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Poliol Sentezinde Metal Tuzlarının Gümüş Nanotel Morfolojisi Üzerindeki Etkisi

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ÖZET

Poliol yöntemi, yüksek kaliteli gümüş nanotellerin (AgNWs) sentezi için basit, ekonomik ve çok yönlü bir yaklaşım sunmasıyla dikkat çekmektedir. Bu yöntemde, kullanılan metal tuzları, elde edilen AgNWs ürününün özelliklerini belirlemede önemli bir rol oynar. Bu araştırma, poliol yöntemiyle sentezlenen AgNWs uzunluğu ve çapı üzerinde farklı metal tuzlarının etkisini incelenmiştir. Her sentezde Sodyum Klorür (NaCl) sabit bir bileşen olarak kullanılmış ve buna ek olarak iki farklı tuz türü eklenmiştir. NaCl ile birlikte eser miktarda Demir (III) Nitrat ($Fe(NO_3)_3$), Bakır(II) Klorür ($CuCl_2$) ve Potasyum Bromür (KBr) kullanılmıştır. Nanotellerin şekli ve boyut dağılımı Alan Emisyonlu Taramalı Elektron Mikroskobu ile analiz edilmiş, nanopartiküllerin kristal yapısı ise X-ışını Kırınımı ile incelenmiştir. KBr tuzu kullanılarak yapılan sentezlerde en yüksek boy/en oranına sahip AgNWs üretildiği görülmüş ve bu nanotellerin uzunluğu 6,2 ± 2,5 µm olarak ölçülmüştür. Ayrıca, $CuCl_2$ tuzu kullanılarak yapılan sentezde, nanotellerin yanı sıra nanoküpler ve nanoüçgenler gibi diğer gümüş nanoyapılarının da önemli ölçüde oluştuğu gözlemlenmiştir.

Influence of Metal Salts on Silver Nanowire Morphology in Polyol Synthesis

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ABSTRACT

The polyol method has gained significant attention for synthesizing Silver nanowires (AgNWs), offering a straightforward, cost-effective, and versatile approach to producing high-quality nanowires. In polyol method, the choice of metal salts performs a crucial function in determining the properties of the final AgNW product. This research specifically explores the influence of different metal salts on the length and diameter of AgNWs synthesized through the polyol method. Each synthesis involved the use of two distinct types of salts, with NaCl being a constant component. Trace amounts of Iron(III) Nitrate ($Fe(NO_3)_3$), Copper(II) Chloride ($CuCl_2$), and Potassium Bromide (KBr) were introduced in conjunction with NaCl. The morphology and dimensional distribution of the nanowires were analyzed using Field Emission Scanning Electron Microscopy, while X-ray Diffraction was applied to study the crystal structure of the nanoparticles. Notably, the utilization of KBr in the synthesis led to the production of AgNWs with the highest aspect ratio, resulting in nanowires measuring 6.2 ± 2.5 µm in length. Additionally, the synthesis assisted by $CuCl_2$ revealed a substantial presence of other silver nanostructures, such as nanocubes and nanotriangles, alongside nanowires.

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INTRODUCTION

Silver nanowires (AgNWs) have emerged as promising nanomaterials for use in various applications owing to their unparalleled properties such as high aspect ratio, high conductivity, and outstanding catalytic activity. These properties allow them to be effectively used in flexible electronics, transparent conductive films, sensors, and energy storage devices, among others [1,2]. Various methods have been employed for synthesizing AgNWs, including chemical reduction [3], electrochemical synthesis [4], and template-assisted methods [5]. However, the polyol method has gained increasing attention as a simple, low-cost, and versatile method for producing uniform and high-quality AgNWs.

In the polyol method, silver precursors are reduced by polyols under high temperature conditions with the incorporation of stabilizing agents. The resulting AgNWs exhibit high aspect ratios, uniform diameters, and narrow size distributions. A polyol is an organic compound containing multiple hydroxyl groups. The function of polyols is as reducing and dissolving agents. The most preferred polyol is ethylene glycol ($C_2H_6O_2$) due to its low cost [6]. Polymers are used as stabilizing agents in polyol method and Polyvinylpyrrolidone (PVP) being the most effective among the polymers used. PVP is employed to ensure the growth of nanowires in one dimension and to direct the growth kinetics of metal surfaces by limiting the growth of multiple twinned particles in the {100} planes while facilitating longitudinal growth in the {111} plane, thereby aiding in the formation of AgNWs. This is due to PVP's strong interaction with the (100) surface and weak interaction with the (111) surface. As a result, the one-dimensional wire structure is shaped by faster growth on the (111) surface [7,8].

Transition metal salts perform a crucial function in the polyol synthesis of silver nanowires by serving as oxygen scavengers, thereby enabling the synthesis process to be conducted under ambient air conditions. During the synthesis of AgNWs in ambient atmosphere, the existence of atomic oxygen can hinder the growth of AgNWs by covering the surface of silver seed nanoparticles. However, transition metal ions can remove the adsorbed atomic oxygen from the silver grains. As a result, in the polyol synthesis of AgNWs conducted in an open atmosphere, the presence of transition metal salts facilitates the unhindered growth of AgNWs by removing the inhibitory effects of atomic oxygen [9]. Also, many researchers have reported that the introduction of halide salts during the synthesis process is responsible for incorporating additional anions into the system. This, in turn, leads to a decrease in the proportions of available Ag^+ ions within the solution, thereby retarding the reduction of silver nitrate. The deliberate reduction in the rate of $AgNO_3$ reduction facilitated by the presence of halide salts enables the successful and efficient growth of nanowires [10–12]. Consequently, many researchers have determined that the successful production of AgNWs requires the presence of both cations and anions. Sarisozen et al. [9] studied different kinds of metal salts including $CuCl_2$, $CoCl_2$, $MnCl_2$, $CrCl_3$, $FeCl_3$, and $ZnCl_2$ serving as support in the polyol synthesis of AgNWs in their experimental conditions and exhibited that AgNWs synthesized in the existence of $CuCl_2$ come up with the longest length. Yamamoto et al. [10] compared the effect of halide salts of NaCl, $CuCl_2$ and NaBr, the longest AgNWs were obtained with NaCl. In another study, Coskun et al. [11] emphasized that below to appropriate NaCl ratio silver microparticles began to form, and above AgCl formation initiated. Ashkarran et al. [13] investigated the impact $FeCl_3$ over the structure of AgNWs and revealed that elevated concentrations result in formation of semi spherical shaped silver nanostructures. Also, a similar result regarding excessive use of $FeCl_3$ was also emphasized by Zhang et al. [14]. Basarir et al. [15] examined the effect of NaCl, $FeCl_3$ and $CuCl_2$ salts and suggested that standard reduction potentials of the ions involved in reaction are effective in AgNW production. Zhang et al. [16] examined how the presence of NaCl, $FeCl_3$, and KBr, affects the size of AgNWs. They reported that as the ratio of KBr increased, there was an observed increase in the formation rate of particles other than nanowires.

This study investigates the impact of different metal salt types on the dimensions of silver nanowires (AgNWs) synthesized using the polyol method. Specifically, two types of salts were utilized in each synthesis: sodium chloride (NaCl), which was kept at a constant amount, and trace amounts of Copper (II) Chloride ($CuCl_2$), Potassium Bromide (KBr), or Iron(III) Nitrate ($Fe(NO_3)_3$). The innovative aspect of this study lies in systematically exploring the combined effects of these salts on AgNW synthesis, which has not been thoroughly investigated in prior research. By elucidating the role of these salts in tailoring AgNW dimensions, this work aims to advance the understanding of synthesis parameters and contribute to the optimization of AgNW fabrication processes for various applications.

MATERIALS AND METHODS

AgNW Synthesis

AgNWs were created through a facile polyol route. The summarized synthesis setup is shown in Figure 1. A mixture of 10 mL Ethylene Glycol (EG Sigma-Aldrich, ≥99.8% purity), 750 mg poly vinylpyrrolidone (PVP, Sigma-Aldrich, Mw ≈ 40,000), 0.007 mg (12 µM) NaCl (Merck, ≥99.5% purity), and 12×10^{-8} mol (12 µM) of chosen salt is heated in a flask connected to a reflux column up to 170 °C. In order to examine the effects of salt types on nanowire formation, 3 different solutions were studied using $Fe(NO_3)_3$ (Merck, ≥98% purity), $CuCl_2$ (Sigma-Aldrich, ≥97% purity) and KBr (Merck, ≥99% purity) salts. Once the temperature has stabilized, Ag source solution prepared with 5 mL of EG and 100 mg $AgNO_3$ (Sigma-Aldrich, ≥99.9% purity) was injected into the 2-necked flask via programmed syringe pump with a drop rate of 5 mL/h. Stirring continued for another 30 minutes, then the solution was allowed to reach room temperature. To separate AgNW from PVP and EG, the solution was diluted with acetone (Merck, ≥99.5% purity) (1:10 ratio) and centrifuged twice at 4500 rpm for 15 minutes. Finally, the AgNW's were dispersed in ethanol (Sigma-Aldrich, ≥95% purity) for characterization and centrifuged again at 4500 rpm for another 15 minutes. The AgNWs produced depending on salt type were presented in Table 1.

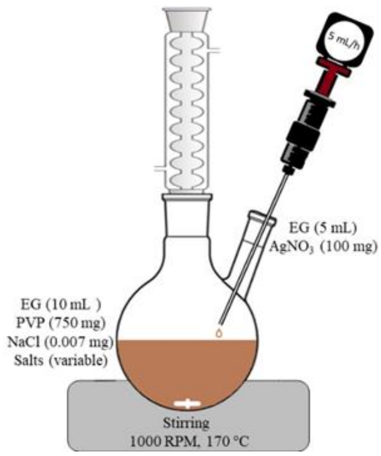

Figure 1
The summarized synthesis setup.

Characterization

AgNWs were analyzed by an X-ray diffractometer (PANalytical Empyrean, 1D mode, fixed divergence slit) between 35-80° at a scanning rate of 0.1°/min using CuKα radiation with a wavelength of 1.54056 Å. The Joint Committee on Powder Diffraction Standards (JCPDS) database was used to determine the phases. The morphology and size of AgNWs were examined using Field Emission Scanning Electron Microscopy (ZEISS GeminiSEM 500, FE-SEM) using 3 kV operation voltage.

RESULTS AND DISCUSSION

The incorporation of different salts in the synthesis of AgNWs by polyol synthesis has led to meaningful differences in the size of AgNWs and the aspect ratio. Fig.2 represents the SEM images of AgNWs synthesized. The length and diameter of the AgNWs were determined using ImageJ software by measuring a minimum of 50 nanowires and calculating the average value. The results obtained are summarized in Table 1.

Table 1
Average diameter, length, and aspect ratio of agnws obtained with different metal salts.

Salt Type	Lenght (µm)	Diameter (nm)	Aspect Ratio
KBr	6.2 ± 2.5	46 ±10	134
$CuCl_2$	4.6 ± 1.1	120 ±29	38
$Fe(NO_3)_3$	1.9 ±1.6	51 ±10	37

It is seen that the highest length/width ratio is obtained with the solution containing KBr salts. As is known, Br^- ions serve both as a capping agent to passivate the (100) surfaces in a growing Ag crystal and help reduce the reduction kinetics by binding to Ag^+ ions. Thanks to this synergistic effect, it is thought that the longest nanowires are obtained with the solution containing Br^- ions [17] . Similarly, in studies in literature where the common use of different salts was investigated, it was observed that the solutions with the largest aspect ratio were obtained with NaCl-KBr salts [16,18]. Table 2 shows the length and width of AgNWs obtained through salt-mediated polyol synthesis in various studies [13,16,19–22].

Table 2
Types of salts and dimensions of AgNWs.

Salt Types	Ag/Metal Salt*	Lenght (µm)	Diameter (nm)	Aspect Ratio*	Ref.
KBr	187	21	26	807	[16]
KBr	17	80	50	3076	[19]
$CuCl_2$	800	10-50	100	500	[20]
$CuCl_2$	881	3.2	102	31	[21]
$Fe(NO_3)_3$	25.5	40	45	888	[22]
$FeCl_3$	100	3.52	96	3.6	[13]

*: Average values

$CuCl_2$ solution dissociates into Cu^{+2} and Cl^- ions, and both ions play a critical role in AgNW synthesis. Cl^- ions electrostatically attract the positively charged Ag^+ ions, which helps to prevent them from agglomerating and promotes the formation of stable Ag seeds. Also, the reaction between Cl^- ions and Ag^+ ions leads to the formation of AgCl, which is a precipitate that can be removed from the solution [10] . This aids in lowering the concentration of free Ag^+ ions in the solution, thereby preventing the uncontrolled growth of Ag seeds. Cu^{+2} ions are changed to Cu^+ ions in the EG solution. This reaction consumes electrons, which can help to prevent the oxidation of AgNWs. Cu^+ ions can react with atomic oxygen, which is a byproduct of the AgNW synthesis reaction. Atomic oxygen can block the reactive sites on the surface of AgNWs, which can prevent their growth. By scavenging atomic oxygen, Cu^+ ions can help to promote the development of longer AgNWs [15,20].

The lowest length and aspect ratio were obtained in AgNWs produced with $Fe(NO_3)_3$ additive. Much like copper ions, Fe(II) extracts atomic oxygen located at the surface of silver nanostructures. The interaction with ethylene glycol (EG) for reduction contends with the oxidation caused by atomic oxygen, resulting in the establishment of an equilibrium between Fe(III) and Fe(II) [23,24].

In all syntheses, a significant amount of irregular shaped silver nanoparticles was observed alongside AgNWs. Especially in the synthesis involving $CuCl_2$, the formation of silver nanocubes and nanotriangles was observed. The Ag/Salt molar ratio was chosen to be ≈5000 in our study, although to the ratio of ≈100 in many studies in the literature as summarized in Table 2. The objective of using a minimal amount of salt is to observe the effect of trace amounts of salt and to minimize environmental impact and financial burden. The shorter length of the obtained nanowires compared to other studies in the literature is attributed to the lower amount of salt used. The environmental hazards of the salts used are outlined in Table 3. By reducing the salt content, the environmental footprint of AgNW production is minimized, making the process more sustainable and economically viable [25–27].

Table 3
Environmental ımpact of salts used in AgNW production.

Salt Types	Environmental Impact	Ref.
KBr	The Br^{-} anion, at high concentrations, can be a source of concern for both human and ecosystem health.	[25]
$CuCl_2$	Cu^{+2}, being a heavy metal, can lead to soil and water pollution. It threatens biological processes.	[26]
$Fe(NO_3)_3$	Ferric ions at high concentrations can accumulate in water and soil, leading to pollution.	[27]

Figure 3 depicts the normalized X-ray diffractograms of AgNWs. The observed peaks located at diffraction angles of 2θ 38.02°, 44.20°, 64.20° and 77.3° corresponded to (111), (200), (220) and (311) Bragg reflections of face-centered cubic Ag (JCPDS card 04-0783) [24,28]. The (111) diffraction peaks of all AgNWs were observed at the same 2θ value (see the zoomed-in graph of the (111) peaks Figure 3). This consistency indicates that the crystal structure of AgNWs remains unaffected by the different salt types used in the synthesis process, suggesting uniformity in the lattice parameters and absence of significant strain or size-related variations among the samples. Additionally, no salt related peaks were observed in the XRD pattern, indicating that all salts were successfully removed during the washing and cleaning stages.

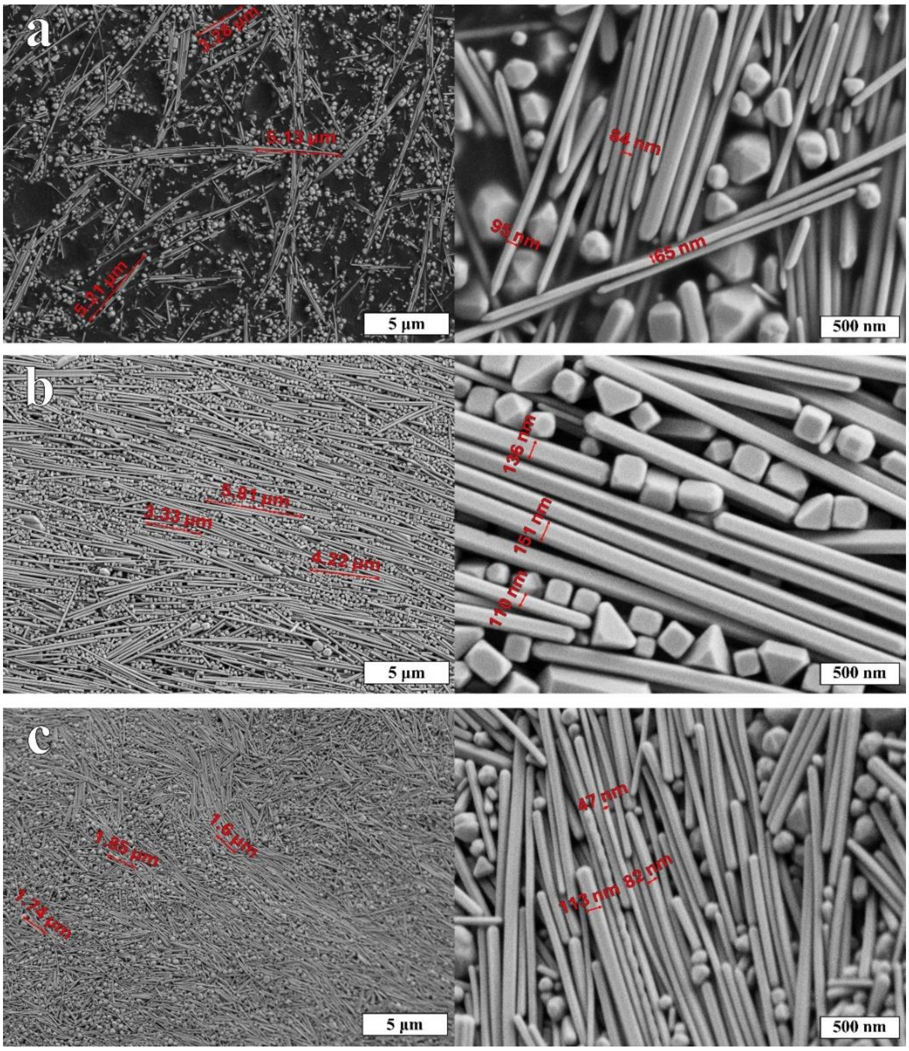

Figure 2
SEM images of AgNWs synthesized with different salts are presented at 10,000× magnification, with insets showing higher magnification at 90,000×: (a) KBr, (b) $CuCl_2$, (c) $Fe(NO_3)_3$.

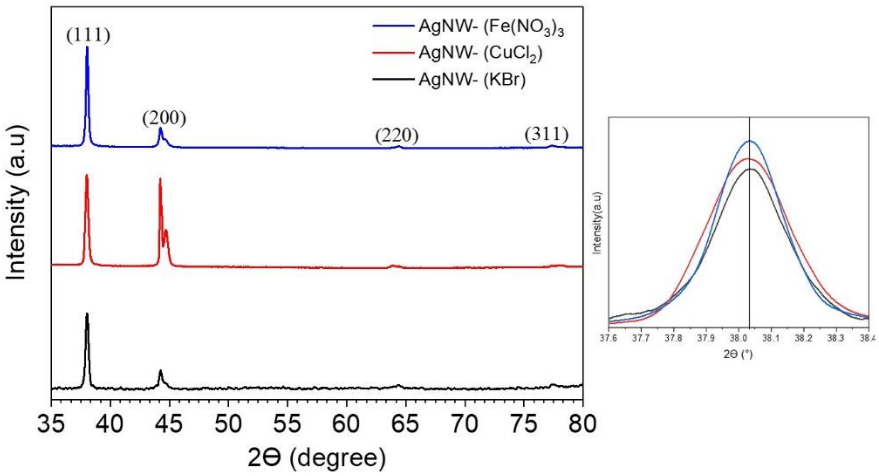

Figure 3
XRD patterns of AgNW's prepared with different type of metal salts.

It is observed that the diffraction peak signals at (111) are larger than those at (200) for the KBr and $Fe(NO_3)_3$ samples. This shows that the atom density in the (111) plane is higher than in the other planes, and atoms prefer to settle on the (111) crystal surface, suppressing crystal growth along other planes. However, this situation is different for $CuCl_2$ samples and the peak corresponding to the (200) plane has almost the same intensity as the peak belonging to the (111) plane. The ratio of area under peaks (111)/ (200) for KBr, $CuCl_2$, and $Fe(NO_3)_3$ is 2.8, 0.9, and 2.9 respectively. This is due to the presence of well-shaped silver nano cubes and nanotriangles as well as nanowires in the $CuCl_2$ sample, as observed in the SEM analysis (Figure 2).

CONCLUSION

AgNWs were synthesized through a polyol method utilizing trace amounts of $Fe(NO_3)_3$, $CuCl_2$, and KBr in conjunction with NaCl. The production using KBr resulted in obtaining AgNWs with the highest aspect ratio, yielding AgNWs with a length of 6.2 ± 2.5 µm. In the synthesis assisted by $CuCl_2$, besides nanowires, a substantial amount of other silver nanostructures such as nanocubes and nanotriangles were observed. XRD analyses indicate the absence of any other salt or residual phase besides metallic silver. We envision that our results would show that metal salts, even in trace amounts, play a decisive role in the production of AgNW obtained via polyol process.

Ethical Statement
This study is an original research article designed and developed by the authors.

Author Contributions
Research Design (CRediT 1) M.K. (%20) – İ.S.D. (%20) – E.B.E. (%60)
Data Collection (CRediT 2) M.K. (%50) – İ.S.D. (%40) – E.B.E. (%10)
Research - Data Analysis – Validation (CRediT 3-4-6-11) M.K. (%50) – İ.S.D. (%40) – E.B.E. (%10)
Writing the Article (CRediT 12-13) M.K. (%20) – İ.S.D. (%20) – E.B.E. (%60)
Revision and Improvement of the Text (CRediT 14) M.K. (%25) – İ.S.D. (%25) – E.B.E. (%50)

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Conflict of Interest
The authors have no conflicts of interest to disclose for this study.

Sustainable Development Goals (SDG)
Sustainable Development Goals: 9 Industry, innovation and infrastructure.

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