

RESEARCH PAPER

Concentration-Engineered AgNO₃-Derived Ag Nanoparticles Embedded in Electro Spun PVA Nano fibers for Optical Band-Gap Tuning and Fluorescence Enhancement

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ARTICLE INFO

Article History:

Received 02 May 2026

Accepted 23 June 2026

Published 01 July 2026

Keywords:

Concentration engineering

Electro spinning

Nano fibers Ag

NPs fluorescence band-gap tuning

PVA

ABSTRACT

In view of the importance of polymers and their Nano fibers at the present time and their uses in many medical, engineering, industrial and other fields, and in order to improve the properties of these fibers, we have prepared, in our study, a pure PVA polyvinyl alcohol solution, then it is impregnated with AgNO₃ salt in different concentrations (1, 2, 3 wt%) and then shaped into Nano fibers using the electro spinning technique at room temperature. XRD FE-SEM, UV, and PL characterizations are used to study the structure, morphology and optical properties of materials. XRD tests showed that the PVA polyvinyl alcohol fibers have a random structure, and the doped films have a cubic crystalline structure, and it was noted that their crystalline size increases with the increase in the doping percentage. Finally, the XRD tests showed that the PVA polyvinyl alcohol fibers have a random structure, and the doped films have a cubic crystalline structure, and it was noted that their crystalline size increases with the increase in the doping percentage. FESEM measurements showed that the diameters of the doped membrane fibers decreased with increasing mixing ratio. The optical properties showed a clear positive effect when adding silver to the fiber structure, as the fiber's absorption of light increased and the energy gap of the polymer decreased with the increase of silver concentration. In contrast, the fluorescent results showed that the best intensity of the fluorescence spectra was obtained at a concentration of 3% silver. The novelty of this work is the direct correlation of AgNO₃-derived Ag nanoparticle concentration with fiber-diameter reduction, Ag crystallite growth, band-gap narrowing, and fluorescence enhancement in one electro spun PVA platform.

How to cite this article

Kamil M., Ibrahim S. Concentration-Engineered AgNO₃-Derived Ag Nanoparticles Embedded in Electro Spun PVA Nano fibers for Optical Band-Gap Tuning and Fluorescence Enhancement. J Nanostruct, 2026; 16(3):4040-4048. DOI:10.22052/JNS.2026.03.087

INTRODUCTION

Polymer fibers, specifically Nano scale ones, have attracted considerable interest for several decades in view of their unique physical and chemical nature. They have a high specific

surface area, a wide range of surface features, and outstanding mechanical properties; for example, they can be utilized in engineering membranes, textile production, and medical devices for various industries [1-6]. The

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development of nanocomposites incorporating metallic nanoparticles within a polymer matrix has attracted increasing attention in the area of materials science because of the extraordinary capacity to synthesize materials possessing variable characteristics. Polymer fibers, when they are reduced to micro size (or nanometer diameter) range, exhibit the following remarkable properties: they have a very high surface area to volume ratio as well as provide versatile surface functionalities and excellent mechanical abilities like hardness and anchoring strength in comparison to other materials. These excellent properties make polymer Nano fibers great candidates for various important applications [7-10]. Electro spinning is the major method employed to fabricate Nano fibers, based on a conventional use rate which is easily controllable [5,11,12]. Polyvinyl alcohol (PVA) was used in the process involving silver (Ag) due to its water solubility, biocompatibility, nontoxic character, and excellent chemical and thermal stability. Due to PVA's inherent features, fiber-forming behavior is quite easy; thus, PVA is extensively used in different applications [6,7]. Researchers have conducted numerous studies on the incorporation of Ag nanoparticles into electro spun Nano fibers as the basis for the fabrication of functional Nano fibrous composites to utilize the inherent properties of both Nano fibers and Ag nanoparticles [8-10]. PVA-based Nano fibers have been extensively synthesized by electro spinning techniques [11,12]. Also, some literature has talked about the influence

of inorganic additives on the properties of Nano scale fibers; but almost no information was reported on how salt affects fiber morphology. In this research, these experiments were carried out in making and analyzing PVA/Ag-based Nano fibers by electro spinning. Also, the effects of silver nanoparticle concentration on optical and structural properties in PVA films was studied by using X-ray Diffraction (XRD), field emission scanning electron microscopy (FESEM), UV-visible spectroscopy (UV-vis), and photoluminescence (PL) measurements. Therefore, the original contribution of the present study is not only the fabrication of PVA/Ag fibers, but the systematic concentration-controlled evaluation of their structural, morphological, absorption, band-gap, and photoluminescence responses under identical electro spinning conditions [2,3].

MATERIALS AND METHODS

To prepare spinning solutions, polyvinyl alcohol (PVA) which had Mw= 85,000 – 1,24,000 was dissolved in distilled water as a first stage, to obtain a pure PVA solution, where approximately (6 gm) was dissolved in (60 ml) of distilled water, then the solution was mixed for (75 min). At a temperature of (60 °C) using a magnetic stirrer, until it is completely melted and a homogeneous solution is obtained. After that, (0.084 gm) of silver nitrate (AgNO₃) was dissolved in (10 ml) of distilled water and mixed for a period not exceeding (5 min) using a magnetic mixer until the solution became homogeneous and the silver nitrate was

Table 1. Optimized electro spinning parameters employed for the fabrication of pristine PVA and PVA/Ag Nano fibers with different silver concentrations.

Processing parameters	Applied DC voltage (KV)	Needle gauge	Solution feed rate (ml/h)	Tip-to-collector distance (cm)
Operating Range	0-50	21-30	0.1-100	1-20
Optimized value	20	21	0.1	18

Table 2. Experimental design of pure PVA and AgNO₃-modified PVA nanofiber samples.

Sample code	Precursor composition	AgNO ₃ concentration	Processing condition kept constant	Purpose
PVA	Pure PVA solution	0 wt%	Same electrospinning settings	Reference sample
PVA/1%Ag	PVA + AgNO ₃	1 wt%	Same electrospinning settings	Low Ag concentration
PVA/2%Ag	PVA + AgNO ₃	2 wt%	Same electrospinning settings	Intermediate Ag concentration
PVA/3%Ag	PVA + AgNO ₃	3 wt%	Same electrospinning settings	High Ag concentration

completely dissolved in water. Then, the silver nitrate solution was added to the (PVA) polymer solution that was previously prepared, and mixed with it using a magnetic mixer at room temperature for a period of (5-10 min), with three different concentrations (1,2,3 ml). To create Nano fibers using the electro spinning system, the parameters were set as shown in Table 1. In this design, AgNO₃ was used as the Ag source/precursor, allowing the Ag-related phase and optical response to be tuned through the selected concentration range rather than by changing the polymer host. The

experimental design, sample codes, precursor compositions, and AgNO₃ concentrations are summarized in Table 2.

RESULTS AND DISCUSSION

Fig. 1 presents the XRD diffraction pattern of pure PVA Nano fibers and shows a broad peak at $2\theta = 20.35677^\circ$ indicating the semi-crystalline structure of PVA polymer. No discernible sharp peaks are in accordance with previous studies [13,14]. Fig. 2 shows diverse X-ray diffraction features of pure polyvinyl alcohol (PVA) Nano

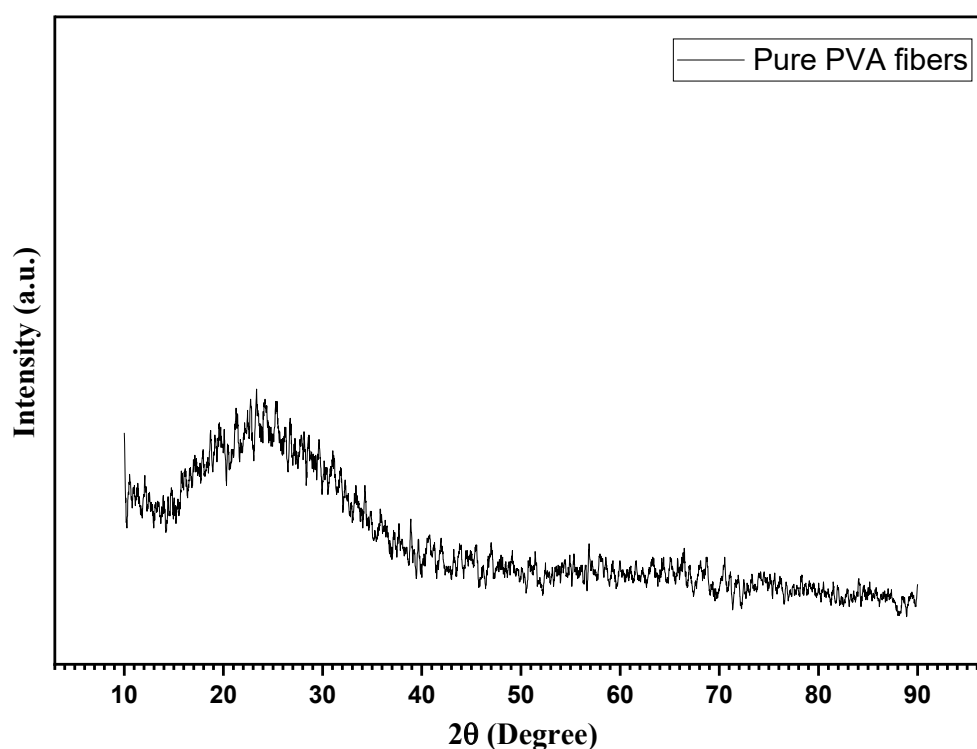


Fig. 1. XRD diffraction pattern for pure PVA Nano fiber.

Table 3. XRD structural summary of pure PVA and PVA/Ag nanofibers.

Sample	Main observed feature	2θ / planes	Calculated crystallite size (nm)	Structural interpretation
Pure PVA	Broad semi-crystalline PVA peak	20.35677°	Not calculated	Semi-crystalline/random polymer structure
PVA/2%Ag	Ag-related FCC diffraction peaks	(111), (200), (220), (311), (222)	59.63	Formation of crystalline Ag phase inside PVA fibers
PVA/3%Ag	Ag-related FCC diffraction peaks with increased size	(111), (200), (220), (311), (222)	65.32	Crystallite size increases with Ag concentration

fibers mixed with silver at concentrations of PVA/ (2%, 3%) Ag. The characteristic diffraction peaks for indices (111), (200), (220), (311), and (222) at angles $2\theta = 38.2634^\circ$, 44.13° , 60.0228° , 77.5481° , 81.5023° with a preferential (111) orientation are apparent. This suggests a crystalline structure indicative of a face-centered cubic arrangement of silver, consistent with previous literature [15-17]. The value of grain size (d) was established using the equation [17]:

$$D_{\text{avg}} = \frac{0.9 \lambda}{\beta \cos \theta} \quad (1)$$

where λ is the X-ray wavelength, β is the full width at half maximum (FWHM) in radians, and θ is the Bragg angle. The determined grain sizes of PVA/(2% Ag) and PVA/(3% Ag) Nano fibers are around 59.63 nm and 65.32 nm, indicating an increase in grain size with higher concentrations of silver nanoparticles. The main findings obtained from the XRD analysis are summarized in Table 3.

FE-SEM image of collected fiber mats is shown in Fig. 3. The diameter of PVA obtained Nano fibers varied from 150 to 400 nm (mean diameter 258.5 nm) (Fig. 2a) [18]. Seen from the FE-SEM pictures which depicted the distribution of PVA

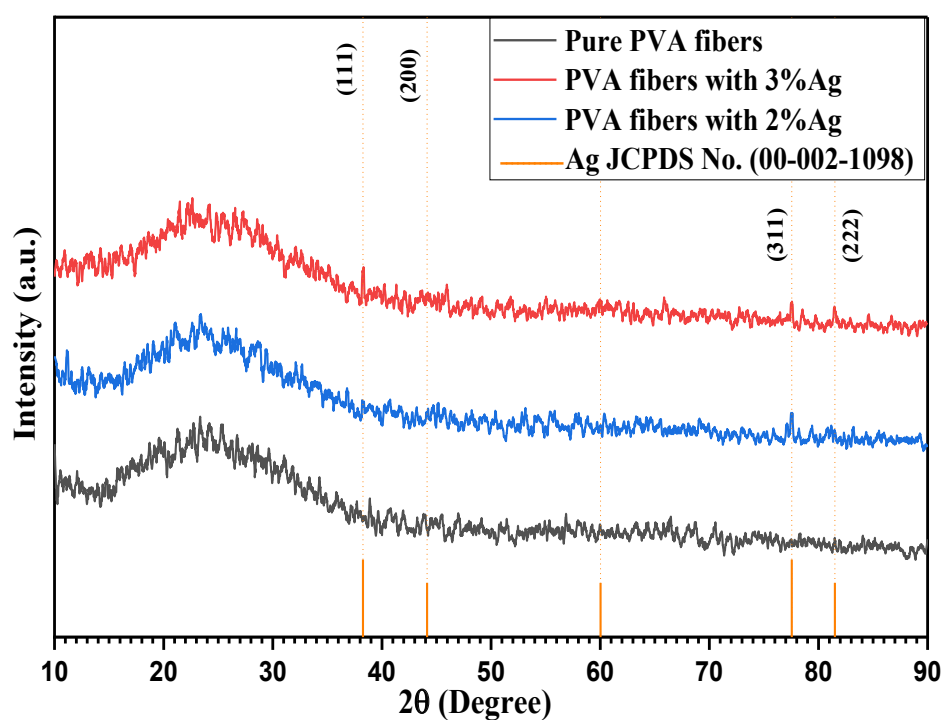


Fig. 2. XRD patterns of pure PVA and Ag-doped PVA Nano fibers with different silver contents.

Table 4. FE-SEM morphological summary of electro spun PVA/Ag Nano fibers.

Sample	Fiber diameter range (nm)	Average diameter (nm)	Morphological observation	Effect of Ag addition
Pure PVA	150-400	258.5	Continuous PVA nanofibers Smooth and homogeneous fibers without obvious structural defects	Reference morphology
PVA/Ag nanofibers	50-350	174.7		Fiber diameter decreases with Ag concentration

Nano fibers (PVA Nano fibers) mixed with AgNO₃ at concentrations of 1, 2 and 3 wt%, morphology indicated a thin film that could fit well into polyvinyl alcohol without clustering. Additionally,

the added salt exhibited uniform homogeneity within the polymer structure, reaffirming that the fibers maintained no structural defects as shown in Fig. 3b-d. As the concentration of silver changed,

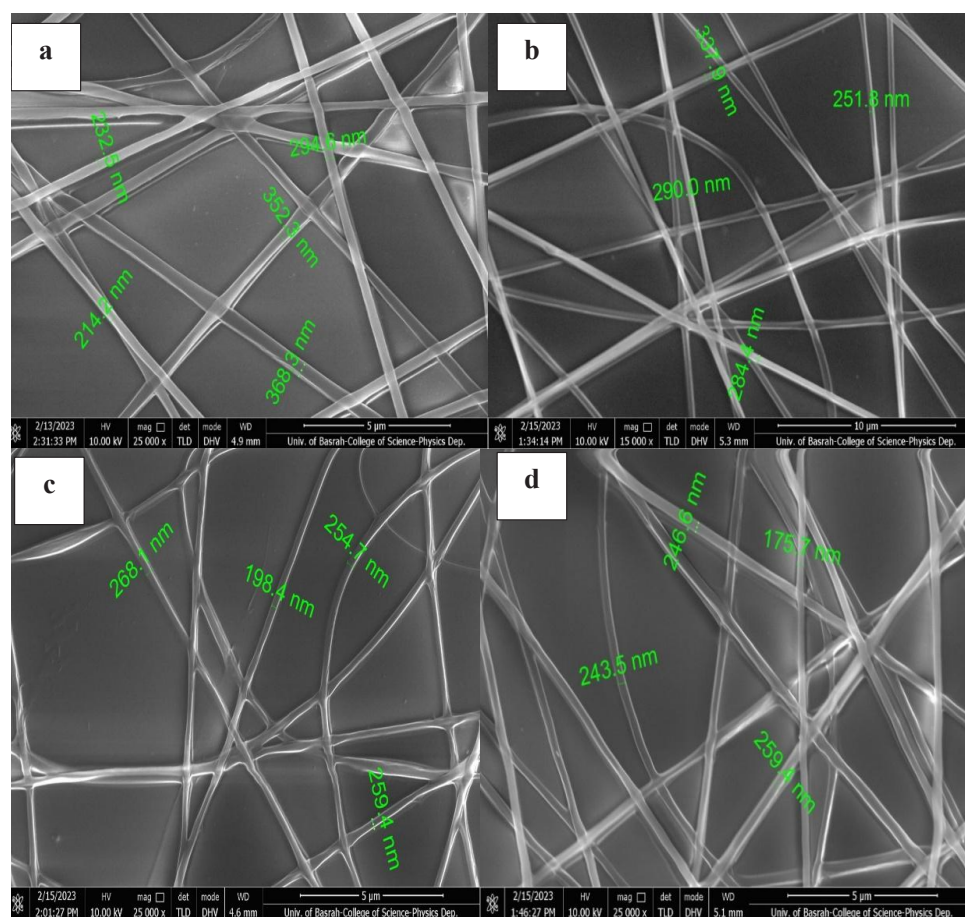


Fig. 3. FE-SEM micrographs of electro spun Nano fibers: (a) pure PVA, (b) PVA-1 wt.% Ag, (c) PVA-2 wt.% Ag, and (d) PVA-3 wt.% Ag.

Table 5. Optical absorption and band-gap variation with Ag concentration.

Sample	Absorption behavior	Optical band gap (eV)	Trend	Interpretation
Pure PVA	Broad absorption near 347 nm	4.17	Highest Eg	Transparent polymer reference
PVA/1%Ag	Absorption increases after Ag addition	4.16	Slight decrease	Initial Ag-induced charge-transfer interaction
PVA/2%Ag	Further absorption enhancement	4.15	Slight decrease	Increased dopant interaction in PVA matrix
PVA/3%Ag	Highest absorption among doped samples	4.10	Clear decrease	More defect/dipole states within the band gap

the diameters of PVA Nano fibers were noticeably decreased with 2% and 3% silvery concentrations, respectively. The diameters of these fibers varied from 50 to 350 nm and were an average of 174.7 nm size which was significantly smaller compared to pure PVA Nano fibers. This is thought to relate with the proper substitution of AgNO₃ [19]. It modified viscosity and prolonged the flow of the solution because of an increased conductivity at high-voltage application—such high voltage can facilitate the detachment of particles or minimize their agglomeration [20]. For clarity, the main FE-SEM observations, including fiber diameter and morphological characteristics, are summarized in Table 4.

The absorption spectra from thin films of pure PVA and PVA doped with silver and recorded in visual form as per the UV-visible spectra are shown in Fig. 4. Pure PVA is given a distinctive peak at 347 nm in the UV-Vis spectra with a small displacement to 350 nm, where this peak

goes up as Ag concentration increases. The result indicates that the energy band gap between PVA/Ag nanoparticles, in the PVA Nano fiber, is smaller than that between pure PVA Nano fiber due to the changes in the form of PVA Nano fiber. Interestingly, no absorption band in the 500-800 nm range was found in all samples analyzed as pure PVA is a colorless polymer that has no visible absorption showing transparency in this region [21]. The increase in the absorbing capacity with higher dopant concentrations can be ascribed to charge transfer complex formation [22], as Ag is incorporated in the PVA polymer. In Fig. 5 the energy gap (E_g) for pure PVA is 4.17 eV. E_g values for Ag-doped thin films are as follows: 4.16 eV, 4.15 eV and 4.10 eV at 1 wt%, 2 wt%, and 3 wt%, respectively. There are no alterations to the optical band gap energy in low silver-doped samples, but a decreasing decrease in optical band gap energy occurs at higher doping concentration levels. Such point defects might arise as a result

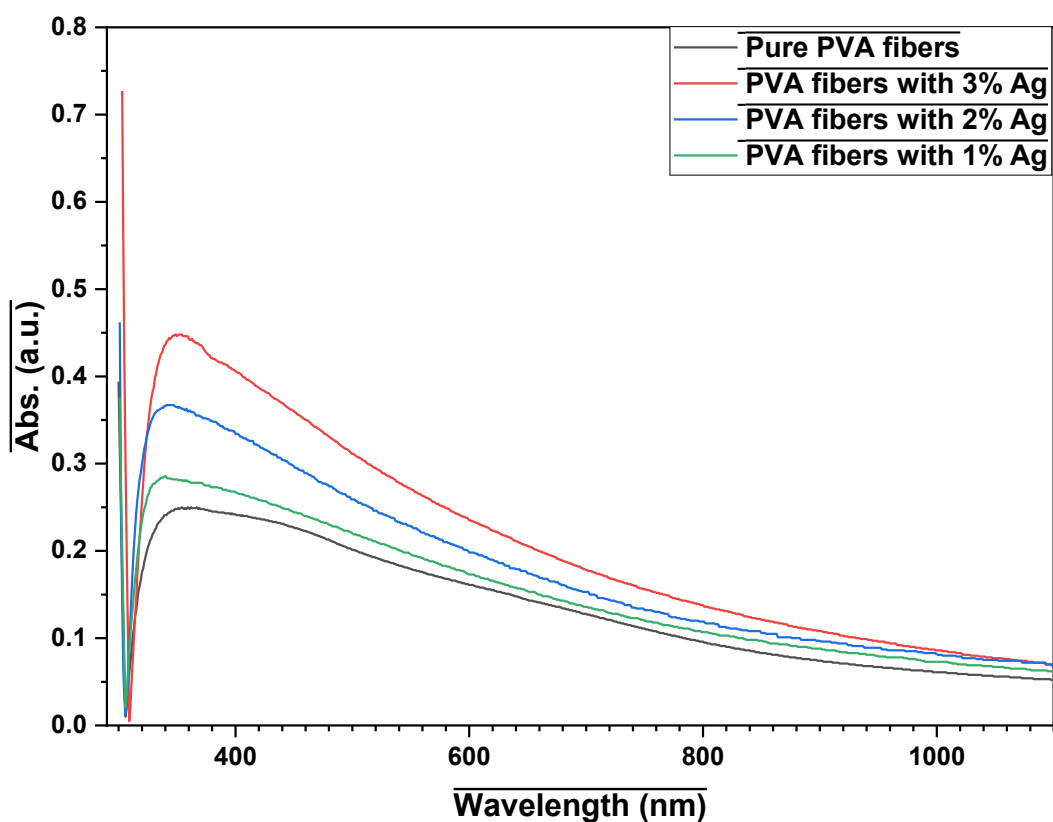


Fig. 4. The UV-vis absorption spectra for PVA Nano fiber thin film doped with Ag.

of formation of molecular dipoles in the band gap [23]. The changes in optical absorption behavior and band gap with increasing Ag concentration are summarized in Table 5.

The fluorescence spectra of pure PVA and the PVA loaded with different concentrations of Ag nanoparticles for absorption were obtained at a room temperature excitation wavelength of 350 nm. The thin films display well-defined peaks at 395 and 460 nm for the pure PVA and for the Ag-doped thin film formulations. But the strength of these peaks decreases when AgNPs are mixed into the PVA at 1% and 2% concentrations. Similarly, the width of surface Plasmon resonance peak on the surface seems to have an influence on the efficiency of electron transmission, leading to the lowering of the efficiency of fluorescence at the

lower concentrations. On the other hand, the thin film with 3 wt% Ag has very pronounced visible emission band at 650 nm. The emission at 390 nm is related to the surface trap effects, whereas the sharp emitted band at 650 nm confirms the electron-hole recombination that takes place among the nanoparticles. The increasing level of peak emission for PVA doped with NP at 3 wt% in particular suggests a redshift. This change has also been attributed to the broad band emission induced by the Nano dopants added, caused by the increase of crystalline size. Especially the increased visible emissions observed at 3 wt% Ag indicating concentration-dependent photo luminescent response highlights the effect of Ag related electronic states and differences in crystallite size on radiative recombination of PVA

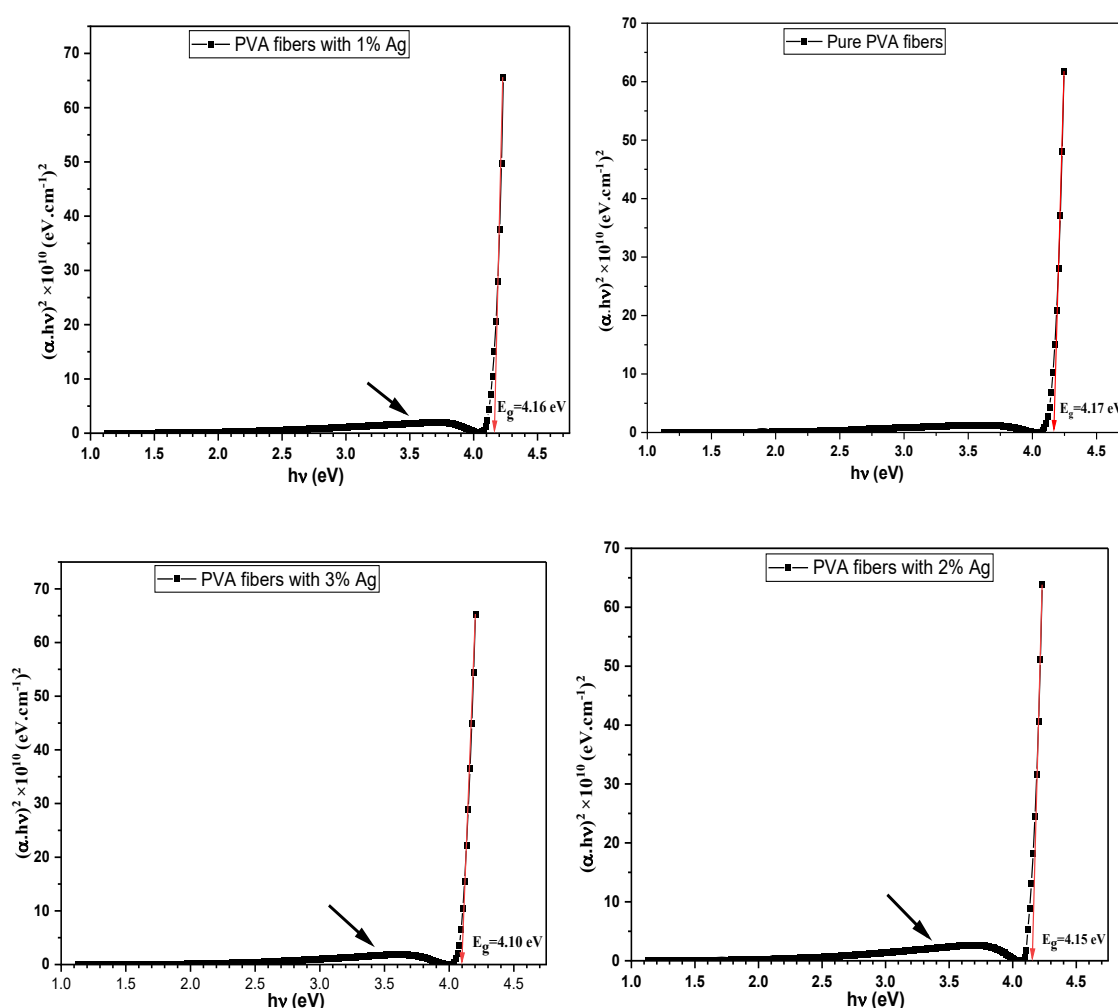


Fig. 5. Optical band gap estimated from Tauc plots for pure PVA and PVA/Ag Nano fibers containing 1, 2, and 3 wt.% Ag.

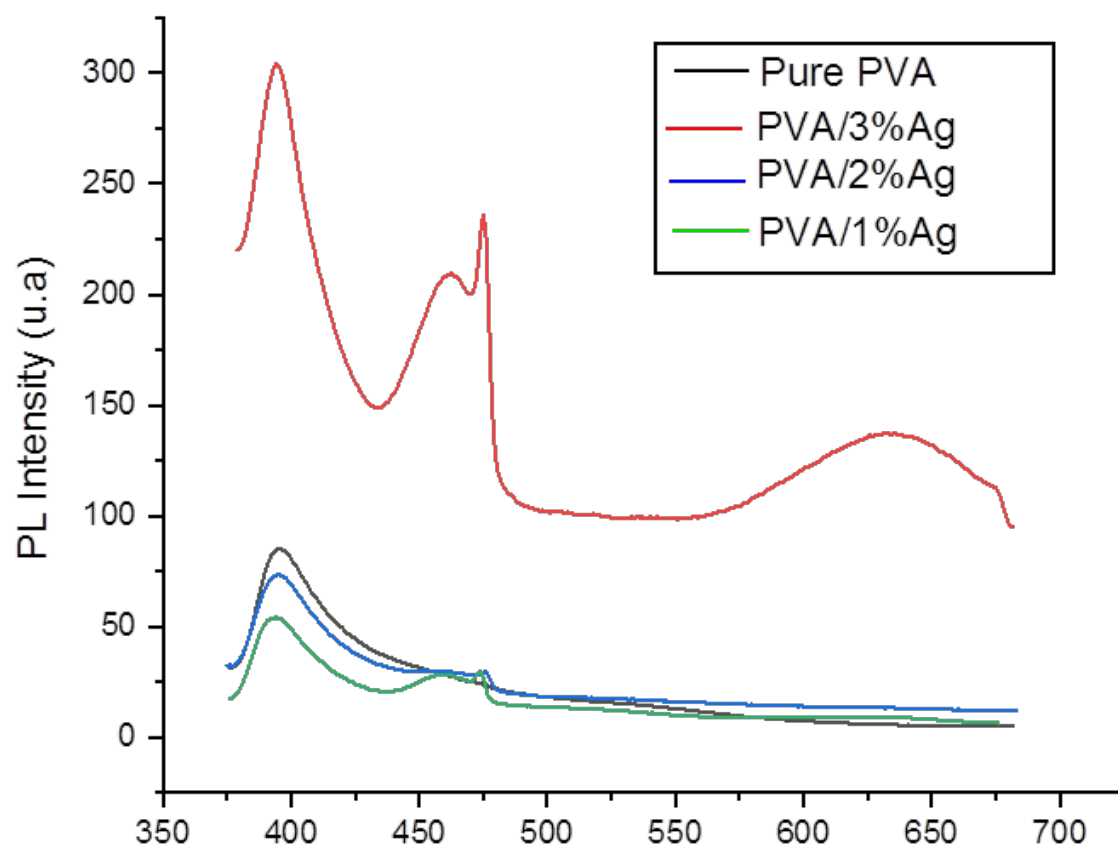


Fig. 6. The fluorescence spectra for PVA doped with Ag electro spun Nano fibers.

Nano fiber matrix [24-26].

CONCLUSION

The result described demonstrates an Ag-concentration-engineered PVA Nano fiber system, in which high levels of structural ordering, low fiber diameter, high optical absorption, low band gap and PL enhancement occur in one material platform. The electro spinning technique was used to produce Nano fibers using pure polyvinyl alcohol (PVA) that were infused with different concentrations of silver (1%, 2%, and 3% wt). High-quality PVA/Ag fibers were obtained as confirmed by XRD, and homogeneous structures and smooth surfaces were shown using field emission scanning electron microscopy (FESEM) measurements. It was observed that the fiber diameter diminished as the silver concentration increased. Moreover, the absorption spectra rose with increased content of Ag and the optical energy gap decreased from

4.17 eV to 4.10 eV at a 3% concentration of Ag. Thus, the material offers great promise for many applications including sensors.

CONFLICT OF INTEREST

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

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