Influence of Ammonium Chloride Buffer Solution on the Photo-Electro-Chemical Cell and Optical Performance of MoS_2 Thin Films

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ORIGINAL RESEARCH

Abstract-Thin films of molybdenum disulphide (MoS_2) of approximately same thickness of 290 nm have been successfully synthesised with and without ammonium chloride (NH_4Cl) buffer solution using electrodeposition growth technique. The experimental results obtained showed that NH_4Cl buffer solution has an obvious effect on the photo-electro-chemical (PEC) cell and optical performance of electrodeposited MoS_2 (ED – MoS_2) thin films. The MoS_2 thin films prepared from NH_4Cl buffer solution possess better electronic properties than the ones prepared without the buffer when measured under dark condition. Open circuit voltage of~0.556 V and ~0.382 V were obtained for samples prepared with and without a buffer solution respectively. MoS_2 from buffer solution had higher maximum power (P_{max}) of 0.710 mW while the ones without a buffer solution had a P_{max} value of 0.258 mW. Also, both materials showed different electrical conductivity type possibly due to the influence of chlorine in the prepared electrolytic bath. The optical results revealed similarity in the energy band gaps of both materials with a little variation. Energy band gaps of 2.15 eV and 2.20 eV were obtained for MoS_2 layers grown with and without a buffer solution respectively. In the same vein, both MoS_2 thin films showed optical conductivity of same order but MoS_2 thin films prepared with buffer solution have higher magnitude than the ones without a buffer solution. These preliminary results revealed that more efficient MoS_2 films for photovoltaic application can be deposited using an electrolytic bath containing NH_4Cl buffer.

Keyword-— Electrodeposition, Electrical Conductivity Type, Energy Band Gap, Photo-electro-chemical (PEC) Cell, Thin Films.

1 INTRODUCTION

Molybdenum disulphide (MoS_2) is a transition metal dichalcogenide (TMD) (Pham and Yeom, 2016; Ye *et al.*, 2015) and a diamagnetic semiconductor (Somoano *et al.*, 1973), that has a layered structure and has shown remarkable mechanical, optical, electrical and optoelectronic properties as well as thermal stability (Ahn *et al.*, 2015; Pham and Yeom, 2016; Ye *et al.*, 2020). MoS_2 is also considered as an analogue of graphene (Dang *et al.*, 2017; Lee *et al.*, 2013). These properties have increased the drive towards numerous technical applications of MoS_2 (Benavente *et al.*, 2002). Exhibition of great potential for applications in optoelectronics, energy harvesting, and detection of environmental molecule due to its ultra-high sensitivity has been reported (Lee *et al.*, 2013; Pham and Yeom, 2016). MoS_2 thin films have also been applied in a variety of devices like solar cells, catalysts, transistors and batteries (Kite *et al.*, 2017; Zhou *et al.*, 2013).

Another interesting quality of MoS_2 is the modulation of its energy band gap. The band gap can be adjusted from the indirect band gap (1.29 eV) to the direct band gap (1.90 eV) (Ahn *et al.*, 2015). Also, a uniform polycrystalline thin film of 1.70 eV has been obtained through electrodeposition (Benavente *et al.*, 2002). Growth techniques like metal organic vapour deposition (MOCVD), chemical vapour deposition (CVD), pulsed laser evaporation, radio frequency sputtering, and electrodeposition have been used to grow nanostructure MoS_2 thin films (Benavente *et al.*, 2002; Kite *et al.*, 2017; Ghosh *et al.*, 2013; Li *et al.*, 2005).

The growth of thin film materials using the electrodeposition method offers a lot of advantages which include scalability, waste minimization, convenient process control, etc. (Echendu *et al.*, 2019). This growth technique provides an easy and convenient manipulation of material properties through variation of growth parameters which include temperature, deposition voltage, concentration and type of the precursors, deposition time, pH etc (Echendu *et al.*, 2019; Olusola *et al.*, 2015). The use of buffer solution plays a critical of role in electrodeposited materials. Akintunde (2000) reported the use of ammonium sulphate salt ($(NH_4)_2SO_4$) as buffer which forms a cadmium ion complex $[Cd(NH_3)4]^{2+}$ that reduce the formation of $Cd(OH)_2$ thereby minimizing the precipitation of cadmium sulphide (CdS) in an electrolytic bath for the growth of CdS (Akintunde, 2000). Antony *et al.*, (2005) reported the use of ammonium nitrate as a complexing agent to avoid the precipitating of compound in the solution and stabilization of the pH in the growth of zinc sulphide thin film. The presence of precipitates in the electrolytic bath gives rise to the growth of thin films with poor quality for device application (Atapattu *et al.*, 2017). It is therefore necessary to study the means to prevent precipitation in electrolytic bath as well as the stabilization of pH to enable the growth of thin films with good quality for device applications.

In a similar pattern, Zhang *et. al.*, (2019) employed different concentrations of ammonium chloride (NH_4Cl) as buffer solution to synthesise thiomolybdates solution. The authors observed variation in the colour of the synthesised molybdates solution as the concentration of NH_4Cl buffer changes which was attributed to the various species of thiomolybdates being produced. The use of NH_4Cl buffer solution has been demonstrated to improve the stability of thiomolybdates solution. Thiomolybdates

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solutions are salts derived from molybdates when one or more of oxygen atoms are replaced with sulphur.
In this work, the synthesis of thiomolybdates solution has been achieved using ammonium molybdates and sodium thiosulphates as *Mo* and *S* precursors respectively with and without an ammonium chloride buffer solution. The synthesised solution was used as the electrolyte to electroplate MoS_2 thin films. This work therefore seeks to study the influence of ammonium chloride buffer solution on the optical and photo-electro-chemical (PEC) cell properties of molybdenum disulphide thin films. The PEC cells are usually formed when an interface is created between the semiconductor and electrolyte. Details of the working principle of the PEC cell and the set – up can be found in (Olusola, 2016). The possibility of using the electrodeposited MoS_2 films for PEC solar cells application has been demonstrated in this work. For the first time, it has been experimentally shown in this work that NH_4Cl buffer solution can cause a type conversion of electrical conduction in MoS_2 thin films.

2 Experimental Details

2.1 Substrate Cleaning and Electrolytic Bath Preparation

Microcontroller – based Cypress Omni-101 Potentiostat has been used in this work for the electrodeposition of molybdenum disulphide thin films (MOS_2). The deposition was carried out on a fluorine – doped tin oxide (FTO) conducting glass substrates having a sheet resistance of 15 Ω/square. To ensure thin film adsorption and proper adhesion to the substrate, the substrate surface must be thoroughly cleaned. Hence, substrate cleaning is an important process in thin film deposition which must be carefully taken into consideration. The substrate cleaning was achieved using soap solution, deionised water and organic solvents namely methanol and acetone for dirt and oily removals. Two electrolytic baths were prepared in this work to carry out the MoS_2 thin films electrodeposition. The first bath was prepared in an aqueous medium without an ammonium buffer solution; that is, it was prepared using only deionised water. The second bath was prepared in an aqueous medium with the inclusion of ammonium buffer solution.

2.2 Preparation of MoS_2 Electrolytic Bath without Ammonium Chloride Buffer Solution

MoS_2 bath without an ammonium chloride buffer solution was prepared from an electrolyte containing two salts concentration which are 0.01 M of hexa-ammonium-hepta-molybdate-tetra-hydrate ($(NH_4)_6Mo_7O_{24}.4H_2O$) and 0.1 M of sodium thiosulphate pentahydrate ($Na_2S_2O_3.5H_2O$) using only deionised water as the aqueous medium. The first salt served as the precursor for *Mo* while the latter served as the sulphur precursor. Both salts were dissolved in a 500 ml beaker containing 400 ml of deionised water. The solution was stirred continuously until the salts completely dissolved in the aqueous solution. The bath colour was observed to be deep bluish after the mixture and continuous bath stirring. The pH of the mixed solution was measured to be ~5.00; this pH was used for the thin films growth.

2.3 Preparation of MoS_2 Electrolytic Bath using Ammonium Chloride Buffer Solution

MoS_2 bath with NH_4Cl buffer solution was prepared from an electrolyte containing an aqueous buffer solution, 0.01 M ($(NH_4)_6Mo_7O_{24}.4H_2O$) as *Mo* precursor and 0.1 M $Na_2S_2O_3.5H_2O$ as *S* precursor. The ammonium chloride buffer solution was prepared by dissolving 0.25 M of NH_4Cl in 400 ml of deionised H_2O. Same concentration of ($(NH_4)_6Mo_7O_{24}.4H_2O$) and $Na_2S_2O_3.5H_2O$ earlier added to the first bath was also added to the bath containing the buffer solution; this was done to allow effective comparison. After the complete dissolution of the salts in the aqueous NH_4Cl buffer solution, the pH was measured as 5.20. The pH was attuned to 5.00 by adding few droplets of diluted sulphuric acid. After a period of continuous stirring, it was observed that the colour of the electrolyte became light bluish with no change in pH value.

2.4 Thin Films Deposition

A 2 – electrode system which encompasses glass/FTO as the cathode / working electrode and graphite rod as the counter electrode was used in this work. Thin films of MoS_2 were grown from the two prepared baths at room temperature, same pH of 5.00, deposition time of 10 minutes, and at constant cathodic potential. The obtained results were compared accordingly to see the effect of ammonium chloride buffer solution on the material properties. Due to the very high deposition current density observed during growth, the magnetic stirring unit was put in the switched – off mode during thin film deposition. The optical properties of the ED – MoS_2 compound semiconductors was studied using Shimadzu UV – Visible spectrophotometer while the electronic parameters were obtained using ISM 490 ISO-TECH solar I – V simulator. The experiments and characterisations were carried out at the Federal University of Technology, Akure, Nigeria.

3 Results And Discussion

3.1 Optical Properties of Electrodeposited MoS_2 Thin Films

Optical parameters of electrodeposited MoS_2 thin films prepared with buffer solution (MDS_B) and without buffer solution (MDS) were obtained from the analysis of optical data gotten from UV – Visible Spectrophotometer. The obtained parameters as given in Table 1 are: energy band gap (E_g), absorption coefficient (α), extinction coefficients (k), refractive index (n), optical conductivity (σ_o), net dielectric ($E_r + E_i$) where E_r and E_i are real and imaginary dielectrics respectively, average transmittance ($T_{av.}$), average reflectance ($R_{av.}$), and average absorbance ($\alpha_{av.}$). As seen in Table 1, MDS_B films have higher absorption and extinction coefficient values than MDS thin films. These higher values are some of the reasons for the lower energy band gaps observed in MDS_B layers. Thus, electrons transiting from the valence band to the conduction band need fewer amount of photon energy to become conduction electrons provided the excited electrons do not get trapped by the defects in the materials band gap.

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Table 1. Summary of the optical parameters of ED-MoS_2 layers grown with and without a buffer solution.

Sample No	E_g (eV)	α $(cm)^{-1}$	k	n	$E_r + E_i$	σ_o (s^{-1})	A_{av}	T_{av}	R_{av}
MDS	2.20	8.14×10^4	0.27	2.08	4.91	0.99×10^{14}	0.23	0.65	0.12
MDS_B	2.15	8.75×10^4	0.30	2.28	5.86	1.32×10^{14}	0.31	0.54	0.15

Note: MDS is MoS_2 films prepared without buffer solution while **MDS_B** is MoS_2 films prepared with buffer solution.

Figure 1 is a diagrammatical representation of how MoS_2 dielectrics change with photon energy. As depicted in Figure 1, an increase was observed in the dielectric value for the films grown in a buffer medium from the infrared region of ~1.39 eV (892 nm) up to visible region of ~2.00 eV (620 nm). In the overall, the average dielectric value of MDS_B is higher than that of MDS as given in Table 1.

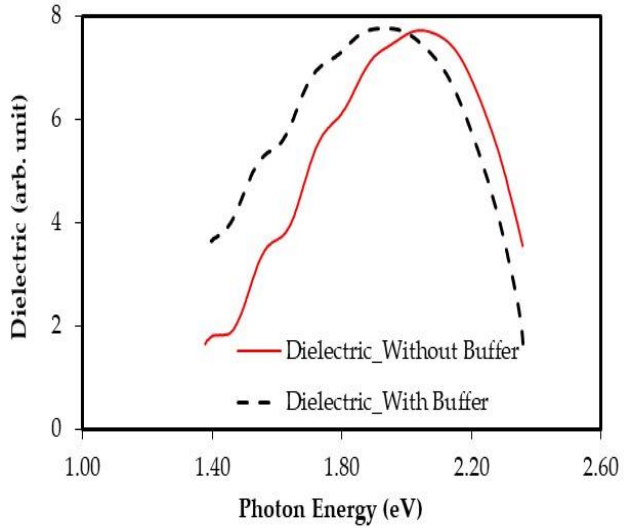

Fig. 1: Dielectric spectra of MoS_2 thin films grown with and without a buffer solution.

As reported by Olusola *et al.,* (2021), the existence of dielectric on the material surface has the potential to enrich the interface of the semiconductor thus, improving the potential barrier at the interface. In the same vein, materials with higher dielectric have improved optical / electrical conductivity due to the presence of a thin oxide layer; this oxide layer has a tendency to reduce the possibility of charge carriers tunnelling between the semiconductor and metal when it is interfaced with a metal contact. It is thus expected for MDS_B to have higher optical conductivity than MDS and this was seen in the results presented in Table 1 and Figure 2. Figure 2 illustrates how the optical conductivity spectra of MoS_2 thin films grown with and without a buffer solution changes with photon energy. As seen in Figure 2, the optical conductivity increases from the infrared region of approximately 889 nm (this is equivalent to a photon energy of ~1.39 eV) to the visible region range of ~541 nm to 546 nm where it reaches a peak of ~2.27 eV and 2.90 eV for MoS_2 films grown with a buffer solution and without a buffer solution respectively. The highest values of optical conductivity observed in the visible region for these set of thin films signify that MoS_2 thin films have the tendency to absorb more visible photons from the solar spectrum. Thus, Figure 2 explains that low and high energy photons can be absorbed by MoS_2 layers. These photons when absorbed excite more electrons from the valence band to the conduction band to contribute to the amount of conduction electrons needed for current generation.

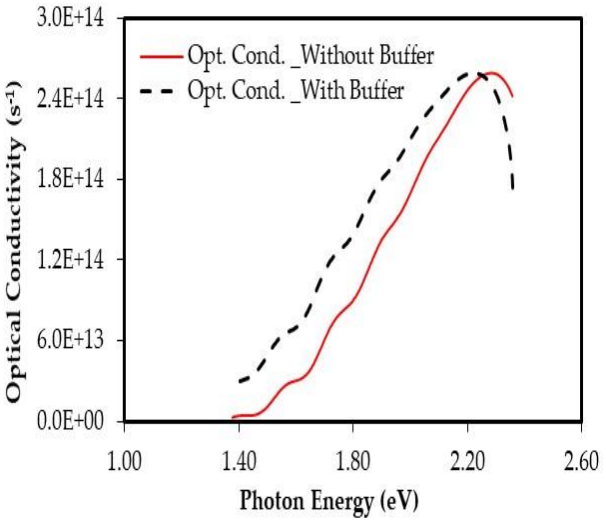

Fig. 2: Optical conductivity spectra of MoS_2 thin films grown with and without a buffer solution.

The average values of transmittance, reflectance and absorbance obtained for MoS_2 films grown with and without a buffer solution are given in Table 1. The average transmittance value of MDS films is generally higher than that of MDS_B films. This explains the reason why MDS films possess higher energy band gap than MDS_B films. The absorbance spectra of ED – MoS_2 layers grown with and without buffer solution as illustrated in Figure 3 decreased with increasing wavelength from the visible to the near infrared region. An assessment of absorbance data as specified in Table 1 gives the average absorbance to be approximately 0.23 and 0.31 for MDS and MDS_B films respectively. A general evaluation of Figure 3 shows that MoS_2 films can absorb both visible and infrared photons; this property makes it viable for photovoltaic applications. The high absorption peaks observed at the onset of the visible region points to the fact that MoS_2 thin films have the tendency to absorb more photons at the visible region of the solar spectrum while lesser amount of photons are absorbed at the near infrared region. Beyond the infrared wavelength of ~850 nm, MDS films offer zero absorbance to the infrared photons while MDS_B films still have an affinity for low infrared photon absorption. This figure explains the advantage of using a buffer solution in the growth of MDS_B compound semiconductors. Aside the fact that MoS_2 layers grown in a buffer medium have higher absorbance from the visible down to the infrared region, the materials continue to absorb photons from the solar spectrum even when the materials grown without a buffer solution ceased absorbing photons.

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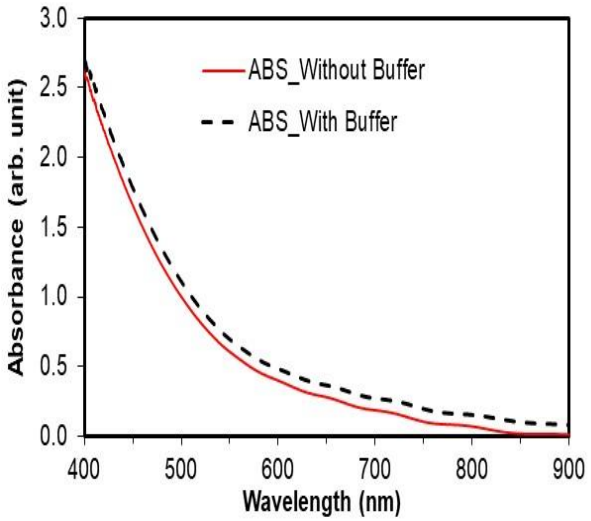

Fig. 3: Optical absorbance of MoS_2 thin films grown with and without a buffer solution.

The energy band gaps of the ED – MoS_2 thin films was estimated using the Tauc plot illustrated in Figure 4. As seen in the figure, extrapolation of the line of best tangent of the absorption curves to the horizontal photon energy axis where $(\alpha h\nu)^2$ is exactly equal to zero gives the material band gap. These values were found to be 2.15 and 2.20 eV for MoS_2 layers grown with and without buffer solutions respectively. Experimentally and theory – wise, it has been proven that bulk MoS_2 exists as an indirect semiconductor with an energy gap of 1.20 eV while monolayer MoS_2 exhibits a direct band gap nature with an energy gap of 1.90 eV. Researchers have also theoretically reported that monolayer MoS_2 can have band gaps as large as 2.20 to 2.80 eV. The report by Ye *et al.*, (2015) showed that there are no experimental proofs for the higher quasi particle band gap observed in MoS_2. However, in this work; it has been experimentally and successfully demonstrated that MoS_2 monolayer can have energy band gap up to 2.20 eV.

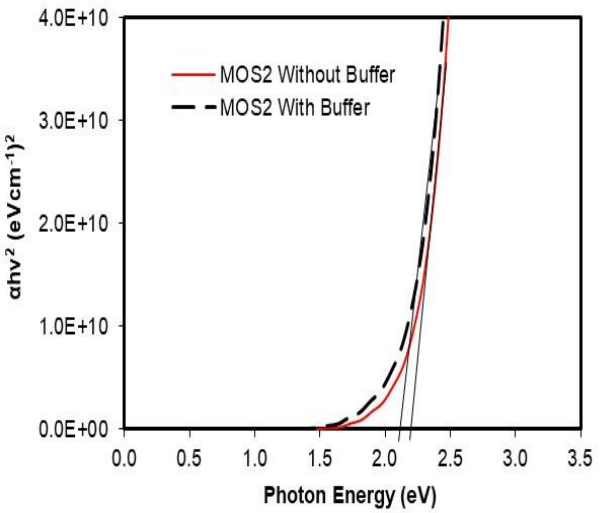

Fig. 4: Energy band gap estimation of MoS_2 thin films grown with and without a buffer solution.

3.2 Electronic Properties of Electrodeposited MoS_2 Thin Films

The chronoamperometry curve commonly referred to as current – time ($I - t$) characteristics is illustrated in Figure 5. As seen in Figure 5, MDS and MDS_B layers grown at same cathodic potential have nearly same high start-up deposition current density of ~16.5 mA.

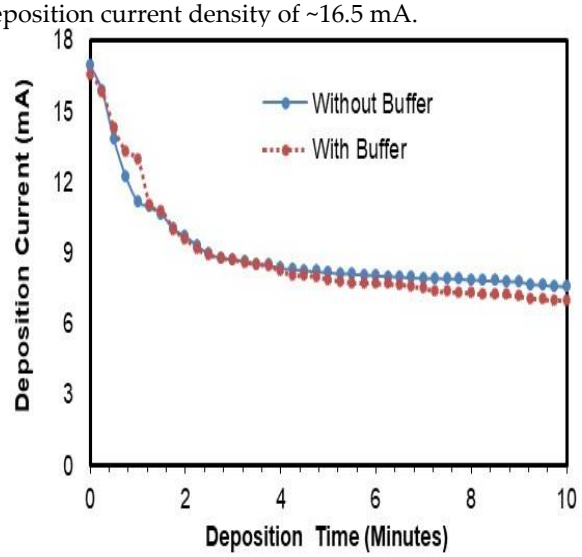

Fig. 5: Chronoamperometric spectra of MoS_2 thin films grown with and without a buffer solution.

As seen in Figure 5, the chronoamperometry curves of MoS_2 films prepared with and without buffer are similar; this explains the reason why both films have approximately same average deposition current of 9.0 mA. The chronoamperometry behaviour of other compound semiconductor has been reported in (Olusola *et al.*, 2021). A gradual decay was observed in the $I - t$ spectra from the onset of the deposition as films are coated on the substrate towards the end of the film growth. Using the estimated average deposition current, ED – MoS_2 thin films of approximately same thickness of 290 nm was electroplated. It was observed that the material thickness does not directly depend on whether the aqueous medium used in the thin film coating contains buffer solution or not. Rather, it is majorly dependent on growth parameters such as deposition duration and potential. The thickness estimation was done using Faraday's law of electrolysis indicated in (Olusola *et al.*, 2015). The PEC cell measurement set – up was used to determine the solar PEC cell performance and the type of electrical conduction exhibited by the electrodeposited thin films. The set – up comprises of a semiconducting electrode of unknown type, a counter electrode, a power supply source, I – V analyser, a digital DC voltmeter, and an electrolyte where the two electrodes are immersed.

A positive (p) PEC signal of 6.0 mV was obtained for MoS_2 thin films grown in an electrolyte devoid of NH_4Cl buffer while a negative (n) PEC signal of -8.0 mV was obtained for MoS_2 thin films grown in a buffer contained electrolyte. Despite the fact that these materials were deposited using similar growth parameters, a change in the electrical conductivity type was observed due to the buffer inclusion in the electrolyte. This type conversion from p – type MoS_2 to n – type MoS_2 can be attributed to the presence of chlorine in the electrolytic bath containing NH_4Cl buffer. Factors such as annealing in air or in a $CdCl_2$ atmosphere, can also cause a type conversion of a material electrical conductivity type (Salim *et al.*, 2015). Substitutional chlorine has been reported to be a donor in CdTe thin films (Valdna *et al.*, 1996). Since *Cl* is a shallow

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donor to MoS_2, the presence of *Cl* in the buffer solution can therefore act as an extrinsic n – type dopant to MoS_2 thin films; this explains the reason for the MoS_2 layers converting from p – type to n – type in an ammonium chloride buffer atmosphere. Generally, the conversion of thin films from p – type to n – type could arise as a result of (i) the activation of residual n – type dopants in the thin film; (ii) diffusion of donors from the grain boundaries into the thin films; (iii) diffusion of n-type dopants from the annealing environment into the thin films; and (iv) diffusion of n – type dopants from the underlying substrate layer into the semiconductor layers.

MoS_2 layers prepared using buffer solution of NH_4Cl salt (MDS_B) exhibited better electronic properties than the ones deposited using deionised water only (MDS) as seen from the PEC solar cell analyser results. MDS_B has higher open circuit voltage (V_{oc}) and short circuit current density (J_{sc}) of 0.556 V and 13.1 $mAcm^{-2}$ respectively while MDS has V_{oc} of 0.382 V and J_{sc} of 8.3 $mAcm^{-2}$. Results from the PEC solar cell $I - V$ analyser also revealed that MDS_B has a fill factor (*FF*) of 0.097 while MDS has a *FF* of 0.082. This signifies that the shape of the PEC solar cell was improved in MDS_B thin films. The fill factor, estimated using equation (1) is directly related to the shape of the solar PEC cell; it is also the fraction of electrical power that can be extracted from the PEC solar cell (Olusola, 2016).

$$FF = \frac{P_{MAX}}{V_{OC} \times J_{SC}} = \frac{V_{MAX} \times I_{MAX}}{V_{OC} \times J_{SC}} \quad (1)$$

where P_{MAX}, V_{MAX} and I_{MAX} are maximum power output, maximum voltage and maximum current respectively. Likewise, MDS_B has a resistance of 41.8 Ω while MDS has a resistance of 46.3 Ω; the current – voltage characteristics for the MDS_B and MDS is illustrated in Figure 6.

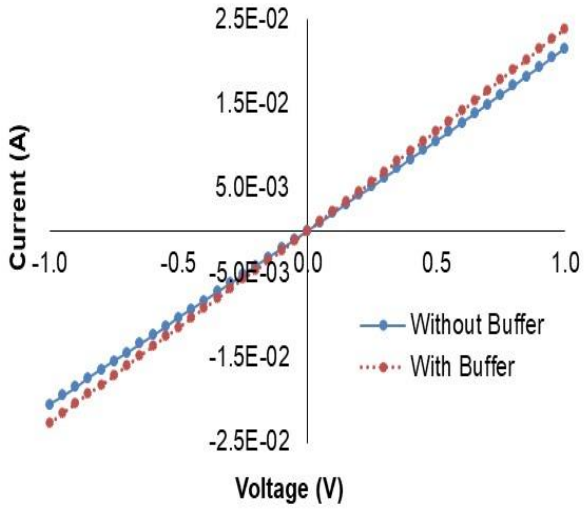

Fig. 6: Current – voltage characteristics of MoS_2 thin films grown with and without a buffer solution.

The lower resistance possessed by MDS_B can be attributed to the presence of chlorine in the electrolytic bath. Chlorine, being an extrinsic n – type dopant to MoS_2 monolayer has the capacity to improve the conductivity of MoS_2 compound semiconductor material. With the improvement in the material conductivity, the resistance of the material is lowered. This explains the reason for lower resistance value in MDS_B layer. Due to the lesser resistance possessed by the MDS_B material, an enhancement was observed in the optoelectronic properties shown in Table 2. With this enhancement, an efficiency of 0.71% was recorded for MDS_B while efficiency of ~0.26% was recorded for MDS layer. The cell efficiency (η) was estimated using equation (2).

$$\eta = \frac{FF \times V_{oc} \times J_{sc}}{P_{in}} \quad (2)$$

where $P_{in} = 100 \ \ mWcm^{-2}$ is the total solar incident power on a unit area under the standard AM1.5 illumination condition.

Table 2. Summary of the electronic parameters of ED – MoS_2 layers grown with and without a buffer solution.

Sample Name	V_{OC} (V)	J_{SC} ($mAcm^{-2}$)	*R* (Ω)	V_{MAX} (*V*)	I_{MAX} (*mA*)	*FF* (%)	η (%)
MDS	0.382	8.3	46.3	0.199	1.3	8.2	0.26
MDS_B	0.556	13.1	41.8	0.284	2.5	9.7	0.71

4 CONCLUSION

Synthesised thiomolybdates solutions with and without an ammonium chloride buffer solution have been successfully used to accomplish the electrodeposition of MoS_2 thin films. The chronoamperometric results revealed that the average deposition current for both MDS and MDS_B films are approximately same, thus revealing that both materials have approximately same thickness. Despite the similar thickness observed in both materials, their optoelectronic behaviours differ. The investigation performed using PEC cell measurements showed that the electrical conductivity type of MoS_2 thin films is influenced by the presence of NH_4Cl buffer solution. MoS_2 layers prepared without a buffer solution exhibited a p – type electrical conduction while the MoS_2 layers prepared in the presence of a buffer solution have n – type electrical conduction. The presence of chlorine in the buffer salt can be a major contributory factor to this type conversion. The experimental findings in this work provide a distinctive way to achieve n – type MoS_2 semiconductor. The optical absorption measurements revealed that MDS_B films have the potential to absorb more visible and infrared photons from the solar spectrum when compared to MDS films. This unique ability makes MDS_B films to thrive better as solar cells material than MDS films; this is further evident in the PEC solar cell parameters obtained for MDS_B films. An efficiency of 0.71% was verified for MDS_B films while efficiency of ~0.26% was obtained for MDS layers.

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