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A study on quantum-sized Ag_2O nanostructures: the effect of chemical interaction time before deposition using the CBD method

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HIGHLIGHTS

- Smooth Ag_2O nanostructures were prepared using the CBD method.
- Interaction time affects optical band gap and particle size.
- The optimal band gap achieved was about 2.25 eV.
- Electrical conductivity varies inversely with interaction time initially.
- SEM and AFM analyses confirm uniform film characteristics.

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ABSTRACT

Ag_2O quantum dots highly versatile promising a wide range of applications from energy conversion to biomedicine making them an important area of research and development in nanotechnology. The tunibilty of the bandgab, make them highly suitable for optoelectronics device, LED, solar cell, were the band gab can be adjested by controlling the size of nanoparticles leading to different light absorbtion and emission properties. In this work, uniform and smooth quantum-sized silver oxide (Ag_2O) nano-films were chemically prepared on a glass substrate using chemical bath deposition method. The study investigated the impact of interaction time before the deposition process, where the effect of three priod of time (0.5, 1 and 1.5) hrs on physical properties have been invistigated. Structural analysis confirmed the cubic silver oxide (Ag_2O) structure, corresponding to the primary diffraction plane (002). The optical results discuss the time dependency of the optical band gap, revealing an energy band gap of approximately 2.25 eV. The electrical conductivity initially shows an inverse relationship with time but increases after a certain point. Additional investigations were conducted on other properties, including electrical characteristics, Atomic Force Microscopy (AFM), and Scanning Electron Microscopy (SEM).

1. Introduction

Nano silver oxide can exist in various phases, including Ag_2O, Ag_3O_4, AgO, and Ag_2O_3 [1-4]. It is a prominent p-type semiconductor known for its high conductivity [5-7]. Among these phases, AgO and Ag_2O are considered the most attractive and commonly formed [8,9]. Researchers have shown significant interest in silver oxide due to its important applications in optoelectronic devices, gas sensors, antibacterial coatings, and photovoltaic cells [10-12].

In previous studies, some preparation methods used to deposit the silver oxide include vapor-liquid-solid procedure, RF magnetron sputtering, pulsed laser deposition, spray pyrolysis method, and chemical bath deposition [13–18]. The preparation method significantly influences the physical properties of Ag_2O, especially the energy band gap value [19–24]. Nathaniel et al [25] and other researchers utilized chemical bath preparation to create silver oxide nano-films on glass substrates, using triethanolamine as a complexing agent and $AgNO_3$ as the precursor at various deposition periods. This is one of several initiatives that have been carried out tocreate Ag_2O nanomaterial [25]. The values of optical constants like the energy gap and index of refraction increase over time. Tuama and colleagues [26] produced Ag_2O films at different triethanolamine (TEA)

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concentrations via chemical bath deposition, and their results showed a clear association between the two. In their investigation of the effects of deposition temperature. Li and Gao [27] employed Chemical Bath Deposition (CBD) to demonstrate how temperature affects the microstructure and nanostructure of silver oxide films. They found that while film porosity increased, grain size decreased with rising temperature. Further, previous studies have given remarkable attention to the impact of chemical deposition time. In particular, the influence of chemical interaction time before depositing Ag_2O thin films on a substrate using the chemical bath deposition approach has garnered significant interest.

2. Experimental part

Ag_2O films were applied on glass substrates using CBD processes. Glass slides (20 mm ×15 mm) in size were cleaned with deionized water and alcohol (methanol) before being air-dried. A transparent solution was created by stirring 0.2 g of silver nitrate ($AgNO_3$), 10 ml of water (distilled), and 2.5 ml of the TEA (Triethanolamine) ($C_6H_{15}NO_3$) at a constant applied temperature of 50 ℃ [28-33]. Three different interaction periods (0.5, 1, and 1.5 hours) were applied before vertically inserting the selected substrate into the prepared solution. The glass slide was left in the mixture for an additional hour to allow for deposition.The structural properties of the deposited nano-films were studied using X-ray diffraction (XRD) with a Cu-Kα X-ray source manufactured by Shimadzu (Model 6000) after the films had been deposited. Utilizing a twin-beam Shimadzu spectrophotometer, the optical characteristics were also investigated. Atomic Force Microscopy (AFM) by SPM AA3000 from "Angsrom Advancedlne", USA, equipment and Scanning Electron Microscope (SEM) using the SEM of the AA-3000 type were used to examine the peculiarities of the morphological discoveries.

3. Results and discussion

Figure 1 shows the effects of chemical contact times (0.5, 1, and 1.5 hours) in the X-ray diffraction (XRD) results. The diffraction peaks are observed at the (200) and (211) planes, corresponding to 2θ values of 38.6° and 45.1°, which are associated with the crystals of Ag_2O and match the standard (00-001-1041). However, the Ag_2O oxide predominantly appears at the (200) diffraction plane. At a contact time of one hour, a significant improvement in the crystalline composition of the film was noticed. These findings are consistent with those reported in earlier studies [34–36].

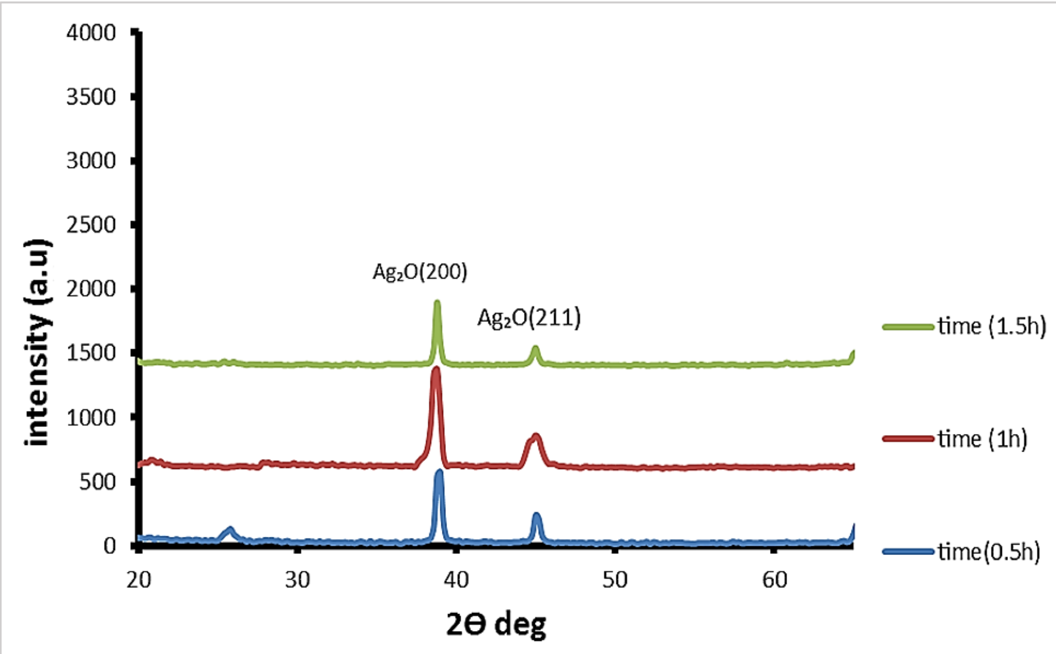


Figure 1: Shows the Ag_2O film's XRD data as a function of contact time (0.5, 1, and 1.5 hours) and constant concentration ratio

The crystal structure parameters, including the values of the structural constants for the deposited and prepared nano-films, were estimated from the X-ray diffraction data using the specified formulae [37-39] as follows:

$$D = \frac{K\lambda}{\beta(\cos\theta)} \quad (1)$$

$$\delta = \frac{1}{D^2} \quad (2)$$

$$\varepsilon = \frac{\beta}{4\tan\theta} \quad (3)$$

where the D: are the grain size in (nm), β: are the FWHM) (radius), *Θ*: re the angle of Bragg, *K*: are the factor shape (constant), ε: are the strain, and δ: are the dislocation density. The calculated values of the structural constants are presented in Table 1, and it was found that the average grain sizes remained constant. As shown in the results, the values of the structural strain and dislocation densities gradually decreased over time. The following formulas [40-44] were used to analyze the X-ray diffraction (XRD) constants and characterize the properties of the prepared films:

$$D = \frac{K\lambda}{\beta(\cos\Theta)} \quad (4)$$

$$\delta = \frac{1}{D^2} \quad (5)$$

$$\varepsilon = \frac{\beta}{4\tan\theta} \quad (6)$$

The values of the intensities for the full width at the half of maximum peaks (FWHM) in radius values refer to β. The angle (2Θ) value refers to the "angle of Bragg." The dislocation density values are δ, the strain areε, and the X-ray wavelength radiation are presented of λ. The "dislocation density" values are δ, the strain values are ε, and the D refers to the crystallite size values. All the estimated values of the structural contents are tabulated and given in Table 1 for the Nano films of silver oxide that were deposited. It was discovered that these values were virtually the same. As presented in the results, the analyzed values of the structure constants vary over time with only a little variation.

Table 1: Structural characteristics of samples at different contact times

Interaction time (h)	2Θ (deg)	Inter planer spacing (A)	hkl	FWHM β Deg.	D (nm)	(δ) *10^{-3} lines/m^2	ε*10^{-3}
0.5	38.8	2.320	200	0.578	14.57	4.70	2.3
1	38.6	2.33	200	0.584	14.42	4.8	2.4
1.5	38.8	2.326	200	0.542	14.51	4.74	2.3

Figure 2 (a-c) show the atomic force microscopy (AFM) images of the prepared Ag_2O films at various contact times. The films exhibit a uniform structure comprising large, island-like particles on the selected substrates. This uniform structure results from an increase in surface mobility caused by the extended contact time of the atoms, which leads to their adsorption onto the surface of the chosen substrate. It was found that increasing the contact duration significantly improves the surface morphology, with a noticeable change in the particle size of the manufactured material. This trend may be attributed to the higher likelihood of particle aggregation and the extended duration of crystal growth. The average particle size, falling within the quantum dot range, confirms the presence of quantum size effects on the film's surface. The results are presented in Table 2 below. Similar results were reported in some previous studies [34, 45 and 46].

Table 2: Data from atomic force microscopy (AFM) investigations of Ag_2O nano-films at different contact periods

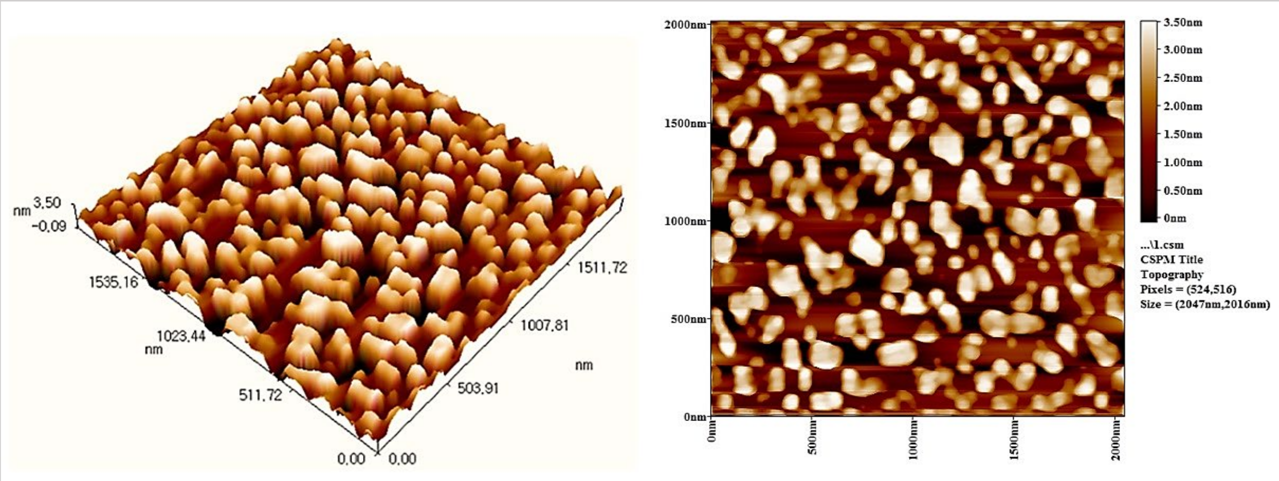
Time of Reaction	Average Roughness (nm)	RMS (nm)
30 min	0.886	1.31
60 min	1.462	1.73
90 min	0.833	0.992

Figure 3 (a-c) displays the FE-SEM images of the Ag_2O samples at different interaction times—0.5, 1, and 1.5 hours. The films are free from pinholes and cracks, providing smooth, uniform, and complete substrate coverage. The aggregation of small particles forming a porous structure suggests that the Ag_2O thin films are microcrystalline, consistent with previous research [30]. Figure 4 shows the transmission of the generated nano-films as a function of the chemical reaction duration. The nano-film deposited with a one-hour contact period exhibited higher transmission than the other conditions. According to earlier studies, Ag_2O is a promising TCO (Transparent Conductive Oxide) material due to its low thickness and precise synthesis, which may contribute to the efficient transmission of the film.

Figure 5 shows the absorption coefficient (α) of Ag_2O thin films as a function of wavelength, ranging from 400 nm to 1100 nm, at different contact times (0.5 hours, 1 hour, and 1.5 hours). The energy band gaps of the deposited nanostructured films were calculated based on the Tauc equation [47,48].

$$a = \frac{A(hv - Eg)}{hv} \quad (7)$$

where Eg is the optical energy gap, ν is the frequency of the photons, h is the constant of Planck's, and n depends on the kind of transition: n=1/2 for direct optical band gaps, or n=2 for the indirect band gaps [49]. The results presented in Figure 5 show that the absorption coefficient values increased after 1.5 hours of contact, likely due to the increased density of the peaks. This scenario leads to a decrease in the absorption coefficient. The highest transmission observed at this condition corresponds to the lowest absorption coefficient after 1 hour of contact time, which can also be attributed to an improved crystallite structure [50–52].



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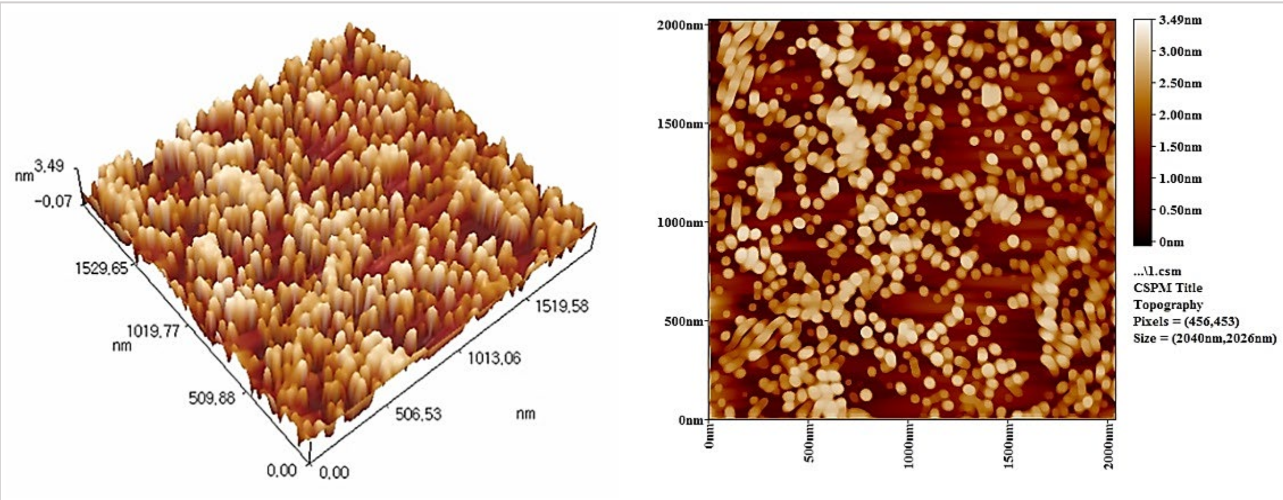

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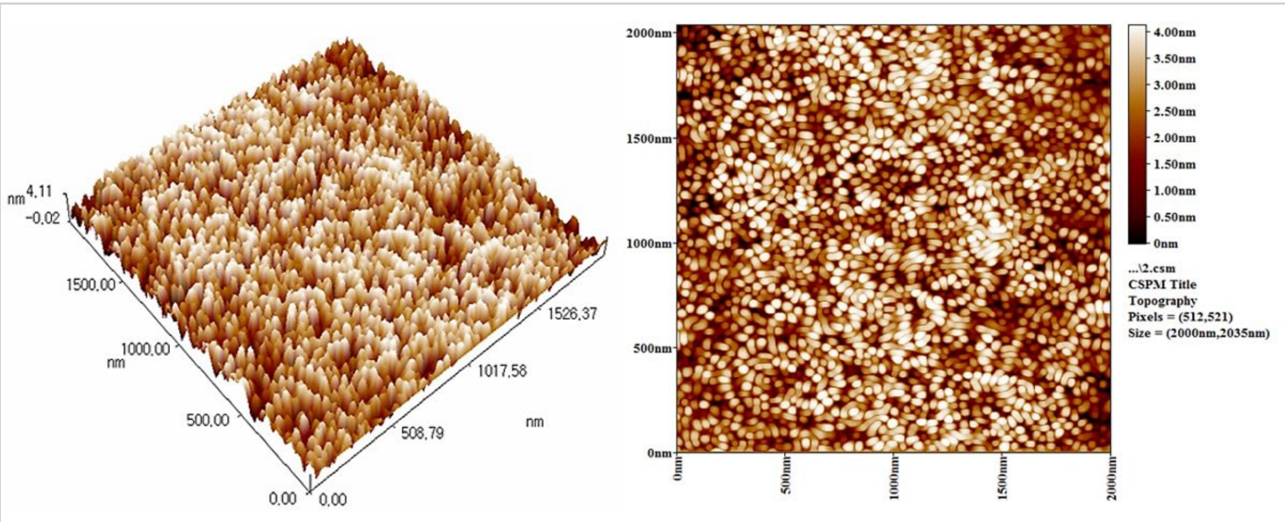

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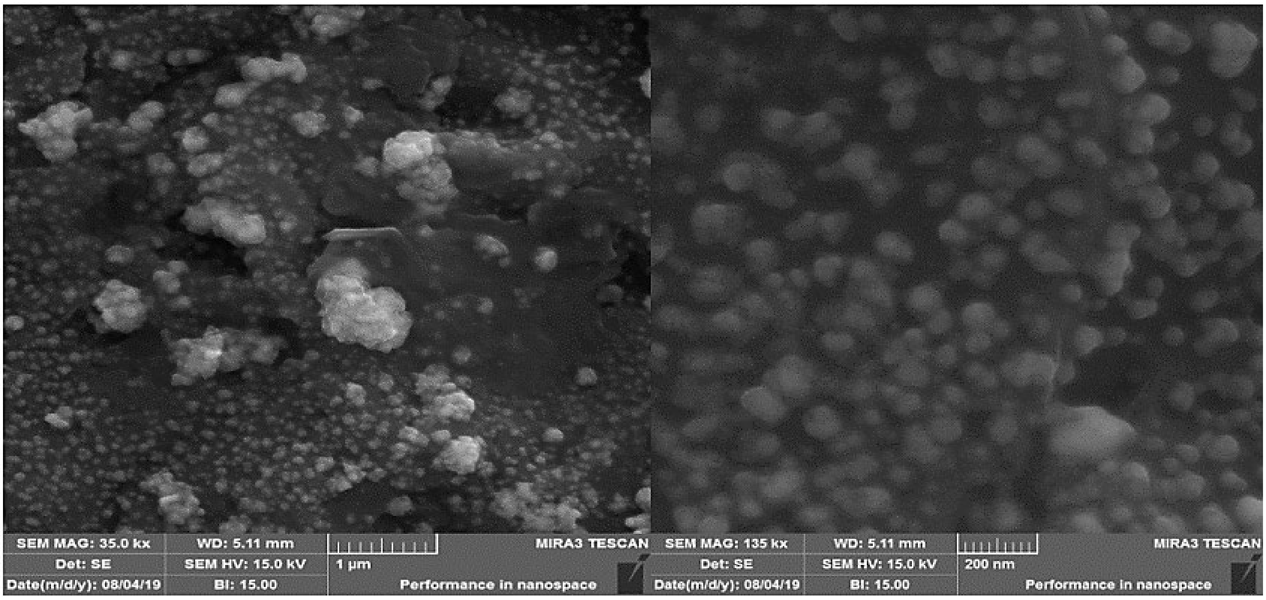
Figure 2: (a-c) 1D and 3D AFM results of Ag_2O films prepared at 0.5, 1, and 1.5 hours, respectively



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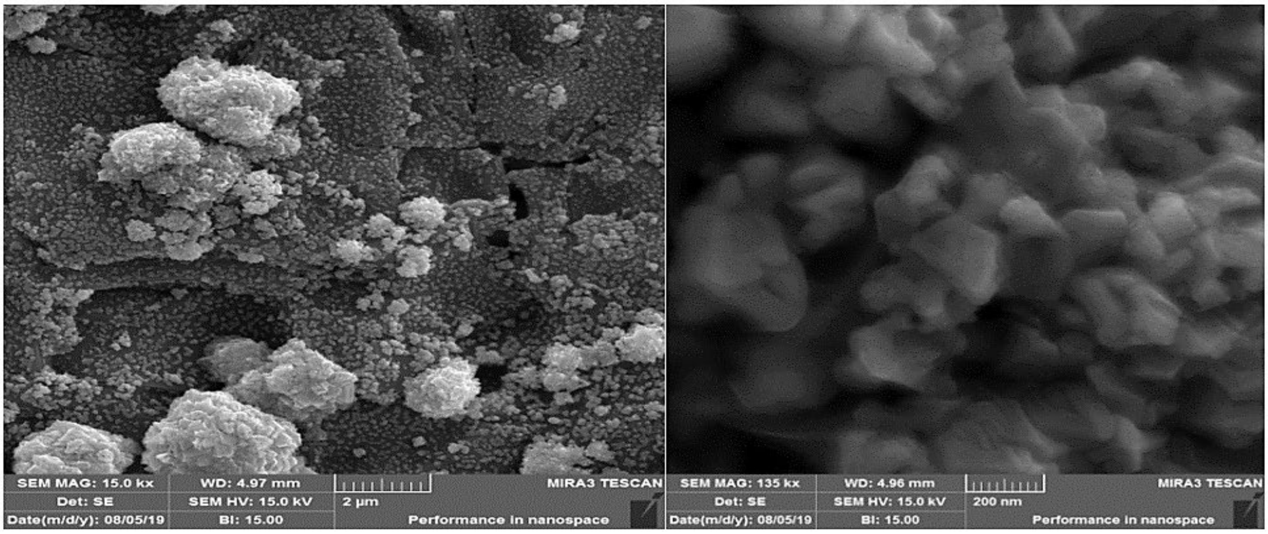

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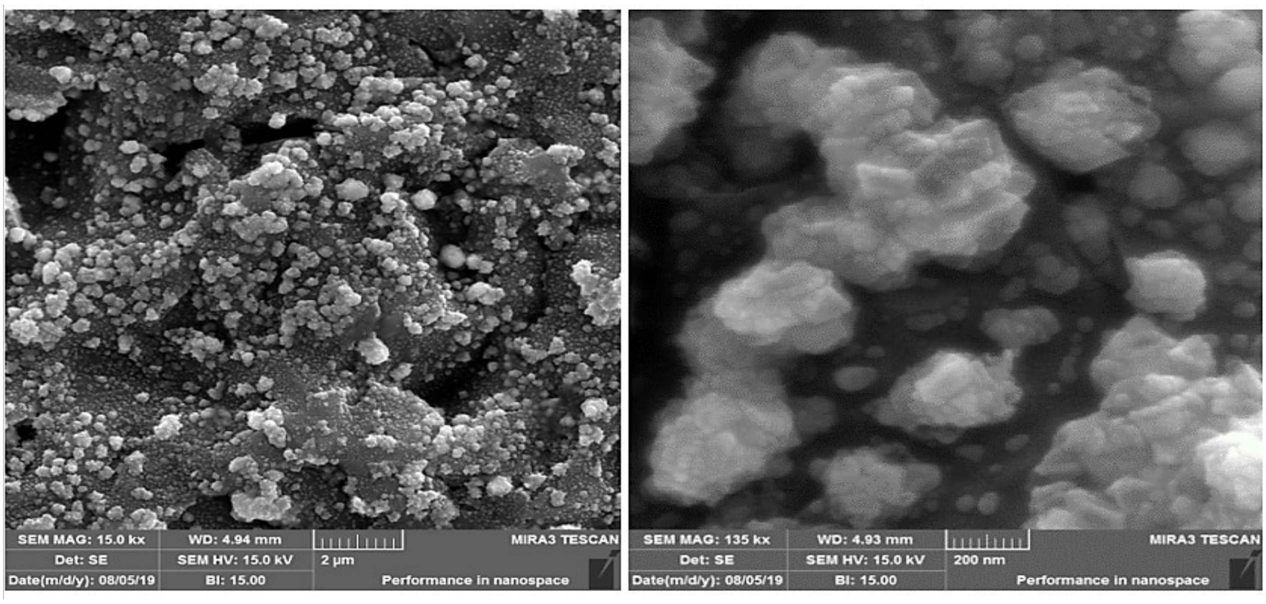

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Figure 3: (a-c): FE-SEM results of Ag_2O as a function of chemical contact time (0.5-1.5 hours)

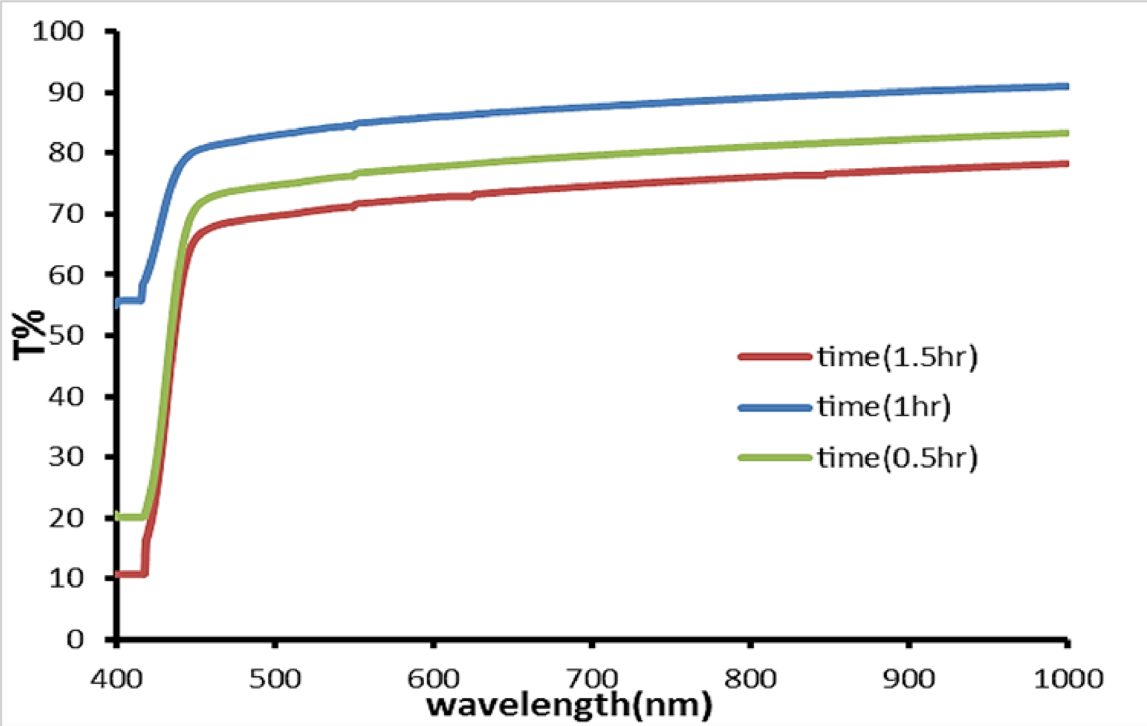


Figure 4: Optical transmission of Ag_2O films plotted against wavelength at different contact periods

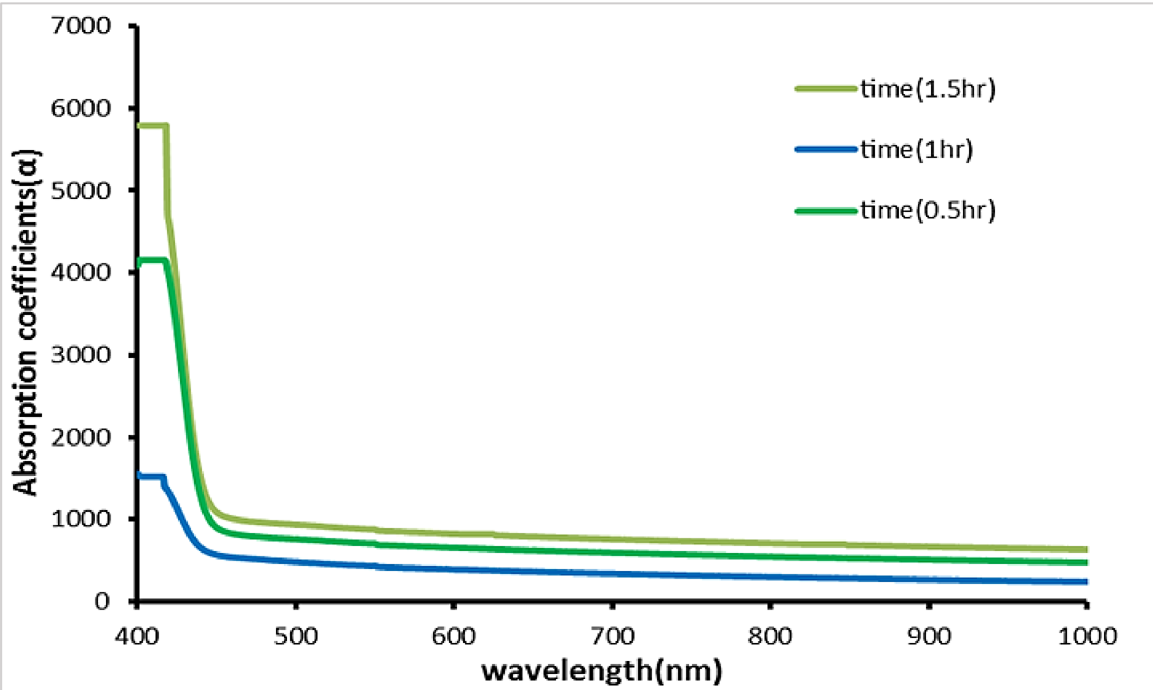
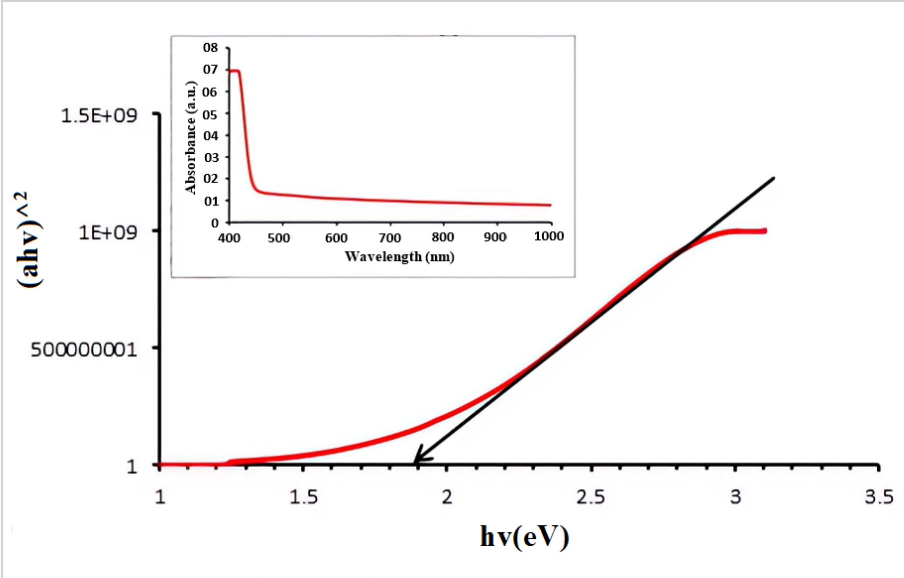


Figure 5: Optical absorption coefficient of Ag_2O as a function of chemical interaction time

Figure 6 (a-b-c) displays the energy band gap values for the deposited Ag_2O nano-films at various times during contact. When the period fluctuates from 0.5 to 1.5 hours, the predicted band gap ranges between 1.8 and 2.295. Based on earlier X-ray diffraction studies, the fluctuation in the optical energy gap is attributed to improvements in the crystalline structure quality of the films [34, 53-54].

The extinction coefficient k as a function of the optical characteristics could be shown in Figure 7 where the value of K has been estimated from $k = \frac{\alpha\lambda}{4\pi}$ where λ is the optical wavelength.

It is possible to display the extinction coefficient of silver oxide nano-films as a function of contact time. Figure 7 shows an increase in the extinction coefficient, which is attributed to the absorption characteristics of the films. Figure 8 (a-c) illustrates the variation in electrical resistivity as a function of chemical reaction time. A clear decrease in resistivity is observed up to 1.5 hours, after which the resistivity increases. This decrease is related to the increase in carrier mobility and concentration.



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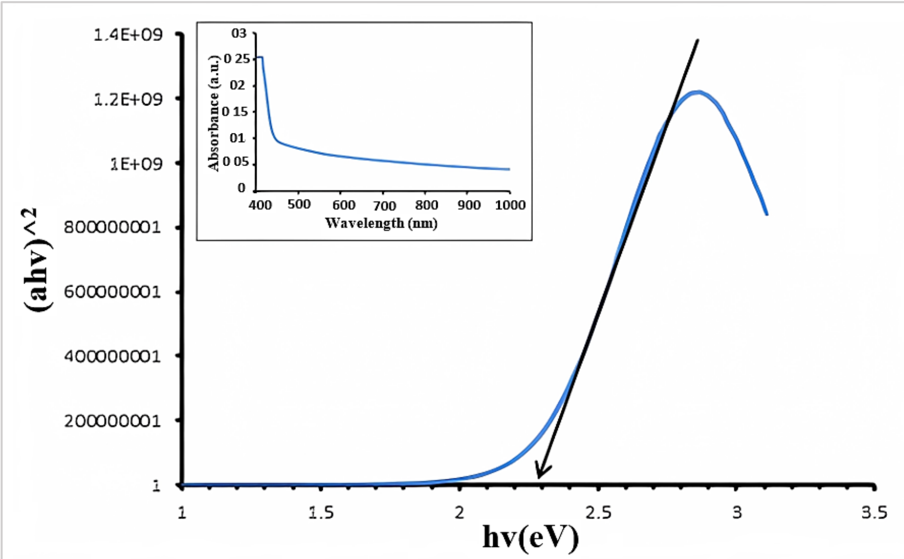

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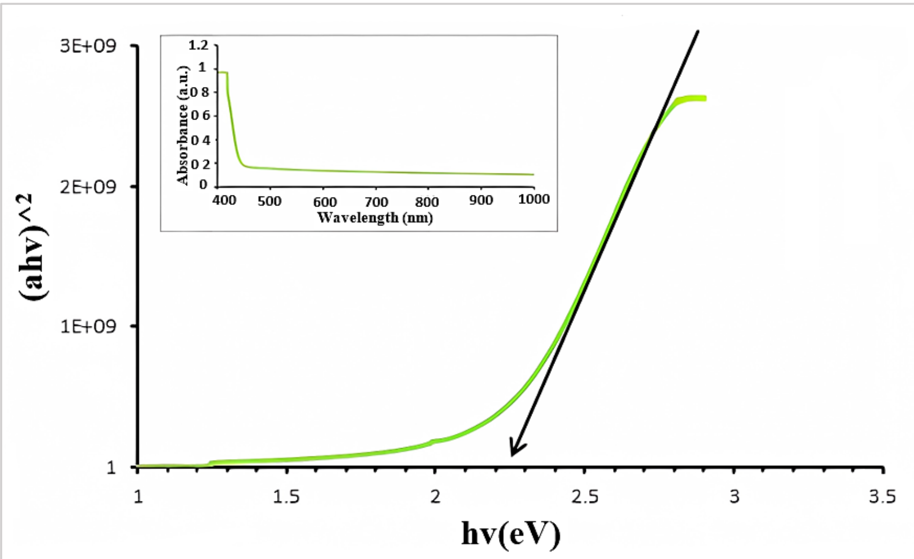

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Figure 6: (a-b-c) The energy band with a time of contact of (0.5-1.5) hrs

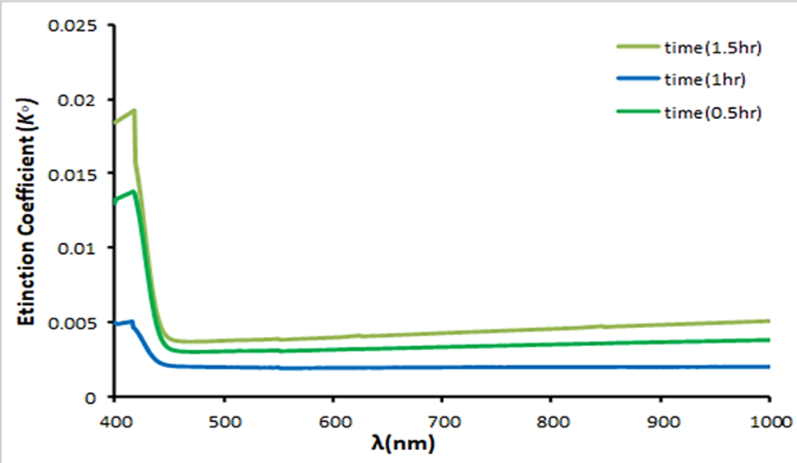
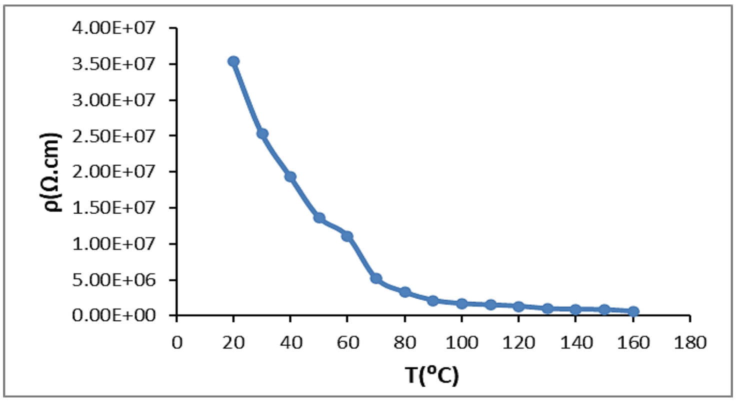


Figure 7: Extinction coefficient of Ag_2O as a function of chemical interaction time



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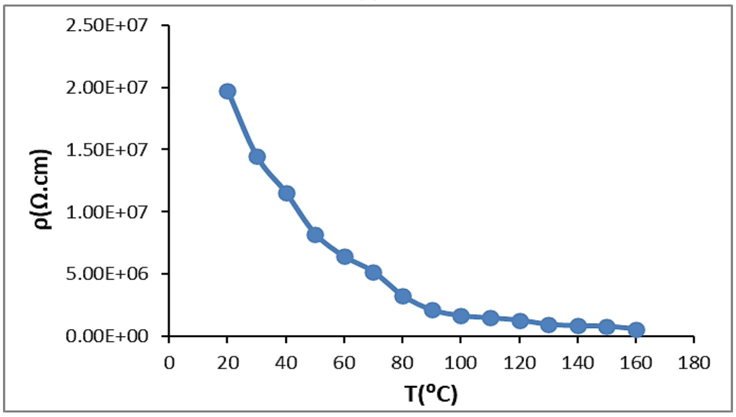

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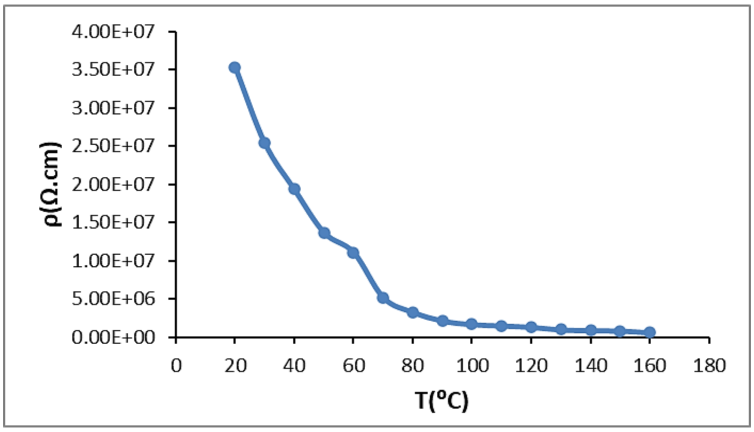

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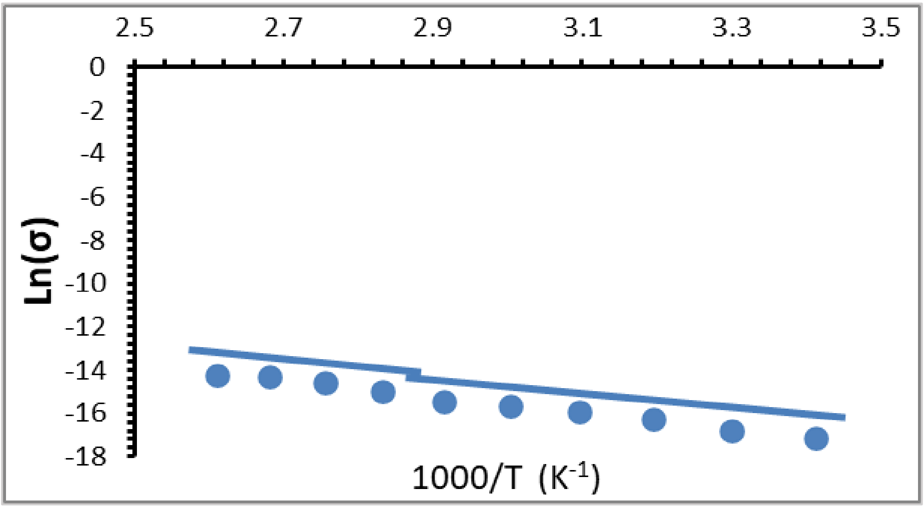
Figure 8: (a-c) Resistivity vs. temperature under different conditions

The activation energy can be determined from the slope of the linear line in the (Ln (σ) vs. (1000/T) plot, as shown in Figure 9 (a-c). The activation energy values decreased from 0.32 to 0.29 over time, as tabulated in Table 3. This decrease is attributed to the increase in film thickness. Additionally, a slight decrease in activation energy is observed due to changes in hole concentration.



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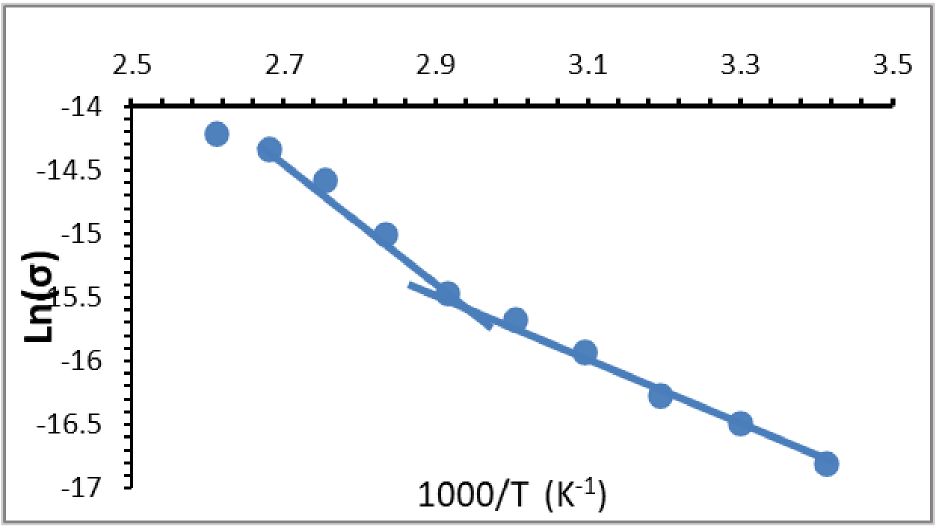

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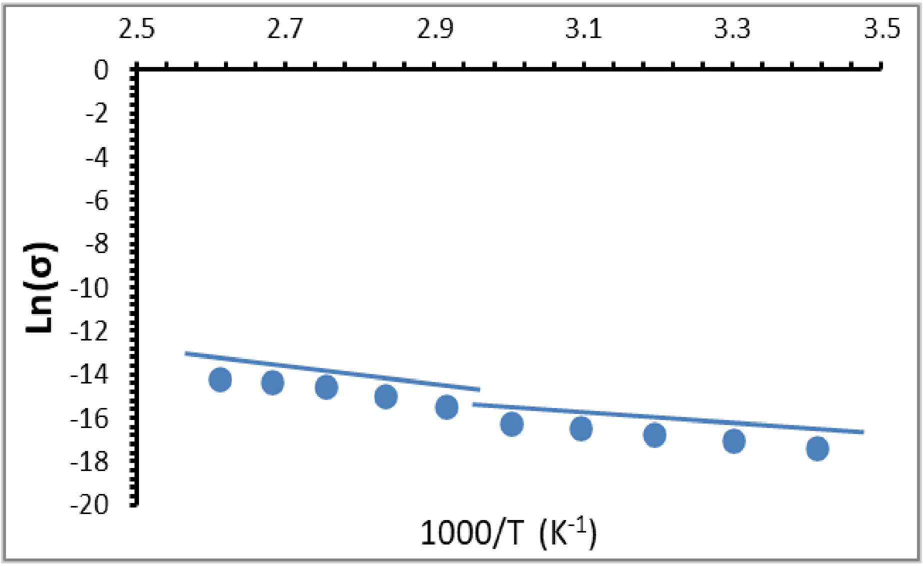

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Figure 9: (a-c) Electrical conductivity vs. temperature for determining activation energy

Table 3: Activation energies (Ea) for nano silver oxide films

Interaction time (hr)	Activation energies Ea (ev)
0.5	0.324
1	0.291
1.5	0.372

Table 4 displays the Hall coefficient under different conditions. The positive Hall coefficient for all samples confirms the p-type conductivity of the material. The Figure of Merit (FOM) was used to determine the optimal conditions for achieving high electrical conductivity and optical transmission.

Table 4: Electrical properties and constants

Interaction time (hr)	Hall coefficient (m^2/C)	Mobility (cm^2/Vs)	Resistivity (Ω.cm)	Conductivity (Ω.cm)-1	Bulk concentrate (Nb) (cm^3)
0.5	4.652*105	1.621*10 1	2.869*104	3.485*10-5	1.342*1013
1	4.962*105	2.652*10 2	1.871*104	5.345*10-5	1.258*1012
1.5	5.632*105	1.621*10 1	6.247*104	1.6*105	1.4*1013

4. Conclusion

In this work, uniform and smooth quantum-sized silver oxide (Ag_2O) nano-films were chemically prepared on a glass substrate using chemical bath deposition method. The study investigated the impact of interaction time before the deposition process, where the effect of three priod of time on physical properties have been investigated. From the experimental results it was conclude the following:

1) A smooth and homogeneous Ag_2O nanostructure thin film was formed using the chemical bath deposition method after one hour of contact.
2) As the contact period increases, a blue shift in the optical energy band gap and a reduction in particle size for the deposited nano-films are observed.
3) The tunibiltyin the bandgab value could be obtained sucssfuly by varying particle size.

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Author contributions

Conceptualization, E. Salim and M. Fakhri; data curation, M. Awayiz, E. Salim, and M. Fakhri.; formal analysis, M. Awayiz, E. Salim, and M. Fakhri.; investigation, M. Awayiz, E. Salim, and M. Fakhri.; methodology, M. Awayiz, E. Salim, M. Fakhri, M. Qaeed, and S. Gopinath.; project administration, E. Salim, and M. Fakhri, resources, M. Awayiz, E. Salim, and M. Fakhri.; software, M. Awayiz, E. Salim, M. Fakhri , M. Qaeed, and S. Gopinath.; supervision, E. Salim.; validation, E. Salim, M. Fakhri.; visualization, M. Awayiz, E. Salim, and M. Fakhri.; writing—original draft preparation, M. Awayiz.; writing—review and editing, E. Salim, M. Fakhri, M. Qaeed, and S. Gopinath All authors have read and agreed to the published version of the manuscript.

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Data availability statement

The data that support the findings of this study are available on request from the corresponding author.

Conflicts of interest

The authors declare that there is no conflict of interest.

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