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Effect of pH on the Optical and Electrical Properties of PbAgS Ternary Thin Films Deposited by Chemical Bath Deposition

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Abstract:
This work presents the successful deposition of Lead Silver Sulphide (PbAgS) by simple chemical bath method under varying pH conditions. Varying pH was achieved using different volume concentration of ammonium hydroxide (NH_4OH) solution. The final bath solution for the deposition of the films contained aqueous solutions of $Pb(NO_3)_2$, $AgNO_3$, thiourea, TEA and EDTA. $Pb(NO_3)_2$, $AgNO_3$ and thiourea served as precursors for Pb^{2+}, Ag^+, and S^{2-} respectively while EDTA and TEA were used as complexing agents. NH_4OH served as pH adjuster. The deposited film properties were characterized for optical and electrical properties using a Janway UV – VIS spectrophotometer and 4 Point Probe (Keithley Four Point Probe: Model 67005). From the spectral analysis of absorbance, other optical properties such as transmittance, reflectance, refractive index, and extinction coefficient and band gap energy were obtained. The films show high absorbance in the UV region and high transmittance in the VIS – NIR regions, while reflectance is generally low. The films showed direct band gap energy range of 1.89 eV – 2.30 eV. The result showed that band gap decreased as pH increases. Film thickness increased from 493.83 nm to 945.52 nm as pH increases. Electrical properties of the films showed that the deposited PbAgS thin films are semiconducting films with electrical conductivity within the range of 1.337×10^{-3} (*s/cm*) and 9.334×10^{-3} (*s/cm*). Electrical conductivity of the films were found to increase as pH increases from 10.30 to 11.20.

Keywords:
Optical Properties, Band Gap, CBD, Electrical Properties

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1. Introduction

The field of material science and technology over the years has experienced indebt research on thin film materials for various application purposes. Thin film materials are described based on the thickness layer formed during the deposition processes. A thin film is a crystalline or non – crystalline thin layer of material (molecular or element) with thickness ranging from a fraction of nanometer up to few microns formed on the surface of conducting or non – conducting substrate either by physical or chemical method [1,2]. Thin film can be binary, ternary, and quaternary, depending on the number of elements that make up the film. Ternary thin films are thin films that contain three different elements [3,4]. Many deposition methods such as chemical bath method, electrodeposition, successive ionic layer adsorption reaction (SILAR), spray pyrolysis, screen printing and many others have been employed for deposition of ternary thin films. Among all these methods mentioned, chemical bath method is one of the best method.

Chemical bath deposition technique is currently attracting a great deal of attention as the technique is relatively cost effective, has minimum material wastage, does not need sophisticated instrument and can be applied in large area deposition at low temperature. It is well studied and produces films that have comparable structural and opto-electronic properties to those produced using other sophisticated thin film deposition technique [5].

Previously, PbAgS thin films have been deposited by [6,7]. The work of [6] showed tenability of the optical properties of PbAgS thin film when the deposition time was varied. They obtained direct energy band gap range of 2.20 eV - 2.40 eV, refractive index range of 2.1 - 2.60, while the thickness of the films increased with deposition time. The work of [7] showed that PbAgS thin films have extinction coefficient that ranged from 0.010 to 0.140. The direct and indirect band gaps ranged from 1.5eV to 2.1eV and 0.3eV to 0.8eV respectively. The real and imaginary parts of the dielectric constant ranged from 0.4 to 5.2 and 0.010 to 0.390 respectively. They suggested that the grown thin films of PbAgS could be useful in electronics industry such as camera lens and eye glass coating and in architectural industry such as in poultry house and coating of windows and doors for passive heating and cooling of house. In this work, we deposited lead silver sulphide (PbAgS) thin film by chemical bath method. Effect of pH on the optical and electrical properties of the deposited films were studied.

2. Materials and Methods

For the deposition of lead silver sulphide thin films unto a non – conducting microscopic glass of dimension (75 mm x 25 mm x 1 mm), reagents that are soluble in water were considered because the solvent chosen for the deposition is water. Double distilled water was used as solvent while the reagents that served as precursors for lead (Pb^{2+}), silver (Ag^{+}) and sulphur (S^{2-}) were lead (II) nitrate ($Pb(NO_3)_2$), silver nitrate ($AgNO_3$), thiourea (CH_4N_2S). Triethanolamine (TEA) and Ethylenediaminetetraacetic acid (EDTA) were used as complexing agents. Prior to the deposition, the substrates were degreased by dipping them in concentrated HNO_3 for 42 hours, after which they were brought out, washed with detergent, rinsed with distilled water and allowed to dry in an open air. The degreased and cleaned surface has the advantage of providing nucleation centre for the growth of the films, hence yielding highly adhesive and uniformly deposited films [6,8,9].

During deposition of PbAgS thin films at varying pH values using ammonium hydroxide, constant dip time of 42 hours at room temperature of about 300 K was observed. The first bath solution for the deposition of PbAgS contained 5 mL of 1 M of lead (II) nitrate solution, 5 mL of 0.1 M of silver nitrate solution, 10 mL of 1 M of

Thiourea, 5 mL of 0.1 M of EDTA, 5 mL of 7.4 M of TEA. These solutions were added at intervals of 3 minutes after stirring using magnetic stirrer. Finally, 2 mL of 14 M of ammonium solution was added then stirred for 5 minutes. 20 mL of double distilled water was added to the final solution and stirred for 5 minutes to form a homogenous solution. Degreased substrate was inserted vertically into the final solution and was allowed to stay for 42 hour. At the end of the stipulated time, the substrate was removed, rinsed with distilled water and dried in an open air for 5 hours. Other three samples were synthesized with the same procedure but with variation in the volume of ammonium solutions of 3 mls, 4 mls and 6 mls respectively. The idea was to determine the effect of pH on deposition of PbAgS thin films. The pH values of the baths were measured with a pH meter and recorded as shown in Table 1. Table 1 shows the bath constituents for the deposition of PbAgS for varying volume of ammonium solution. The equations for the reaction has been given in previous works by [6,7].

Table 1. *Variation of Volume of Ammonium Solution (pH variation) for the Deposition of PbAgS.*

	$Pb(NO_3)_2$ (ml)	$AgNO_3$ (ml)	EDTA (ml)	TEA (ml)	$(NH_4)OH$ (ml)	Thiourea (ml)
pH: 10.30	5.00	5.00	5.00	5.00	2.00	10.00
pH: 10.50	5.00	5.00	5.00	5.00	3.00	10.00
pH: 10.80	5.00	5.00	5.00	5.00	4.00	10.00
pH: 11.20	5.00	5.00	5.00	5.00	6.00	10.00

2.1. Characterization and Computation of Optical and Electrical Properties

Optical characterization of the films was carried out using Janway 6405 UV / VIS spectrophotometer at wavelength interval of 300 nm to 1100 nm. Absorbance values of the films were obtained using the spectrophotometer and other optical properties such as transmittance, reflectance, refractive index, extinction coefficient, absorption coefficient, energy band gap and optical conductivity were evaluated using the following equations as obtained from literatures.

Transmittance of the film was evaluated using equation (1) given by [10,11]

$$T = 10^{-A} \quad (1)$$

Reflectance was obtained using the expression in equation (2) as given by [12, 13]

$$R = 1 - (A + T) \quad (2)$$

Refractive index of the films was calculated using equation (3) as given by [14, 15]

$$\eta = \frac{(1+\sqrt{R})}{(1-\sqrt{R})} \quad (3)$$

The absorption coefficient (α) was calculated from the transmittance values using the equation (4) as given by [16,17,18]

$$\alpha = \frac{1}{t}\text{In}\left(\frac{1}{T}\right) \quad (4)$$

Were (t) is the thickness of the deposited thin films. Extinction coefficient was obtained using equation (5) [19,20,21,22]

$$k = \frac{\alpha\lambda}{4\pi} \quad (5)$$

Optical conductivity was estimated using equation (6) [23,24,14]

$$\sigma_o = \frac{\alpha\eta c}{4\pi} \quad (6)$$

Where c is the speed of light.

The energy band gap was estimated using Tauc's model given in equation (7) as [25,19].

$$(\alpha hv)^n = \beta(hv - E_g) \quad (7)$$

Where β is a constant, n = 2 for direct band gap. The energy band gaps of the films were obtained by extrapolating the straight portion of the plot of $(\alpha hv)^2$ against the photon energy (hv) at $(\alpha hv)^2 = 0$.

The sheet resistivity was calculated according to equation (8) as given by [26,27,28]. That is,

$$\rho = \frac{\pi t}{\ln 2}\left(\frac{V}{I}\right) = 4.523t\left(\frac{V}{I}\right) \quad (8)$$

while the electrical conductivity was evaluated by finding the reciprocal of the sheet resistivity as giving in equation (9).

$$\sigma = \frac{1}{\rho} \quad (9)$$

3. Results and Discussions

3.1. Optical Properties

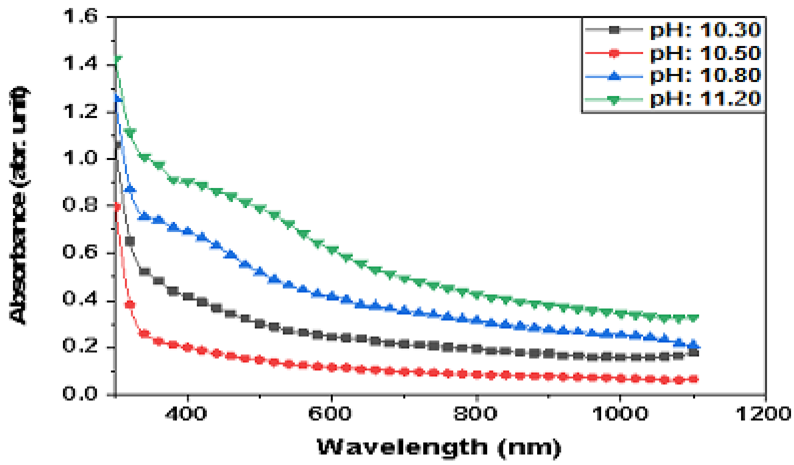

Figure 1. *Plot of Absorbance of PbAgS Thin Films against Wavelength for different pH.*

Absorbance of the films against wavelength is shown in Figure 1. The spectral absorbance of the films varies with wavelength in a similar manner. Film deposited at pH of 10.30 has absorbance values of 1.10 to 0.42 in UV region, 0.42 to 0.22 in VIS region and 0.22 to 0.18 in NIR region. Film deposited at pH of 10.50 has values of 0.80 to 0.2 in UV region, 0.20 to 0.10 in VIS region and decreases slightly to 0.07 in NIR region. Film deposited at pH of 10.80 has values of 1.26 to 0.69 in UV region, 0.69 to 0.36 in VIS region and 0.36 to 0.21 in NIR region. Film deposited at pH of 11.20 has values of 1.43 to 0.90 in UV region, 0.90 to 0.50 in VIS region and 0.50 to 0.33 in NIR region. These results showed that the deposited PbAgS thin film are highly absorbing within UV region and decrease in absorption as wavelength increased was noticed. The result is in line with result obtained by [7]. Figure 2 showed the graph of spectra transmittance of the films plotted against wavelength. The spectral transmittance of these films varies in a similar manner. Film deposited at

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pH value of 10.30 has transmittance values of 9 % to 38 % in UV region, 38 % to 61 % in VIS region and 61 % to 69 % in NIR region. Film deposited at pH value of 10.50 has values of 16 % to 63 % in UV region, 63 % to 80 % in VIS region and 80 % to 85 % in NIR region. Film deposited at pH value of 10.80 has values of 6 % to 20 % in UV region, 20 % to 44 % in VIS region and 44 % to 62 % in NIR region. Film deposited at pH value of 11.20 has values of 4 % to 13 % in UV region, 13 % to 32 % in VIS region and 32 % to 46 % in NIR region. These results showed that bath pH affect the transmittance of PbAgS.

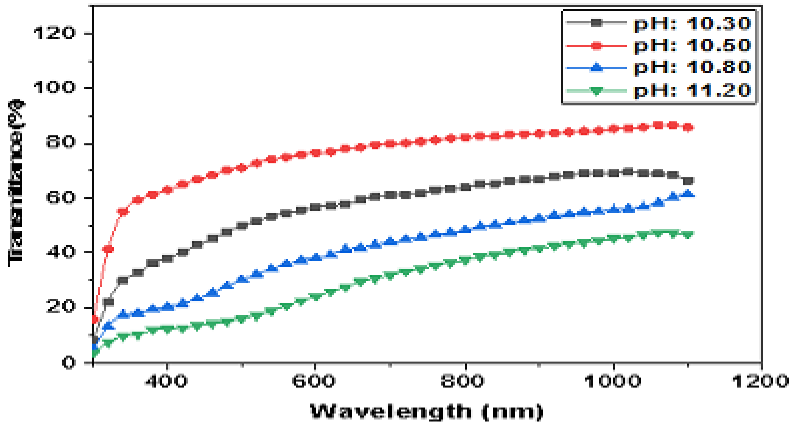

***Figure 2.** Plot of Transmittance of PbAgS Thin Films against Wavelength for different pH.*

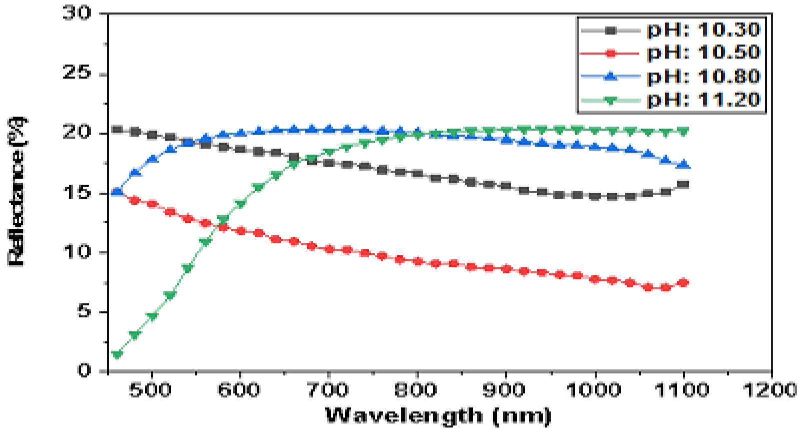

***Figure 3.** Plot of Reflectance of PbAgS Thin Films against Wavelength for different pH.*

Figure 3 showed the plot of spectra reflectance of the films against wavelength. The reflectance of the films was studied at wavelength range of 460 nm to 1100 nm. This is because the films have no reflectance within the wavelength of 300 nm to 450 nm. Films deposited at pH values of 10.30 and 10.50 have similar reflectance pattern that decreased as wavelength increases. Film deposited at pH of 10.30 has reflectance of 20 % at 460 nm to 18 % at 700 nm and further decreased to 16 % within NIR region. Film deposited at pH of 10.50 has reflectance of 15 % to 10 % in VIS region and 10 % to 7 % in NIR region. Films deposited at pH of 10.80 and 11.20 have similar reflectance pattern that increased as wavelength increases within VIS region but decreased in value within NIR region. Film deposited at pH of 10.80 has reflectance of 15 % to 20 % in VIS region and 20 % to slight decrease to 17 % in NIR region. Film deposited at pH of 11.20 has reflectance values that increased from 1.48 % to 18.51 % within VIS region. The value increased slightly to 20.22 % within NIR region. The graphs of refractive index of the films against wavelength is presented in figure 4. Film deposited at pH of 10.30 has refractive index of 2.64 at 460 nm to 2.44

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at 700 nm and decreases further from 2.44 to 2.32 in NIR region. Film deposited at pH of 10.50 has values of 2.27 to 1.95 in VIS region and 1.95 to 1.75 in NIR region. Films deposited at pH value of 10.80 has values of 2.27 to 2.64 in VIS region and 2.64 to 2.43 in NIR region. Film deposited at pH of 11.20 has refractive index values of 1.28 to 2.51 within VIS region which increased slightly to 2.63 at 1100 nm.

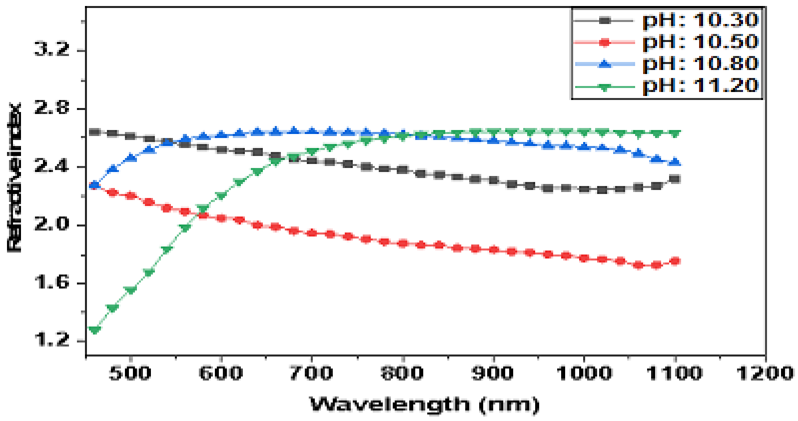

Figure 4. *Plot of Refractive Index of PbAgS Thin Films against Wavelength for different pH.*

Figure 5 showed the plot of extinction coefficient of the films plotted against wavelength. The extinction coefficient of the films decreases as the wavelength increases. They have peak values at 300 nm. Film deposited at pH of 10.30 has extinction coefficient of 8.4×10^{-2} to 3.3×10^{-2} in UV region, 3.3×10^{-2} to 1.7×10^{-2} in VIS region and 1.7×10^{-2} to 1.4×10^{-2} in NIR region. Film deposited at pH of 10.50 has extinction coefficient of 6.3×10^{-2} to 1.6×10^{-2} in UV region, 1.6×10^{-2} to 7.7×10^{-3} in VIS region and 7.7×10^{-3} to 5.3×10^{-2} in NIR region. Film deposited at pH of 10.80 has values of 1.0×10^{-1} to 5.6×10^{-2} in UV region, 5.6×10^{-2} to 2.8×10^{-2} in VIS region and 2.8×10^{-2} to 1.7×10^{-2} in NIR region. Film deposited at pH of 11.20 has values of 1.1×10^{-1} to 7.2×10^{-2} in UV region, 7.2×10^{-2} to 3.9×10^{-2} in VIS region and 3.9×10^{-2} to 2.6×10^{-2} in NIR region.

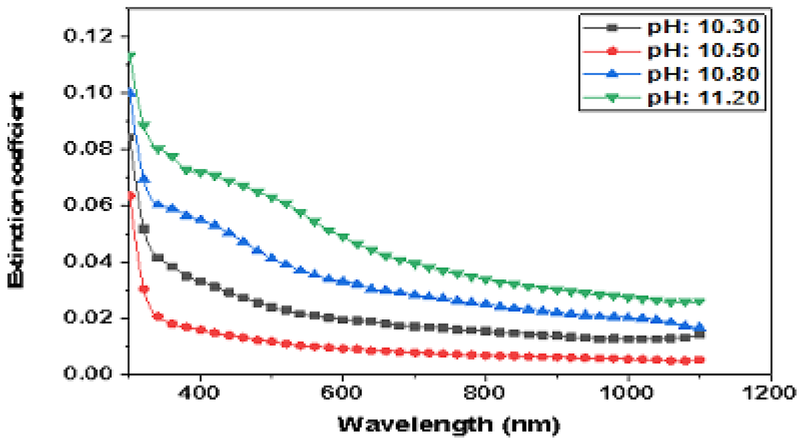

Figure 5. *Plot of Extinction Coefficient of PbAgS Thin Films against Wavelength for different pH.*

The graphs of $(\alpha hv)^2$ plotted against photon energy is shown in figures 6. The optical band gap energy (E_g) of the films were estimated from the plot of $(\alpha hv)^2$ versus photon energy (hv). The straight nature of the plots indicates the existence of direct transition. The direct band gap energies of as – grown films were determined by extrapolating the straight portion of the graph to the photon energy (hv) axis at $(\alpha hv)^2 = 0$. Energy band gap values were found to be 2.30 eV, 2.27 eV, 2.15 eV and

1.89 eV for film deposited at pH values of 10.30, 10.50, 10.80 and 11.20 respectively. These results showed that band gap energy decreased as pH of the reaction bath increases. Figure 7 showed the plot of thickness and energy band gap of the deposited films plotted against pH values of the deposition bath. The result showed that the films are relatively thick with least thickness of 493.83 nm and optimal thickness of 945.52 nm. This increase in thickness as pH increases corresponded to results obtained by [29]. The result showed that thickness of the deposited films increased as pH of the reaction bath increased while estimated energy band gap values decreased with increase in pH from 10.20 to 11.30. This decrease in energy band gap as pH increased is in line with result obtained by [30,31,32]. According to [31], this red shift in band gap energy can be explained on basis of quantum confinement effect.

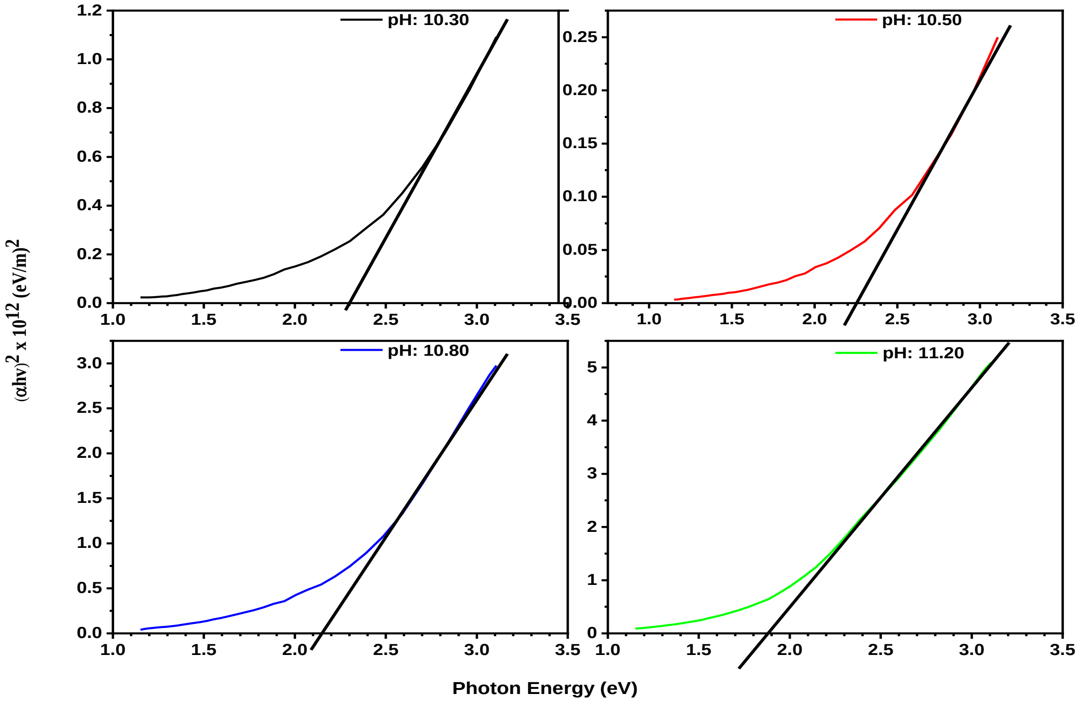

Figure 6. *Plot of (αhv)2 against Photon Energy for different pH.*

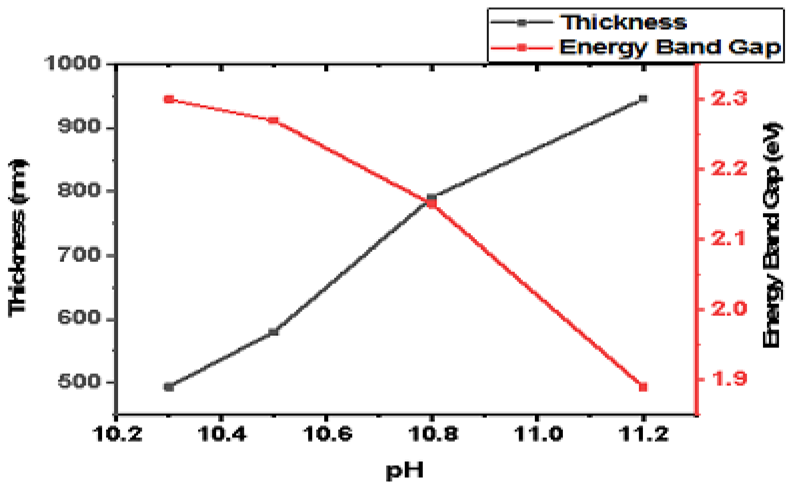

Figure 7. *Plot of Thickness (nm) and Energy Band Gap (eV) against pH for the deposited films.*

Figure 8 showed the plot of optical conductivities of the deposited films plotted against wavelength. Optical conductivities of these films decreases with increase in wavelength in a similar pattern. Film deposited at pH of 10.30 has optical conductivity values of $1.01 \times 10^{14}\ s^{-1}$ to $6.53 \times 10^{13}\ s^{-1}$ within UV region, $4.72 \times 10^{13} s^{-1}$ to $1.79 \times 10^{13} s^{-1}$ in VIS region and reduced further to $8.90 \times 10^{12} s^{-1}$ in NIR region. Film deposited at pH of 10.50 has values of $1.07 \times 10^{14}\ s^{-1}$ to $2.86 \times 10^{13}\ s^{-1}$ within UV region, $1.95 \times 10^{13} s^{-1}$ to $6.44 \times 10^{12} s^{-1}$ in VIS region and to $2.51 \times 10^{12} s^{-1}$ in NIR region. Film deposited at pH of 10.80 has values of $9.08 \times 10^{13}\ s^{-1}$ to $8.09 \times 10^{13}\ s^{-1}$ within UV region, $7.01 \times 10^{13}\ s^{-1}$ to $3.21 \times 10^{13} s^{-1}$ in VIS region which decreased down to $1.11 \times 10^{13} s^{-1}$ in NIR region. Film deposited at pH of 11.20 has values of $5.57 \times 10^{13} s^{-1}$ to $4.24 \times 10^{13} s^{-1}$ in VIS region and reduced to $1.88 \times 10^{13} s^{-1}$ in NIR region. The results showed that films of PbAgS have good optical response with UV, VIS and NIR regions except for film deposited at pH of 11.20 that showed optical responses only with VIS and NIR regions.

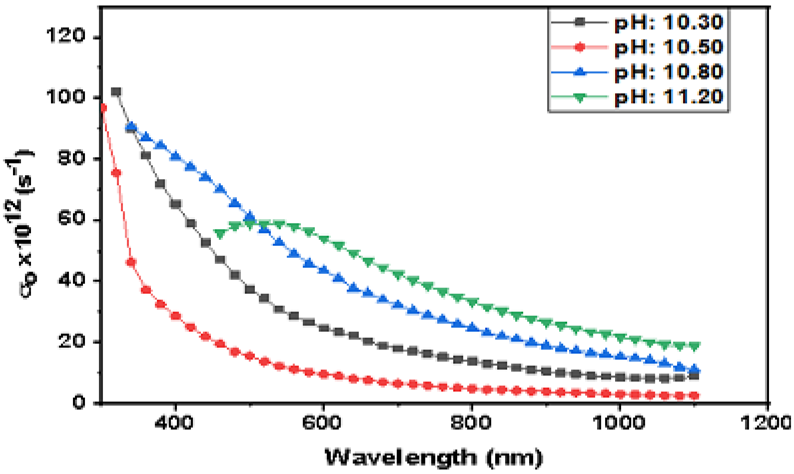

Figure 8. *Plot of Optical Conductivity of PbAgS Thin Films against Wavelength for different pH.*

3.2. Electrical Properties of PbAgS thin films

Electrical characterization of $PbAgS$ films was done using four point probe. The values of current and voltage for each of the films were recorded and used to calculate the electrical properties of these films. Sheet resistivity and electrical conductivity of the films were evaluated using equation (8) and (9). The results showed that the pH of the films affect the electrical properties of the films. Sheet resistivity of the films was found to decrease as pH of the reaction bath increase while the electrical conductivity of the films increased as pH of the films increases. This result is in line with decrease in resistivity as pH decreased as obtained by [33,34]. Figure 9 showed the variation of sheet resistivity and electrical conductivity plotted against pH. The electrical conductivity of the films ranged from $1.337 \times 10^{-3} S/cm$ to $9.334 \times 10^{-3} S/cm$. Sheet resistivity values obtained in this work are within the order of $10^{2}\ \Omega\, cm$ for PbS obtained by [35] and the electrical conductivity values obtained are within the order of 10^{-6} to $10^{-3} S/cm$ obtained by [36]. These results are also in line with electrical properties of Ag_2S obtained by [37].

Table 2. *Variation of Electrical Properties of PbAgS with pH.*

pH	Voltage x 10^{-1} (m)	Current (μA)	Thickness x 10^{-5} (cm)	Resistivity x 10^{2} (Ω cm)	Conductivity x 10^{-3} (S/cm)
10.3	8.369	0.250	4.938	7.479	1.337

10.5	6.170	0.486	5.803	4.545	2.200
10.8	4.374	0.578	7.914	1.988	5.031
11.2	7.233	2.890	9.455	1.071	9.334

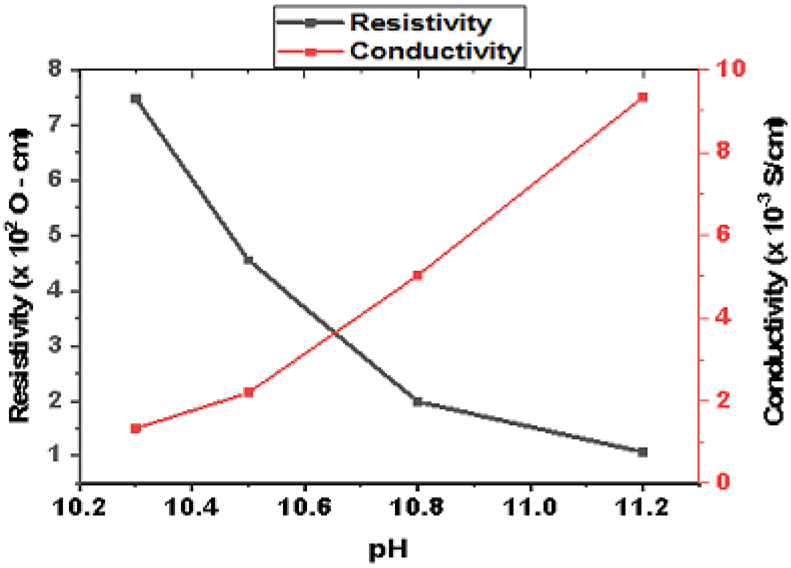

Figure 9. *Plot of Variations of Electrical Properties against pH of PbAgS thin films.*

4. Conclusion

Chemical bath deposition of lead silver sulphide thin films were carried at room temperature using precursors for lead, silver, sulphur and ammonium hydroxide solution. The effect of pH of the reaction bath for the deposition of these thin films was achieved by varying the volume of ammonium hydroxide used in each bath. The pH of the bath was measured with a pH meter. Optical properties of the films studied within UV, VIS and NIR showed that they possess good optical properties suitable for various industrial applications. Absorbance values of the films were found to be within the range of 1.40 abr. units to 0.07 abr. unit while the transmittances ranged from 3.76 % to 86.70 %. Reflectance of the films ranged from 1.48 % to 20.35 % while the refractive index ranged from 1.28 to 2.64. The extinction coefficient is within the order of 10^{-3} to 10^{-1} and the optical conductivity ranged from $2.51 \times 10^{12} s^{-1}$ to $1.06 \times 10^{14}\ s^{-1}$. The energy band gap of the films ranged from 2.30 eV to 1.89 eV. Film thickness ranged from 493.83 nm to 945.52 nm. The electrical conductivity of the films ranged from $1.337 \times 10^{-3} S/cm$ to $9.334 \times 10^{-3} S/cm$. These values showed that the deposited films are semiconducting thin films. Based on these properties, these films could be useful in electronics industry such as camera lens and eye glass coating and in architectural industry such as in poultry house and coating of windows and doors for passive heating and cooling of house.

Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this article.

Author Contributions

Conceptualization: E. E. E., E. I. A.; Methodology: E. E. E., E. I. A., O. N. L.; Software: E. E. E., O. N. L.; Validation: E. I. A.; Formal analysis: E. E. E., E. I. A., O.

N. L; Investigation: E. E. E., E. I. A., O. N. L; Resources: E. E. E.; Data Curation: E. E. E., O.N.L., E. I. A.; Writing – original draft preparation: E.E.E., O.N.L.; Writing – review and editing: E. E. E., E. I. A., O. N. L.; Supervision: E. I. A.; Project administration: E. E. E., E. I. A., O. N. L.; Funding acquisition: E. E. E

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