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Original Research Article

Effect of Volume of Zn2+ Solution on the Optical Properties of Spray-Deposited *H.Sabdariffa*(Zobo) Dye-Doped ZnS Thin Films

Igweoko Anthony Egwu[1]*, Idenyi Ndubuisi Edennaya[2], Idu Hyacinth, K.[3], Agbo Alfred[4]

[1]Department of Mechatronics Engineering, Alex Ekwueme Federal University, Ndufu-Alike, Ikwo, Nigeria
[2]Department of Mechanical and Bioresources Engineering, Ebonyi State University, Abakaliki, Nigeria
[3]Department of Industrial and Medical Physics, David Umahi Federal University of Health Sciences, Uburu, Nigeria
[4]Department of Metallurgical and Materials Engineering, Enugu State University of Science and Technology, Enugu, Nigeria

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***Corresponding author:** Igweoko Anthony Egwu
Department of Mechatronics Engineering, Alex Ekwueme Federal University, Ndufu-Alike, Ikwo, Nigeria

Abstract

This work presents the successful deposition of *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with different volumes of Zn^{2+} solutions on cleaned glass substrate slides using inexpensive chemical spray-pyrolysis technique with the substrates kept at a constant temperature of 200°C. The final spray solution for the growth of the films contained aqueous solutions of $ZnSO_4.7H_2O$, $SC(NH_2)_2$, and *H.Sabdariffa* dye extracts. $ZnSO_4.7H_2O$ and $SC(NH_2)_2$ served as precursors for Zn^{2+} and S^{2-} respectively while *H.Sabdariffa* was used as dye. The sprayed film properties were characterized for optical properties using a HINOTEK 756S UV-VIS spectrophotometer. From the absorbance's spectral analysis, other optical parameters such as transmittance, reflectance, absorption coefficient, band gap energy and optical density were calculated. The films indicated high absorbance in the UV region and high transmittance in the VIS – NIR regions, whereas reflectance is generally low. The films indicated direct band gap energy range of 2.10eV to 2.62eV. The optical density indicated range of 0.5 to 6.16. Based on relatively low reflection, strong absorption in UV region, high transmission and wide band gap indicated by the films. Therefore, it can be concluded that the films are suitable for the fabrication of solar cells. Also, the films are good for anti-dazzling coatings and thermal control coatings in automotive and architectural industries respectively.

Keywords: Absorbance, dye extracts, *Hibiscus Sabdariffa* (Zoborodo), solutions, optical constant.

1. INTRODUCTION

In recent years, the field of materials science and engineering has witnessed an ever-growing interest in exploring organic compounds as potential enhancers of various material properties. One such compound that has attracted significant attention is *Hibiscus Sabdariffa* (Zoborodo) dye extracts. It is obtained from *Hibiscus Sabdariffa* (Zoborodo) plants. This dye extract holds promise for its unique chemical composition and potential applications in modifying the properties of thin films. Particularly intriguing is its potential influence on spray-deposited zinc sulphide (ZnS) thin films. ZnS is among the most potential semiconductors for use as window layers in the production of inexpensive PV cells. This is because its basic elements, sulfide (S) and zinc (Zn), are inexpensive and widely available. In comparison with cadmium materials, it is eco-friendly (Institute of Solid State Physics, 2020; Global Safety Management, Inc, 2015; ThermoFisher Scientific, 2015). Zinc sulphide has direct wide band gap energy of 3.68eV (Unaogu and Okeke, 1990). It can be applied in several devices such as solar cells, light emitting displays, optoelectronic devices, photocatalysis, and luminescent materials (Seung *et al*., 2010). Recently, there has been a lot of interest in the usage of organic dye extracts. However, this is the case in many nations due to the stringent regulations placed on certain substances because of their toxicity and related allergic reactions (Kamel *et al*., 2005). Synthetic dyes, as, have been shown by research, are suspected to release harmful chemicals which are allergic, carcinogenic and dangerous to human health (Neha and Vidya, 2011). On the other hand, organic dyes are eco-friendly, non-toxic, inexpensive and are locally readily available in abundance. In the literature, there are numerous papers published on the deposition of ZnS thin films using variety of thin film deposition technologies. ZnS thin films have also been employed in diverse fields of applications like antireflection coatings, optical coatings, phosphor, electroluminescent and other light emitting devices. Various precursor solutions have also been employed to grow ZnS films such as acetates, chlorides, nitrates. Also various dopants (organic and inorganic) have been employed to alter the inherent characteristics of ZnS thin films, hence enhancing their adaptability for use in a variety of device applications. This research is novel for modifying the properties of ZnS for enhanced device applications because, as far as we know, no author has reported the introduction of dye extracts of *Hibiscus* at various volumes, to optimize the properties of ZnS films. In the present investigation, the aim of the study is to grow thin films of *H. Sabdariffa*(Zobo) dye-doped ZnS with various volumes of Zn^{2+} solutions and to characterize the layers using optical spectroscopy to investigate the optical constants. The effect of the volumes of Zn^{2+} solutions on the optical properties is reported. This report is a fundamental step toward exploring new pathways for utilization of ZnS-based thin films in various device designs.

2. MATERIALS AND METHOD

2.1 Substrate cleaning and reagents

Because it lowers defect creation, non-uniformity, grain boundary formation, dislocation density, and improves the overall quality of the thin film, substrate cleaning is essential to thin film deposition. The 75 x 25 mm, 1.0 mm thick soda-lime substrates (glass slides) were purchased from nearby vendors. Following a 60-minute immersion in strong hydrochloric acid, the soda-lime substrates were taken out, sponge-washed in a kiln detergent solution, and rinsed with sachet water prior to the spray. After that, distilled water was used to rinse the glass slides. Lastly, a sterilizing oven was used to adequately dry them. All the source chemicals employed for the spray-deposition of ZnS thin films were analytical grade, and were obtained from Sigma Aldrich UK through local suppliers and used directly without further purification. The source of the zinc ions and sulphur ions were zinc sulphate ($ZnSO_4.7H_2O$) and Thiourea ($SC(NH_2)_2$) respectively.

2.2 Extraction of the *Hibiscus Sabdariffa* (Zoborodo) dye extracts

For four hours, 100g of dry Zobo leaves were boiled in 200ml of distilled water at 70°C. The leaves were carefully removed from the solvent used for extraction. After filtering into a previously dry and clean container, the dye extracts were sealed with a cap and kept at room temperature.

2.3 Deposition of *H. Sabdariffa*(zobo) dye doped ZnS thin films with different volumes of Zn^{2+} solutions

5 milliliters of 0.02M ZnSO4.7H2O, 20 milliliters of 0.02M $SC(NH_2)_2$, and 20 milliliters of *H. Sabdariffa* were measured into a 50 milliliter glass beaker. The mixture was then vigorously stirred at high speed for approximately 15 minutes using a magnetic stirrer to obtain a homogenous solution. Following the stirring, 10 ml of the solution was measured out with a pump syringe and placed into a spray-pyrolysis sample bottle attached to the air brush's nozzle valve rod. The solution was then sprayed for 30 seconds onto heated substrates (glass slides) using an electrical hot plate to raise the substrate temperature to 200ºC. The above procedures were repeated when the volumes of zinc ion solutions were varied to 10ml, 15ml, and 20ml and keeping the volumes of *H. Sabdariffa* and sulphur ion solutions constant for other depositions.

The idea was to investigate the effect of volumes of zinc ion solutions on deposition of *H.Sabdariffa* dye- doped ZnS thin films. The volumes of the zinc ion were measured with a beaker and recorded as shown in Table 1. Table 1 shows the spray constituents for the deposition of *H.Sabdariffa* dye-doped ZnS thin films with varying volume of zinc ion solution.

Table 1: Variation of volume of Zn^{2+} solution for the spray-deposition of *H.Sabdariffa* (Zobo) dye-doped ZnS thin films

Sample	$ZnSO_4.7H_2O$ (ml)	$SC(NH_2)_2$ (ml)	*H.Sabdariffa* dye (ml)
5ml ZnS dye extracts	5.00	20.00	20.00
10ml ZnS dye extracts	10.00	20.00	20.00
15 ml ZnS dye extracts	15.00	20.00	20.00
20ml ZnS dye extracts	20.00	20.00	20.00

2.4. Characterization and Computation of Optical Properties

The absorbance of the spray-deposited films in the wavelength range of 400–1100 nm was measured using an HINOTEK 756S UV-VIS Spectrophotometer at the Nano Laboratory of the University of Nigeria Enugu State, Nsukka (UNN). Other optical parameters such as transmittance, reflectance, absorption coefficient, energy band gap and optical density were calculated using the following equations as obtained from literatures. Transmittance of the films were calculated using Equation (1) given by (Emegha *et al*., 2019, Offor *et al*., 2020).

$T = 10^{-A}$ ……………….(1)

Reflectance of the films were obtained using the expression in Equation (2) as given by (Offor *et al*., 2020).

R = 1- (A+T) …………………. (2)

The absorption coefficient (α) of the films were calculated from the transmittance values using the Equation (3) as given by (Igweoko, 2019; Ukpai, 2021):
α = 2.303A/t …………………... (3)

Were (t) is the thickness of the deposited thin films.

The energy band gaps were estimated using Tauc's model given in Equation (4) as (Meshram and Thombre, 2019; Meshram and Thombre, 2021).
$(\alpha h v)^{n} = (hv - Eg)$ …………………. (4)

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 $(\alpha h\nu)^2$ against the photon energy (hν) at $(\alpha h\nu)^2 = 0$.

Optical densities were estimated using Equation (5) (Nwofe, 2017).
ρ = αt ………………. (5)

Where t is the film thickness.

3. RESULTS AND DISCUSSION

3.1 Absorbance spectra

The absorbance spectra for *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with different volumes of Zn^{2+} solutions are shown in Fig.1. The absorbance generally decreased with wavelength exhibiting a minimum in the longer wavelength region (infrared region). This decrease in absorbance with increase in wavelength agrees with previous reports in the literature (Onochie, *et al.*, 2021; Igweoko, 2018; Nwofe, 2017, Saeed and Suhail, 2012; Ozutok *et al*., 2012; Offor *et al*., 2018). In addition, the maximum absorbance of 2.2, 2.6 and 2.7 for *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with 15ml, 20ml, and 5ml volumes of Zn^{2+} solutions respectively were above the limit of 2.0 stipulated by Lambert-Beer's law. Nevertheless, the maximum absorbance of 1.6 for *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with 10ml volume of Zn^{2+} solution was below the limit of 2.0 stipulated by Lambert-Beer's law. The concentration of the reacting species may have contributed to the high absorbance. Improved absorption occurred in the shorter wavelength region (higher energy region) of 440nm. For *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with 20ml volume of Zn^{2+} solution, absorbance decreased from 2.6 at 440nm to 0.8 at 1100nm. For *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with 15ml volume of Zn^{2+} solution, absorbance decreased from 2.2 at 440nm to 0.4 at 1100nm. For *H. Sabdariffa*(zobo) dye-doped ZnS thin films with 10ml volume of Zn^{2+} solution, absorbance decreased 1.6 at 440nm to 0.2 at 1100nm. For *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with 5ml volume of Zn^{2+} ion solution, absorbance decreased from 2.7 at 440nm to 0.7 with 1100nm. Strong absorptions were observed at wavelength of 440nm. Hence the films have potential application in fabrication of solar cells.

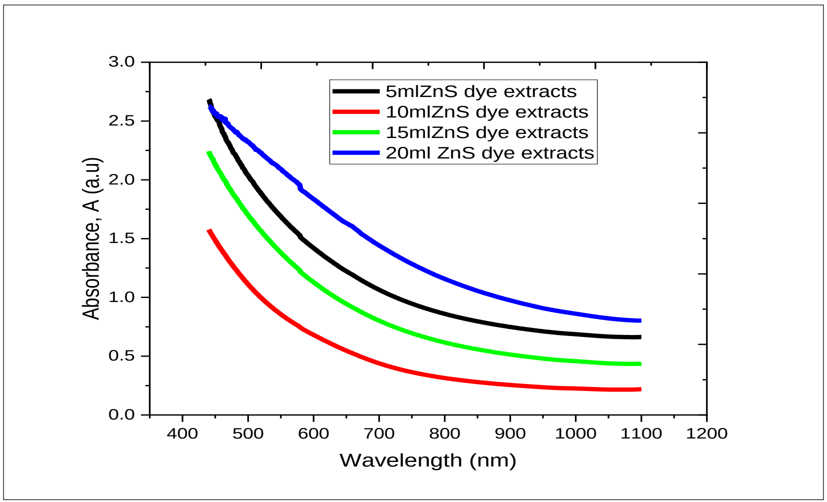

Fig. 1: Plots of absorbance against wavelength for *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with different volumes of Zn^{2+} solutions

3.2 Transmittance spectra

The transmittance spectra for *H.Sabdariffa*(Zobo) dye-doped ZnS thin films with different volumes of Zn^{2+} solutions are shown in Fig.2. The transmittance generally increased with wavelength exhibiting a maximum in the longer wavelength region (infrared region) which concurs with the report of other authors (Wanjala, 2016; Nwofe, 2017; Igweoko, 2018; Offor *et al*., 2020). For *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with 20ml volume of Zn^{2+} solution, transmittance increased from 0.5% at 400nm to 37%. For *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with 15ml volume of Zn^{2+} solution, transmittance increased from 0.5% at 400nm to 22% at 1100nm. For *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with 10ml volume of Zn^{2+} solution, transmittance increased from 2.5% at 400nm to 62% at 1100nm. For *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with 5ml volume of Zn^{2+} solution, transmittance increased from 0.5% at 400nm to 16% at 1100nm. In addition, there is no clear trend in relation between transmittance and volume of Zn^{2+} solution as can be seen in the plots. Agbo (2013) reported no clear trend in relation between transmittance and annealed temperature. The high transmittance shown by the films in the NIR region makes the films good materials for thermal control coatings inside buildings and construction of poultry walls and roofs for warming of young chicks.

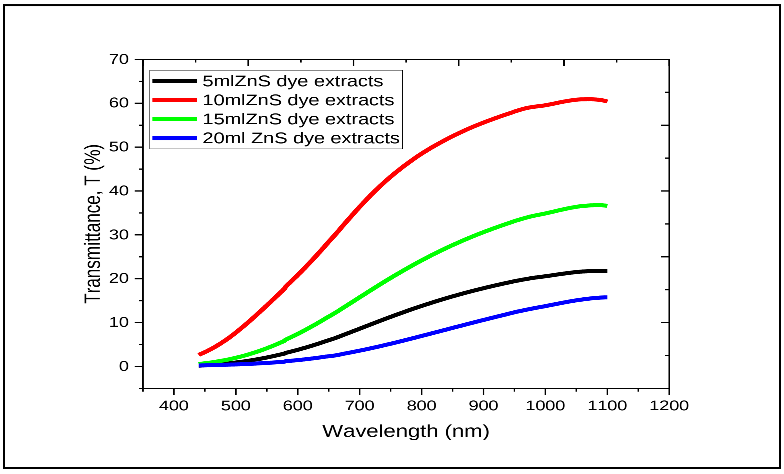

Fig. 2: Plots of transmittance against wavelength for *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with different volumes of Zn^{2+} solutions

3.3 Reflectance spectra

As observed in the reflectance spectra in Fig 3, the peak reflectance is about 20%. Generally, the reflectances of the films were low suggesting that most of the light were either absorbed or transmitted. These findings support the report of other authors in the literature (Ukpai, 2021). In addition, similar observation of increase in reflectance as wavelength increased have been reported by Igweoko (2018) for ZnS thin films dye doped with *Naulcea latifolia* (Uvuru-ilu) extracts grown by solution growth technique. Nevertheless, the peak reflectance value was lower in magnitude when compared with the report of Igweoko (2018). Furthermore, the low reflectance exhibited by the *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with different volumes of Zn^{2+} solution showed that these films are poor reflectors of light and hence they are appropriate for photovoltaic cell applications.

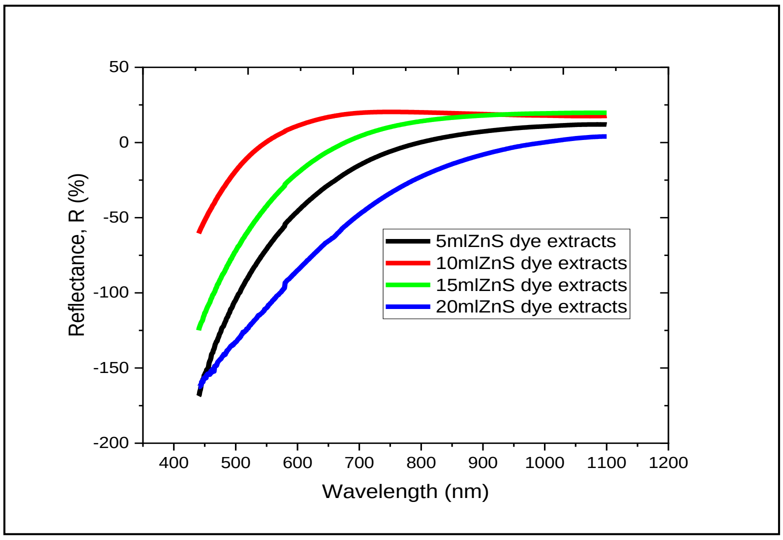

Fig. 3: Plots of reflectance against wavelength for *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with different volumes of Zn^{2+} solutions

3.4 Absorption cofficient

The graphs (Fig 4) show that absorption coefficient (α) generally increased with increase in photon energy. For *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with 20ml volume of Zn^{2+} solution, absorption coefficient increased from 0.05 x 10^9m^{-1} at 1.13eV to 0.17 x 10^9m^{-1} at 2.82eV. For *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with 15ml volume of Zn^{2+} ion solution, absorption coefficient increased from 0.024 x 10^9m^{-1} at 1.13eV to 0.12 x 10^9m^{-1} at 2.82eV. For *H.*

Sabdariffa(Zobo) dye- doped ZnS thin films with 10ml volume of Zn^{2+} solution, absorption coefficient increased from 0.0095 x $10^9 m^{-1}$ at 1.13eV to 0.074 x $10^9 m^{-1}$ at 2.82eV. For *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with 5ml volume of Zn^{2+} solution, absorption coefficient increased from 0.05 x $10^9 m^{-1}$ at 1.13eV to 0.21 x $10^9 m^{-1}$ at 2.82eV. This variation in coefficient of absorption with increase in photon energy agrees with the report of others in the literature (Ghazai *et al*., 2020; Offor *et al*., 2020; Meshram and Thombre, 2021).

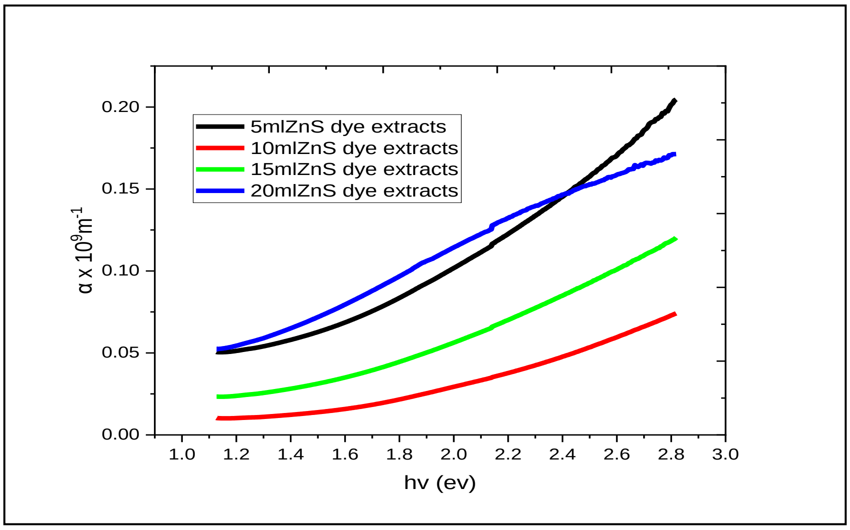

Fig. 4: Plots of absorption coefficient (α) as a function of photon energy for *H.sabdariffa* dye-doped ZnS thin films with different volumes of Zn^{2+} solutions

3.5 Band gap

The optical energy gap for the direct allowed transition between valence bands and conduction bands of *H.sabdariffa* dye-doped ZnS thin films with different volumes of Zn^{2+} solutions were calculated from Equation (2.27) using n=1/2.The values of the band gap of *H.sabdariffa* dye-doped ZnS thin films with different volumes of Zn^{2+} solutions for the direct transition can be determined by extrapolating the straight line portion of the $(\alpha h\nu)^2$ versus (hv), as shown in Fig 4 & 5. Optical energy gap was typically in the range 2.10-2.62eV. The obtained range of optical energy gap was above the range reported by Thiruvenkadam and Leo Rajesh (2014). However, it is below the range reported by Suhail and Ahmed (2014) for copper- doped ZnS thin films deposited by spray pyrolysis method, Suhail and Ahmed (2014) for lead-doped ZnS thin films deposited by spray pyrolysis method, Okpara (2018) for manganese-doped ZnS thin films deposited by spray pyrolysis method and Wanjala (2016) for tin-doped ZnS thin films deposited by spray pyrolysis method. Thin films with band gap energy lower than 1.90eV are used as absorber materials in solar cell architecture while those with higher band gap energy can be used as window layers (Asogwa *et al*., 2009). Since the values obtained in this research are above 1.90eV, it is expected that a high efficiency solid state solar cell (SSSC) window material for photovoltaic conversion of solar energy, is plausible.

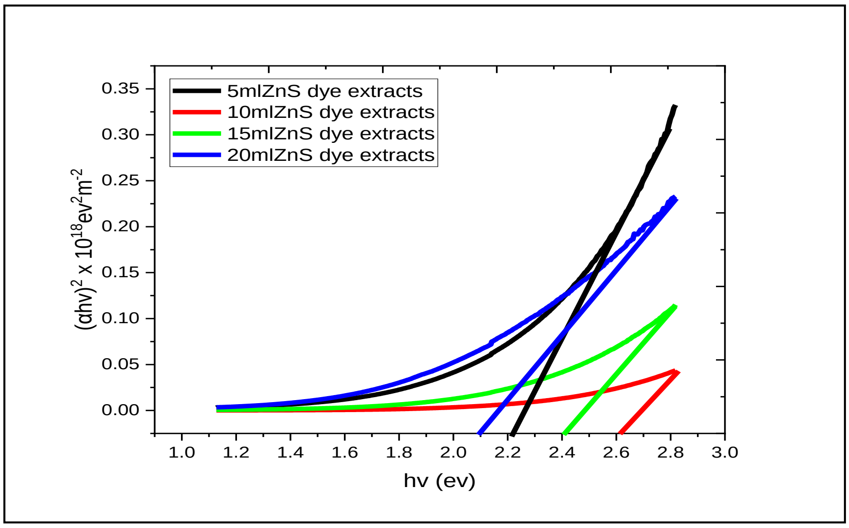

Fig. 5: Plots of $(\alpha h\nu)^2$ as a function of photon energy (hv) for *H.sabdariffa* dye- doped ZnS thin films with different volumes of Zn^{2+} solutions

3.6 Optical density

Fig 6 shows plots of optical density (ρ) as function of photon energy (hv) for *H. Sabdariffa*(Zobo) dye-doped ZnS thin films doped with different volumes of Zn^{2+} solutions. It was observed that the optical density increased as the photon energy increased. For *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with 20ml volume of Zn^{2+} solution, optical density increased from 1.81 at 1.13eV to 6.04 at 2.82eV. For *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with 15ml volume of Zn^{2+} solution, optical density increased from 0.99 at 1.13eV to 5.17 at 2.82eV. For *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with 10ml volume of Zn^{2+} solution, optical density increased from 0.50 at 1.13eV to 3.61 at 2.82eV. For *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with 5ml volume of Zn^{2+} solution, optical density increased from 1.53 at 1.13eV to 6.16 at 2.82eV. Similar variation of increase in optical density as photon energy increased have also been reported for ZnS thin films dye doped with *Naulcea latifolia*(Uvuru-ilu) extracts by Nwofe (2017). The obtained range of values are higher in magnitude when compared with the range reported by Nwofe (2017). This difference in range of values could be attributed to the type of dye, deposition conditions and method used for the film growth.

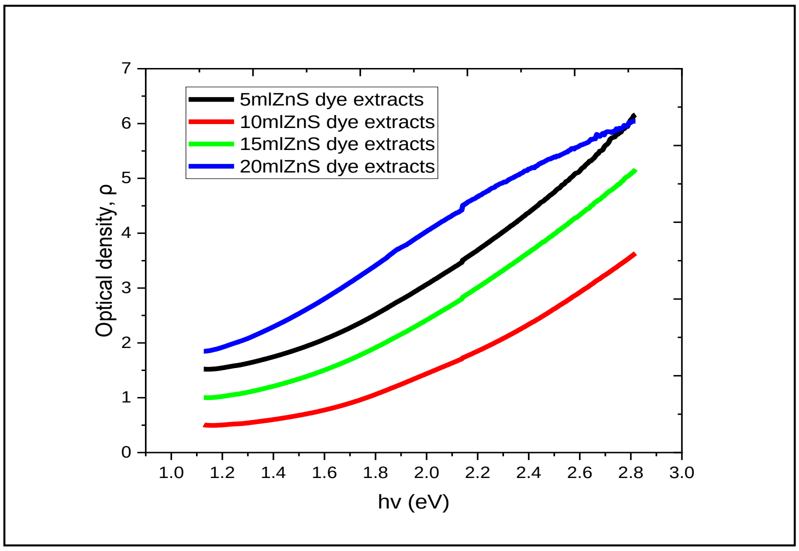

Fig. 6: Plots of optical density as a function of photon energy (hv) for *H.sabdariffa* dye-doped ZnS thin films with different volumes of Zn^{2+} solutions

4. CONCLUSION

Spray-pyrolysis technique was used for the deposition of *H. Sabdariffa*(Zobo) dye-doped ZnS thin films with different volumes of Zn^{2+} solutions. Optical properties of the films investigated within UV, VIS and NIR regions indicated that they possess good optical properties suitable for various industrial applications. Absorbance values of the films were found to be within the range of 0.2 abr. units to 2.7 abr. unit while the transmittances ranged from 0.5 % to 62 %. Reflectance of the films ranged from -170 % to 20 %. The absorption coefficient ranged from 0.0095 x $10^9 m^{-1}$ to 0.21 x $10^9 m^{-1}$. The energy band gap of the films ranged from 2.10eV to 2.62eV. Optical density ranged from 0.5 to 6.16. Based on the properties indicated by the films, they could be useful in automotive industries for anti-dazzling coatings to minimize the effect of dazzling light from vehicles following behind at night; in architectural industries for thermal control coatings inside buildings such as in coating of roofs and walls of poultry to generate heat for the purpose of warming young birds and in solar industries as promising candidate materials for window layers in solar cells fabrication.

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