

## A new one-step electrospun polyvinyl alcohol nanofibers with uniformly dispersed silver nanoparticles

*Gümüş nanoparçacıkların eşit olarak dağıldığı yeni tek adımlı elektroğrılmış polivinil alkol nanofiberleri*

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• Received: 23.10.2025

• Accepted: 20.02.2026

### Abstract

This study makes electrospun polyvinyl alcohol (PVA) nanofibers that are functionalized with silver nanoparticles (AgNPs) in one step using a new and environmentally safe process. This new approach dissolves PVA directly in a colloidal solution of pre-synthesized AgNPs. This solution is made utilizing industrial-grade glucose as a cheap and scalable reducing agent in an environmentally friendly fashion. This idea gets rid of hazardous surfactants and processing steps by exploiting PVA's dual role as a fiber-forming matrix and natural nanoparticle stabilizer. FTIR, FE-SEM, EDX, and elemental mapping showed that AgNPs were made with an average size of  $90 \pm 15$  nm and were evenly spread throughout the PVA nanofibers. Adding AgNPs to the solution made it more conductive, which made the average fiber diameter go from 536 nm to 337 nm, making the fibers more uniform. This work creates a platform for making functional nanofibers that is strong, scalable, and cost-effective. The proposed materials might connect lab-scale innovation with industrial-scale production for antimicrobial wound dressings, smart packaging, water purification membranes, and sensor technologies.

**Keywords:** Electrospinning, Green chemistry, Nanocomposites, Poly(vinyl alcohol), Silver nanoparticles

### Öz

*Bu çalışma, yeni ve çevre dostu bir süreç kullanarak, gümüş nanoparçacıkları (AgNPs) ile fonksiyonelleştirilmiş elektroçekilmiş polivinil alkol (PVA) nanoliflerini tek adımda üretmektedir. Bu yeni yaklaşım, PVA'yı önceden sentezlenmiş AgNP'lerin koloidal çözeltisinde doğrudan çözmektedir. Bu çözüm, endüstriyel sınıf glikozun çevre dostu bir şekilde ucuz ve ölçeklenebilir bir indirgeyici madde olarak kullanılmasıyla üretilmiştir. Bu fikir, PVA'nın hem lif oluşturu bir matris hem de doğal nanopartikül stabilizatörü olarak çift yönlü rolünden yararlanarak tehlikeli yüzey aktif maddelerden ve işlem adımlarından kurtulur. FTIR, FE-SEM, EDX ve element haritalaması, AgNP'lerin ortalama  $90 \pm 15$  nm boyutunda üretildiğini ve PVA nanoliflerine eşit olarak dağıldığını göstermiştir. Çözeltiye AgNP'ler eklenmesi, çözeltiyi daha iletken hale getirdi ve bu da ortalama lif çapının 536 nm'den 337 nm'ye düşmesini sağlayarak lifleri daha düzgün hale getirdi. Bu çalışma, güçlü, ölçeklenebilir ve uygun maliyetli fonksiyonel nanolifler üretmek için bir platform oluşturmaktadır. Önerilen malzemeler, antimikrobiyal yara örtüleri, akıllı ambalajlar, su arıtma zarları ve sensör teknolojileri için laboratuvar ölçekli yeniliği endüstriyel ölçekli üretime bağlayabilir.*

**Anahtar kelimeler:** Elektroğirme, Yeşil kimya, Nanokompozitler, Poli(vinil alkol), Gümüş nanoparçacıklar

### 1. Introduction

Silver has been widely used throughout history due to its antimicrobial properties in both medical and everyday applications. Silver compounds have played a crucial role in various biomedical applications, including wound healing, disinfection, and infection control (Rai et al., 2009; Hadrup & Lam, 2014). Recent progress in nanotechnology has resulted in extensive research on the synthesis and properties of silver nanoparticles (AgNPs). The synthesis of AgNPs by both physical and chemical methods and the properties of the obtained nanoparticles have been published in many studies in the literature. These studies highlight

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the production of nanoparticles by chemical reduction due to several advantages, such as high yield and a narrow range in particle size distribution. The use of non-toxic reducing agents under mild conditions represents a significant advancement in green chemistry synthesis (Oliveira et al., 2005; Siddiqui et al., 2023). In theory, silver nanoparticle suspensions can be pure; however, in practice, they are more likely to be mixtures containing silver ions, nanoparticles, sub-nanometer particles, and aggregated nanoparticles of nano or larger sizes. Therefore, the characterization of the obtained nanostructures is of great importance to assess whether the expected efficacy of the nanoparticles is achieved (Bouwmeester et al., 2011). Due to nanoscale size, AgNPs exhibit large surface area, unique optical and electrical properties, and significant antibacterial and antifungal effects, making them highly attractive materials (Morones et al., 2005; Miura & Shinohara, 2009).

Nanofibers, materials composed of fibers with nanometer-scale diameters, are typically produced using techniques such as electrospinning. These fibers possess advantages such as a high surface area, high porosity, and mechanical durability, making them useful in diverse applications, including biomedical fields, filtration, textiles, and composite materials (Li & Xia, 2004; Greiner et al., 2007). Functionalization of nanofibers, which involves incorporating nanoparticles with specific physical or chemical properties, enhances their functionality and broadens their range of applications. In this context, silver nanoparticle-functionalized nanofibers have enormous potential, particularly for antibacterial applications (Tian et al., 2007; Reneker & Yarin, 2008; Yetisen et al., 2016). Polyvinyl Alcohol (PVA) is a widely employed synthetic polymer due to its water solubility, biocompatibility, and biodegradability. PVA is extensively utilized in various biomedical and industrial applications, such as hydrogels, biosensors, tissue scaffolds, and pharmaceutical carriers (Jin, 2022; Nathan et al., 2023). PVA nanofibers are particularly preferred due to their high mechanical strength, excellent biodegradability, and environmentally friendly nature. PVA nanofibers produced via electrospinning are commonly employed in biomedical fields and environmental applications such as water purification (Huang et al., 2003; Lee et al., 2023). Silver nanoparticle-loaded PVA nanofibers are functional nanocomposites that combine the mechanical strength and biocompatibility of PVA with the antimicrobial properties of AgNPs. These structures hold significant potential for applications in wound dressings, drug delivery systems, biosensors, water filtration, and antimicrobial coatings (Velgosova et al., 2023). The synthesis and characterization of AgNP-loaded PVA nanofibers are critical steps for their effective utilization. While prior studies have reported the synthesis of PVA-AgNPs composites using glucose (Meshram et al., 2013; Tang et al., 2013; Pham et al., 2021), this study introduces a novel and simplified approach. Commercial (industrial-grade) glucose was utilized for the green synthesis of AgNPs and subsequently dissolved PVA directly into the pre-synthesized AgNPs colloid. This method eliminates the need for surfactants or post-processing steps. The single-step integration of green-synthesized AgNPs into the electrospinning precursor, coupled with the use of low-cost industrial glucose, distinguishes this study and underscores its scalability and economic feasibility compared to methods using analytical-grade reagents or multi-step routes.

Therefore, this study focused on the synthesis and physicochemical characterization of PVA nanofibers functionalized with AgNPs, which will be especially important for biomedical applications. In this study, a colloidal solution containing AgNPs was prepared using the green chemistry strategy, and PVA was dissolved in this colloidal solution to prevent the aggregation of AgNPs and to obtain a homogeneous polymer solution. The solution was transformed into AgNP-functionalized PVA nanofibers (AgNPs/PVA) using the electrospinning technique. This method may decrease costs by 70–80% and shows a scalable, environmentally friendly way that can be used in industry.

## 2. Materials and methods

### 2.1. Materials and equipment

Silver nitrate salt was used as the silver source, and polyvinyl alcohol (PVA) (Mw 13,000–23,000 g/mol, 87–89% hydrolyzed) was purchased from Sigma. The reducing agent, glucose (C<sub>6</sub>H<sub>12</sub>O<sub>6</sub>), was of industrial grade (purity 90–95%, HNS KIMYA). This choice was motivated by the goal of developing a cost-effective and scalable synthesis route, moving beyond the analytical-grade reagents typically used in laboratory-scale studies. Deionized water was supplied by the Millipore Milli-Q IQ 7000 Ultrapure Water System.

UV–vis. absorption spectra were recorded by using a Shimadzu UV-3600 Plus UV-Vis. absorption spectrophotometer. The concentrations of the prepared solutions containing colloidal silver nanoparticles were measured with a Perkin Elmer AAS, AAnalyst 400 Atomic Absorption Spectrometer (AAS). Fourier Transformed Infrared Spectroscopy (FT-IR) spectra were taken on a Perkin Elmer FT-IR spectrometer between 4000 and 400  $\text{cm}^{-1}$  using ATR. Inovenso's Basic System electrospinning device was used to prepare the nanofibers. The morphological characteristics of the electrospun PVA fibers were analyzed using a ZEISS Gemini SEM 300 field emission scanning electron microscope (FE-SEM). Additionally, an OXFORD Link ISIS energy-dispersive X-ray spectroscope was utilized in conjunction with FE-SEM for elemental analysis and mapping. ImageJ software was used to measure the size of the nanoparticles and diameters of the electrospun nanofibers.

## 2.2. Preparation of colloidal silver nanoparticles

Colloidal silver nanoparticles were synthesized with minor modifications based on published procedures (Birimoglu & Sonmez, 2023). Specifically, a 100 mL solution of 2.3 mmol glucose was prepared in a 250 mL beaker. Subsequently, 100 mL of a freshly prepared 4.7 mmol silver nitrate solution was added dropwise using a dropping funnel. The reaction took place at room temperature for two hours, with stirring at 500 rpm the whole time. The obtained silver nanoparticles were separated from the medium by centrifugation and washed several times with pure water to remove the unreacted silver nitrate and glucose. The structure of the colloidal silver nanoparticles was elucidated by UV-Vis, AAS, and FE-SEM.

## 2.3. Electrospinning methods for PVA and AgNPs/PVA nanofibers

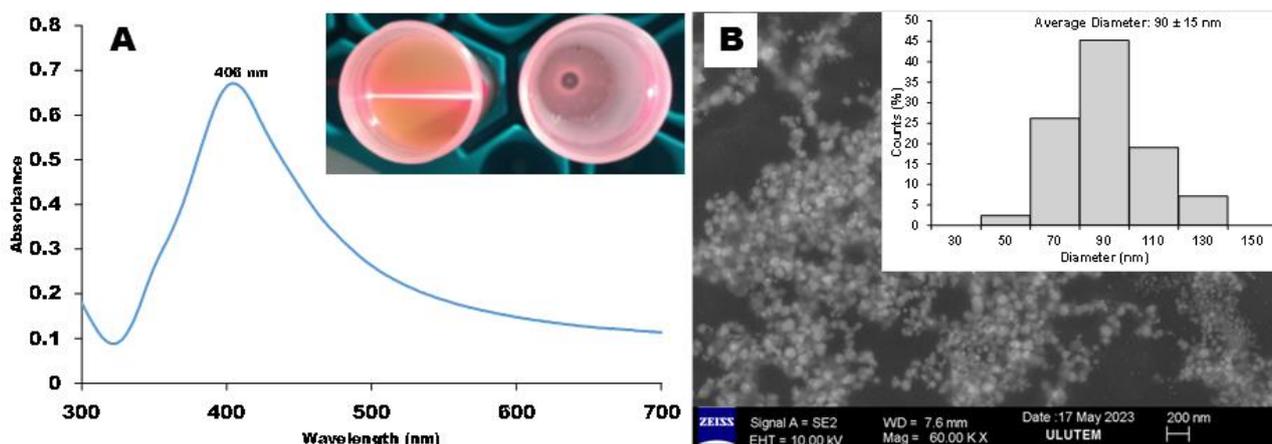
The colloidal AgNPs solution (2500 ppm) was diluted to 1000 ppm using ultrapure water. A 10% (w/w) PVA solution was then prepared by gradually adding PVA polymer into the diluted AgNPs colloid under continuous stirring. The mixture was stirred at room temperature for 24 hours to obtain a homogeneous electrospinning solution. For comparison, a pure PVA solution (10% w/w) was prepared similarly using ultrapure water. The homogeneous solutions were put into a 10 mL syringe, which was then connected to the electrospinning apparatus. Electrospinning parameters were optimized based on preliminary trials to obtain bead-free, uniform nanofibers. Parameters such as voltage (15 kV), tip-to-collector distance (15 cm), and flow rate (20  $\mu\text{L}/\text{min}$ ) were selected accordingly. The produced nanofibers were collected onto aluminum foil. Pure polyvinyl alcohol and silver nanoparticle-functionalized polyvinyl alcohol (AgNPs/PVA) electrospun nanofibers were characterized using FTIR, FE-SEM, EDX, and mapping techniques. Electrospinning parameters (voltage, tip-to-collector distance, and flow rate) were optimized through preliminary experiments. Parameters were chosen to ensure bead-free and uniform fibers. A single AgNPs concentration (1000 ppm) was selected for this initial feasibility study; future work will systematically vary concentrations and spinning parameters to study their effect.

## 3. Results and discussion

### 3.1. Synthesis and characterization of colloidal silver nanoparticles

Previously published studies in the literature (Birimoglu & Sonmez, 2023) were used as references for the preparation of colloidal silver nanoparticles. A review of the literature indicates that silver nitrate salt is widely used as a silver source for the synthesis of silver nanoparticles (Carotenuto et al., 2000; Dawadi et al., 2021; Ivanov et al., 2023). In this study, silver nitrate, a water-soluble silver salt, was selected as the initial reactant. To reduce  $\text{Ag}^+$  ions to metallic  $\text{Ag}^0$ , various reducing agents, including  $\text{NaBH}_4$ ,  $\text{LiAlH}_4$ , citric acid, and tannic acid, have been employed in previous studies (Chou et al., 2005). Glucose used as a reducing agent in previous studies was of analytical purity (Zhang et al., 2022; El-Shishtawy et al., 2023), and in this study, industrial-grade glucose was used for the first time, and the reduction reaction was carried out at room conditions in accordance with the green chemistry strategy (Bhattarai et al., 2018). Industrial-grade glucose, with a lower purity (90-95%) and significantly lower cost compared to analytical-grade glucose ( $\geq 99\%$ ), proved effective for nanoparticle synthesis. This paper highlights the practical, scalable nature of this green method. Additionally, this study highlights a simple, effective, and low-cost method for commercially producing colloidal silver nanoparticles and presenting them as antibacterial products for materials such as textiles, paints, and glues.

AgNPs were produced successfully in the form of colloidal nanoparticles through the sol-gel technique (Maharjan et al., 2020). The formation of nanoparticles was first indicated by the change of color of the reaction mixture to red like other reports on the AgNP synthesis (Balavandy et al., 2014). The optical characteristics and the existence of the synthesized AgNPs were determined using UV-Vis spectroscopy, a widely used method to characterize nanoparticles and complexes (Kapoor, 1998; Behzadi et al., 2015). The spectrum obtained had a clear peak of absorption at 406 nm (Figure 1A). Such a peak falls in the typical 390-420nm wavelength range of surface plasmon resonance of AgNPs (Sarhan & Fahmy, 2021) thus confirming their success in formation. Moreover, the concentration of metallic silver in the colloidal solution was measured using the Atomic Absorption Spectroscopy (AAS) which is a reputable chemical method of measuring the concentration of metals in liquid samples due to their ability to absorb certain wavelengths (Lewen, 2011). The AAS test indicated the concentration of silver in the synthesized colloidal solution was about 2500 ppm under the reaction conditions which were used.



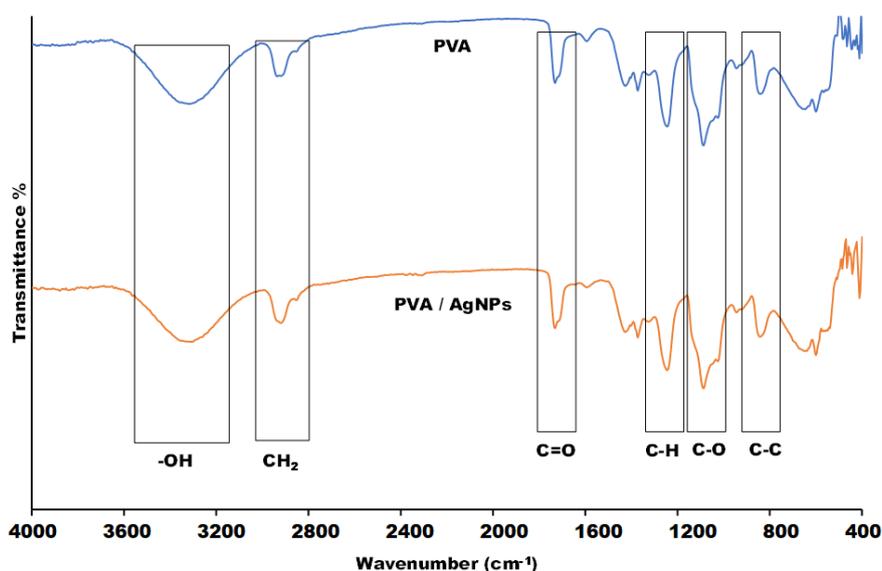
**Figure 1.** UV-Vis spectra showing maximum absorption at 406 nm wavelength of colloidal silver nanoparticles (A); the inset shows the Tyndall effect visualized by laser light scattering through the colloidal silver solution (left) and pure water as a reference (right); and FE-SEM image of AgNPs taken at 60K magnification (B); the inset shows the histogram of particle size distribution.

The FE-SEM images of the sample are presented in Figure 1B. The images, captured at 60,000× magnification with a 200 nm scale bar, reveal that there is a narrow size distribution of AgNPs, and the average particle size distribution of the silver nanoparticles is  $90 \pm 15$  nm. A review of the literature shows that silver nanoparticles synthesized under different reaction conditions and with various reagents exhibit diverse particle sizes and morphologies (Cumberland & Lead, 2009; Khan et al., 2011; Desai et al., 2012). In this study, AgNPs with particle sizes of  $90 \pm 15$  nm were successfully prepared under the mild conditions using industrial glucose with medium reducing power, and this was determined by the characterization techniques.

### 3.2. Preparation and characterization of PVA electrospun nanofibers functionalized with silver nanoparticles (AgNPs/PVA)

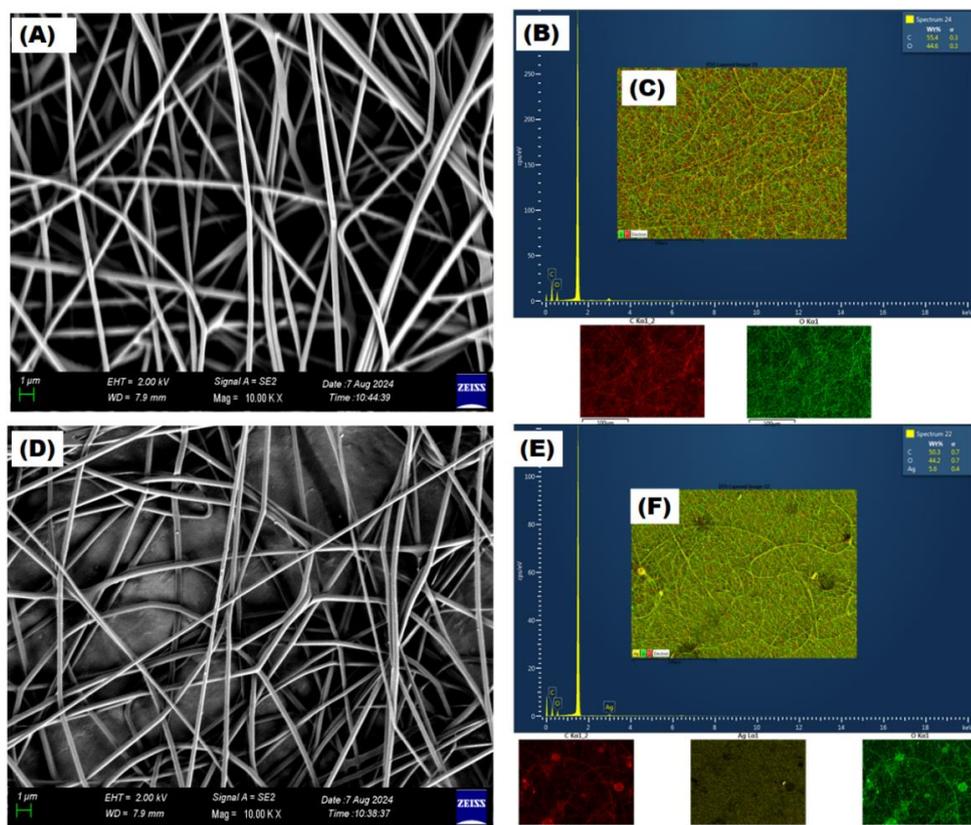
Many studies in the literature report that PVA polymer is used as a stabilizing agent to prevent the aggregation of nanoparticles (Becaro et al., 2015; Díaz-Cruz et al., 2016; Badineniet al., 2021). This study utilized PVA polymer as both the carrier polymer for the nanofibers and as a stabilizing agent for the nanoparticles in an aqueous medium. The preparation of polyvinyl alcohol electrospun nanofibers functionalized with silver nanoparticles was modified based on previous studies in the literature (Zhang et al., 2016). However, future studies may investigate a range of concentrations to optimize fiber performance. This ratio (1000 ppm) was chosen based on the stability of the colloid and the observed quality of the resulting fibers. Different AgNP loadings were not tested in this study, which is noted as a limitation and will be addressed in future investigations.

Figure 2 displays the FTIR spectra for both pure PVA nanofibers and those containing AgNPs. In the spectra, broad vibrational bands corresponding to  $-OH$  functional groups were observed at  $3321 \text{ cm}^{-1}$  and  $3348 \text{ cm}^{-1}$  for pure PVA and AgNPs/PVA nanofibers, respectively.



**Figure 2.** FTIR spectra of electrospun nanofibers.

The vibrational bands of aliphatic  $-CH$  groups appeared at  $2912\text{ cm}^{-1}$  and  $2943\text{ cm}^{-1}$ , while characteristic vibrational bands of  $C=O$  groups in partially hydrolyzed structures were detected at  $1732\text{ cm}^{-1}$  and  $1735\text{ cm}^{-1}$ , respectively. Bending vibrational bands of  $-CH_2$  groups were noted at  $1423\text{ cm}^{-1}$  and  $1438\text{ cm}^{-1}$ , and stretching bands of  $C-O$  in acetyl groups appeared at  $1087\text{ cm}^{-1}$  and  $1095\text{ cm}^{-1}$ . Additionally,  $C-C$  stretching bands were observed at  $848\text{ cm}^{-1}$  and  $852\text{ cm}^{-1}$ , respectively. The FTIR results for the PVA electrospun nanofibers were in line with what other studies have found (Thomas et al., 2004; Tretinnikov & Zagorskaya, 2012). When the FTIR spectrum of AgNPs/PVA electrospun nanofibers was analyzed, minor red shifts were detected in all characteristic peaks. This minor red shift in the  $O-H$  stretching band from  $3321$  to  $3348\text{ cm}^{-1}$  and in the  $C=O$  band from  $1732$  to  $1735\text{ cm}^{-1}$  was observed. The small alterations suggest a weak coordination contact between Ag nanoparticles and the oxygen-containing groups of PVA, so affirming the integration of nanoparticles at the molecular level. These shifts are attributed to the presence of silver nanoparticles in the structure (Tombe et al., 2013).



**Figure 3.** FE-SEM images, EDX, and mapping results of pure PVA (A, B, and C) and AgNPs/PVA electrospun nanofibers (D, E, and F). Scale bars in FE-SEM images represent  $1\text{ }\mu\text{m}$ , and images were acquired at  $10,000\times$  magnification. Elemental mapping results show the homogeneous distribution of carbon (C), oxygen (O), and silver (Ag) atoms.

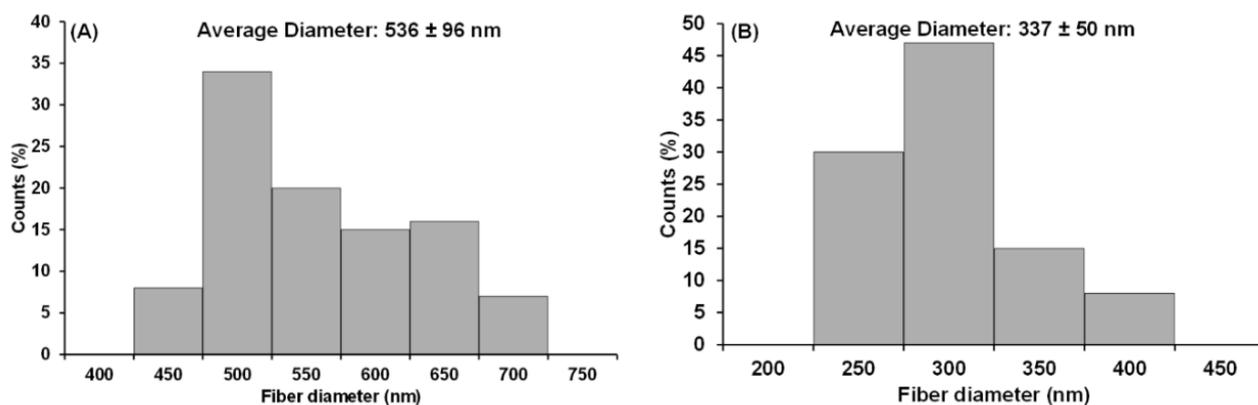
Figure 3 shows the morphological images, EDX analysis, and elemental mapping results for the PVA and AgNPs/PVA electrospun nanofibers. The FE-SEM image of the PVA electrospun nanofibers, captured at 10,000× magnification (Figure 3A), reveals that the produced fibers are smooth, bead-free, droplet-free, and consist of thin nanofibers with an average diameter of approximately 536 nm. The EDX spectrum and elemental mapping findings indicate that the PVA nanofibers contain carbon (C) and oxygen (O) elements (Figures 3B and 3C) (Kawady et al., 2022). Figures 3D, 3E, and 3F illustrate the FE-SEM morphological image, EDX spectrum and results, and elemental mapping with the distribution of elements for AgNPs/PVA electrospun nanofibers, respectively. FE-SEM analysis showed that the AgNPs/PVA nanofibers had a narrower fiber diameter distribution than pure PVA nanofibers made in the same way. The average diameter was about 337 nm. This reduction in fiber diameter is attributed to the increased conductivity provided by the silver metal within the AgNPs/PVA nanofibers, which leads to the formation of smoother, more uniform, and thinner fibers (Tombe et al., 2013; Uthaman et al., 2021). Moreover, the EDX spectrum and elemental mapping results of the AgNPs/PVA electrospun nanofibers demonstrate that the AgNPs are homogeneously distributed within the PVA fibers.

**Table 1.** Quantitative EDX data of AgNPs/PVA nanofibers

Element	wt%	at%
C	50.3	59.9
O	44.2	39.4
Ag	5.5	0.7

Additionally, the quantitative EDX examination showed that the composition was 50.3 wt% C, 44.2 wt% O, and 5.5 wt% Ag, which is around 59.9, 39.4, and 0.7 atomic percent (at%), respectively (Table 1). The Ag concentration is just 0.7 at%, but the fact that it is equally distributed throughout the PVA matrix indicates that the nanoparticles were added without joining together. These findings confirm the successful fabrication of Ag-functionalized PVA nanofibers. Nevertheless, to unambiguously confirm AgNPs incorporation, complementary XRD analysis will be performed in future studies.

The average fiber diameter decreased from  $536 \pm 96$  nm for pure PVA to  $337 \pm 50$  nm for AgNPs/PVA, indicating a narrower size distribution and improved uniformity due to enhanced conductivity (Figure 4) (Lin & Yang, 2005; Dubey et al., 2015). The reduction in nanofiber diameter (from 536 nm to 337 nm) and improved surface uniformity in the AgNPs/PVA composite are directly attributed to the enhanced conductivity and nanoparticle dispersion achieved through this colloid based preparation (Montes-Hernandez et al., 2021; Guo et al., 2023). This outcome supports the effectiveness of the simplified one step approach. While EDX and mapping confirmed the presence and distribution of silver, future studies will include X-ray diffraction (XRD) analysis to further verify the crystalline nature of the incorporated AgNPs. Furthermore, this method circumvents challenges associated with in situ reduction or post-electrospinning modification processes. The use of commercial glucose further increases the practicality and reproducibility of the synthesis strategy for industrial scaling.



**Figure 4.** Fiber diameter distribution histograms of pure PVA (A) and AgNPs/PVA (B) electrospun nanofibers.

#### 4. Conclusion

This study successfully demonstrated a novel, simple, and environmentally benign strategy for the fabrication of AgNPs-functionalized PVA nanofibers via electrospinning. The methodology's core innovation lies in its two-fold approach: first, the green synthesis of AgNPs using cost-effective industrial-grade glucose as a reducing agent under mild conditions; and second, the direct dissolution of PVA polymer in the pre-synthesized AgNPs colloid to form a homogeneous electrospinning precursor. This strategy efficiently utilizes PVA's dual function as both a fiber-forming matrix and a nanoparticle stabilizer, eliminating the need for toxic chemicals, additional surfactants, or complex post-processing steps. The use of industrial-grade glucose, a key aspect of this work, underscores the scalability and economic feasibility of the proposed process, distinguishing it from laboratory-scale studies that rely on high-purity reagents. This approach presents a significant step towards the commercial viability of such functional nanomaterials. Comprehensive characterization techniques, including UV-Vis spectroscopy, AAS, FE-SEM, EDX, elemental mapping, and FTIR, confirmed the successful synthesis of AgNPs with an average size of  $90 \pm 15$  nm and their uniform distribution within the PVA nanofibers. A significant finding was the reduction in the average fiber diameter from 536 nm (pure PVA) to 337 nm (AgNPs/PVA), attributed to the increased electrical conductivity of the polymer solution upon the addition of metallic nanoparticles, which resulted in smoother and more uniform fibers. While this study primarily focuses on establishing a robust synthesis and characterization framework, the homogeneous distribution of AgNPs within the fibers strongly suggests high potential for applications in antimicrobial wound dressings, water filtration membranes, and smart packaging materials. It should be noted that antibacterial efficacy tests were not conducted in this initial study, which represents a limitation. Future work will include quantitative antimicrobial assays to evaluate the potential of AgNPs/PVA nanofibers in biomedical and hygienic applications.

#### Acknowledgments

This study was supported by the Naci Topçuoğlu Vocational School Project Coordination Office (PDK) and HNS KIMYA. The analyses reported in this paper were partially performed at GAUN ULUTEM, High Technology Application and Research Center (GAUN ULUTEM resources).

#### Author contribution

The entire article was written by the corresponding author.

#### Conflicts of interest

The author stated that there are no conflicts of interest regarding the publication of this article.

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