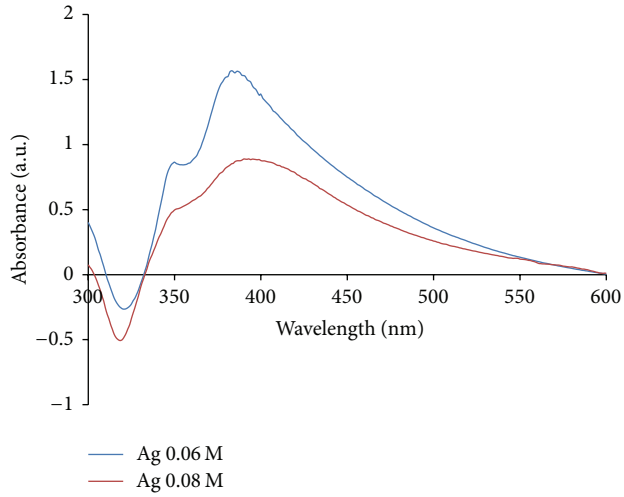


Figure 13: Absorption spectra of the as-synthesized AgNWs (with NaCl mediated agent) at different molar of $AgNO_3$: (a) 0.06; (b) 0.08.

At this point, optical signatures similar to those of bulk Ag also began to appear as indicated by the shoulder peak around 350 nm. As the concentration of $AgNO_3$ increases from 0.06 to 0.08 M, the intensity of these peaks increased significantly and red-shifted to 394 and 351 nm, respectively. This result indicated that the AgNWs increased in number with apparent growth in length. Again, the spectra show that the sample is completely AgNWs.

4. Conclusion

AgNWs were successfully synthesized by using polyol technique with and without mediated agents. It was found that the addition of $CuCl_2$ or NaCl to the polyol reduction of $AgNO_3$ in the presence of PVP greatly facilitated the formation of AgNWs. Without the mediated agents, the final product synthesized was a mixture of AgNps and AgNWs. Both the cation and the anions are crucial for the successful production of AgNWs.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgment

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