

SYNTHESIS AND PHOTOELECTROCHEMICAL CHARACTERIZATION OF WSe₂ PHOTOELECTRODES

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Abstract

Semiconductor electrodes based on thin films of tungsten diselenide have been prepared by electrochemical codeposition technique. Potentiostatic and galvanostatic methods of deposition were compared in terms of quality of electrodeposited films. Electrochemical conditions required for the formation of these materials endowed with acceptable functional activity was ascertained by investigating current voltage characteristics using electrochemical solutions of interest. The electrodeposits were characterized by their examination of current-voltage characteristics in dark and under illumination. All the prepared tungsten diselenide thin films exhibit p-type semiconductivity. WSe₂ films prepared galvanostatically exhibit somewhat enhanced photoresponse and substantially improved resistance towards electrochemical corrosion in comparison to those prepared under potentiostatic control.

Keywords: tungsten diselenide, electrochemical corrosion, p-type semiconductivity, photoresponse, thermal treatment

Introduction:

The generation of electricity by thermal power plant generates pollution and dumps a lot of green house gases to atmosphere. The nuclear power plants became threat for society if the area is earthquake prone. Moreover no technique has yet been developed to destroy the nuclear waste. Further nuclear fuels are also limited. The hydroelectric power plants are also not possible at every part of the world due to uneven distribution of water bodies. Thus the capture and conversion of solar energy into electricity is the only way of generation of non-polluting energy¹. This requires photovoltaic and photoelectrochemical cells. These cells consist of semiconducting materials. Production of cheap photovoltaic and photoelectrochemical cells is a constant preoccupation of semiconductor electrochemists²⁻⁵. In spite of this development the efficient photoelectrochemical conversion of practical interest has not yet been possible. Strategies devised to obtain semiconductor based system for sustained and efficient photoelectrochemical conversion has not led to any spectacular advancement⁶⁻⁸ specially WSe₂ metal chalcogenides is currently considered attractive because of its band gap matches adequately the solar spectral distribution. Some characteristics of this system has been investigated by other workers⁹⁻¹³.

In the present investigation, we have prepared titanium based WSe₂ by electrochemical codeposition technique under potentiostatics as well as galvanostatic control. The electrochemically deposited films were subjected to thermal treatment. These Ti-WSe₂ thin films were characterized by studying their capacitance characteristics and examination of current voltage characteristics in dark and under illumination of examination of these WSe₂ semiconductor thin films show that when they are prepared by galvanostatic control and the

subjected to thermal treatment. They exhibit acceptable activity.

Experimental:

For electrochemical synthesis of WSe₂ films, the working electrode consist of titanium foil (M/s. Titanium Equipment and Anode Manufacturing Co. Ltd., Madras) were cleaned with emery paper grade 1/0, polished with diamond lapping paste (Metses Fluid, Madras Metallurgical Service Pvt. Ltd.) and washed successively with acetone and distilled water. A three electrode system consisting of a working electrode, a titanium counter electrode and a saturated calomel electrode (SCE) was used for electrodeposition. For galvanostatic deposition, passage of constant current was ensured using constant current power source (Lake Shore 120, USA). During galvanostatic deposition variation in the potential of the working electrodes was monitored using SCE. Preparation of WSe₂ films was carried out using different constant current, density and different time intervals. Tungstic acid and SeO₂ (FlukaChemica, Switzerland) were used for the preparation of electroplating solutions. The electrodeposited thin films of CdSe₂ were tested for their photoresponsiveness using 1KW Tungsten lamp as a light-source. For photoaction spectral studies f/3.4 monochromator (Applied Photophysics, London) was used.

Results and discussion:

Preparation of solution of tungstic acid is a difficult task because of its insolubility in water. For preparation of electroplating solution 0.05M tungstic acid was dissolved in 100 ml of 10M ammonia solution. 0.02M SeO₂ dissolved in 100 ml of water was then added. The solution thus obtained was employed for studying current voltage behavior to ascertain the potential domain within which formation of tungsten diselenide was expected. The result is shown in Fig. 1. Some electrodeposited films within this domain were prepared under potentiostatic control and tested for their photoresponsiveness. These electrodeposited films even on thermal treatment did not exhibit attractive photoactivity.

Constant current method was then applied for the preparation of WSe₂. Various deposited films of WSe₂ was prepared using constant currents ranging from 1 mA to 6 mA. The films thus obtained were tested in various redox solutions to check the suitability of redox solutions. The electrolyte containing 1.9 M KI and 50 mM I₂ is found to be suitable. As deposited films does not exhibit acceptable photoactivity. However after thermal treatment these preparations showed somewhat impaired photoactivity.

During the deposition of WSe₂, the temperature at which electrolysis is carried out affects the quality of preparations. Exploratory studies show that a temperature of 60⁰ to 70⁰C yield, preparations endowed with improved photoresponsiveness.

Current voltage behavior of the semiconductor electrodes in dark and under illumination was studied to obtain information concerning the nature of semiconducting materials. These studies show that WSe₂ films exhibit p-type semiconductivity. But on thermal treatment, they becomes n-type This is evident from the current voltage characteristics depicted in Fig.2.

Light intensity dependence of photopotential has been examined at $\lambda = 436$ nm, the wavelength which generates maximum photoeffect. The photocurrent increases with increase in light intensities The result is presented in Fig.3. However a linear relationship exists between photopotential and Log_e(Light intensity). This result is presented in Fig.4. This behavior indicates semiconducting nature of the electrodeposits¹⁴. The data are also consistent with equation.

$$E_p = (AKT/e)\ln I_L + B$$

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In this equation A is ideality factor, I_L denotes light intensity.

Examination of these results reveals that the WSe₂ is endowed with an ideality factor \approx 1.37 which shows the preparation is not ideal. For ideal systems, this factor should be unity¹⁵.

WSe₂ electrodeposits are likely to be susceptible to corrosion. With a view to ascertain ability of this semiconductor to withstand photocorrosion The deposited films weresubjected to uninterrupted illumination in electrolyte solution. Results presented in Fig.5 show that the electrodeposits are resistant towards photocorrosion in substantial measure. These electrodeposited WSe₂ films were first equilibrates in CH₃CN solution for 24 hours and then subjected to intermittent illumination in the same solution for about two hours. Photoaction spectral studies of thermally treated WSe₂ films were carried out to determine the band gap of the electrodeposits. Results show that band gap of WSe₂ was about 1.58 eV.

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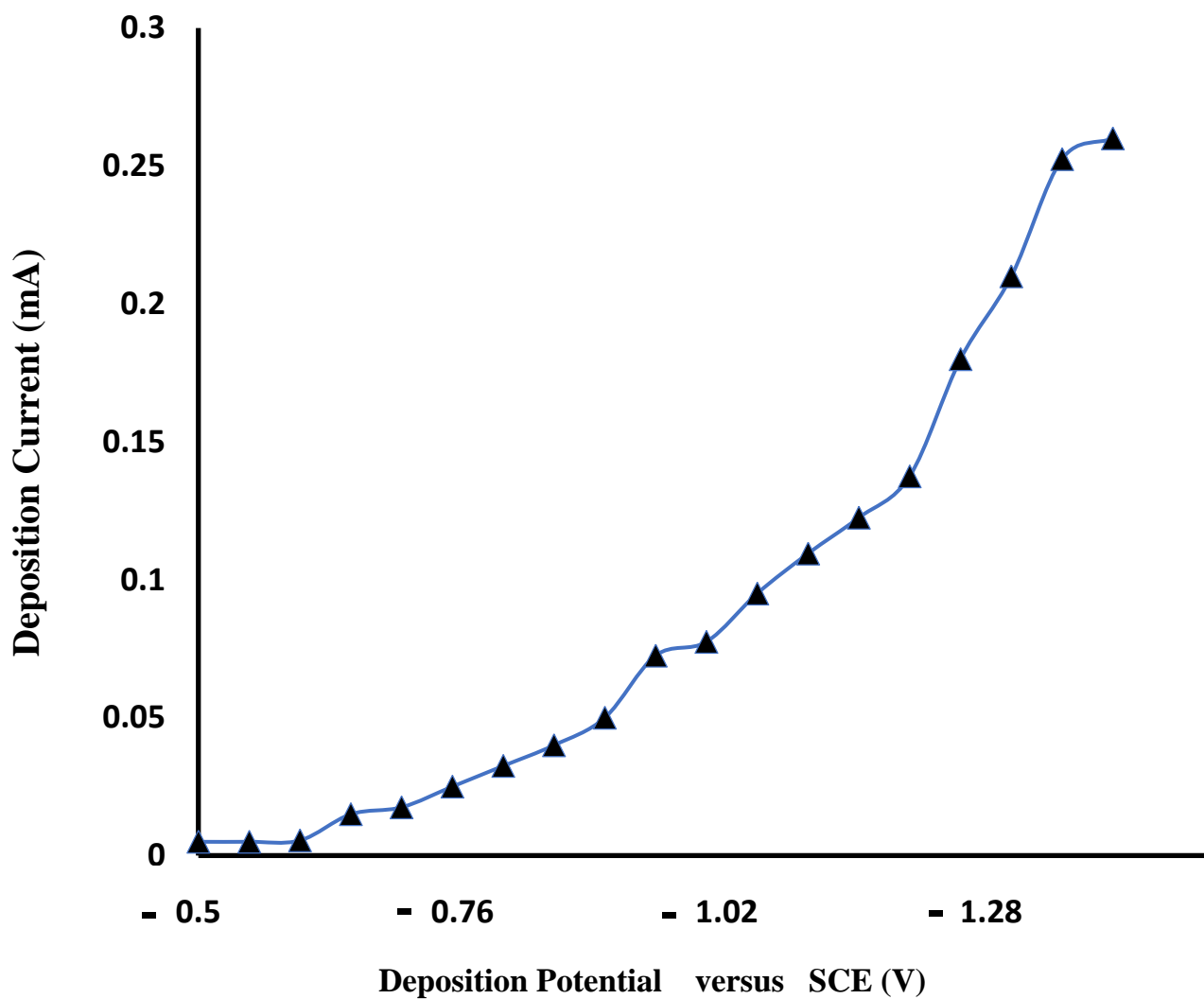


Fig. 1 Current voltage behavior in electroplating solution containing 0.01M sodium tungstate and 0.01M selenium dioxide

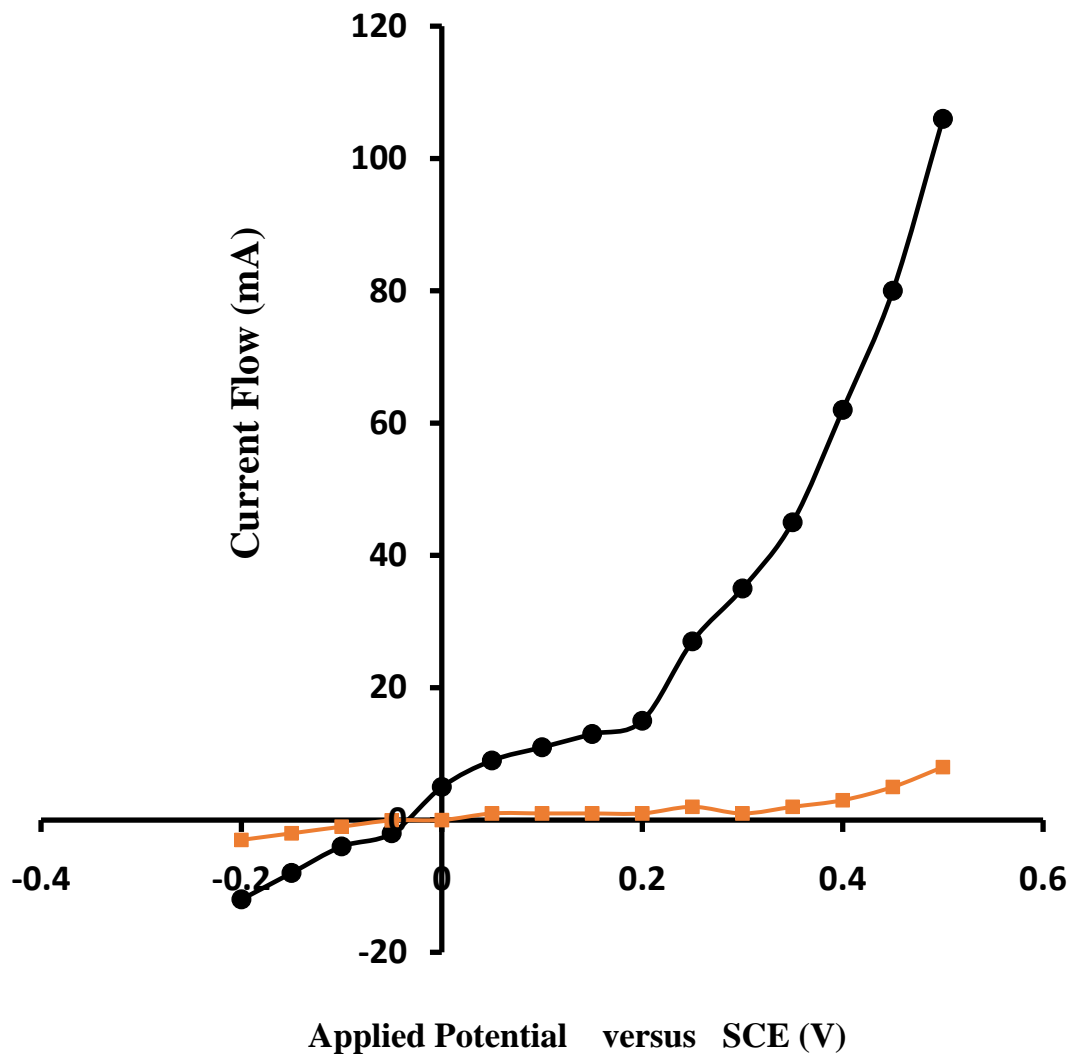


Fig. 2 Current voltage characteristic of thermally treated n- WSe₂ in dark (---□---) and under illumination (---●---)

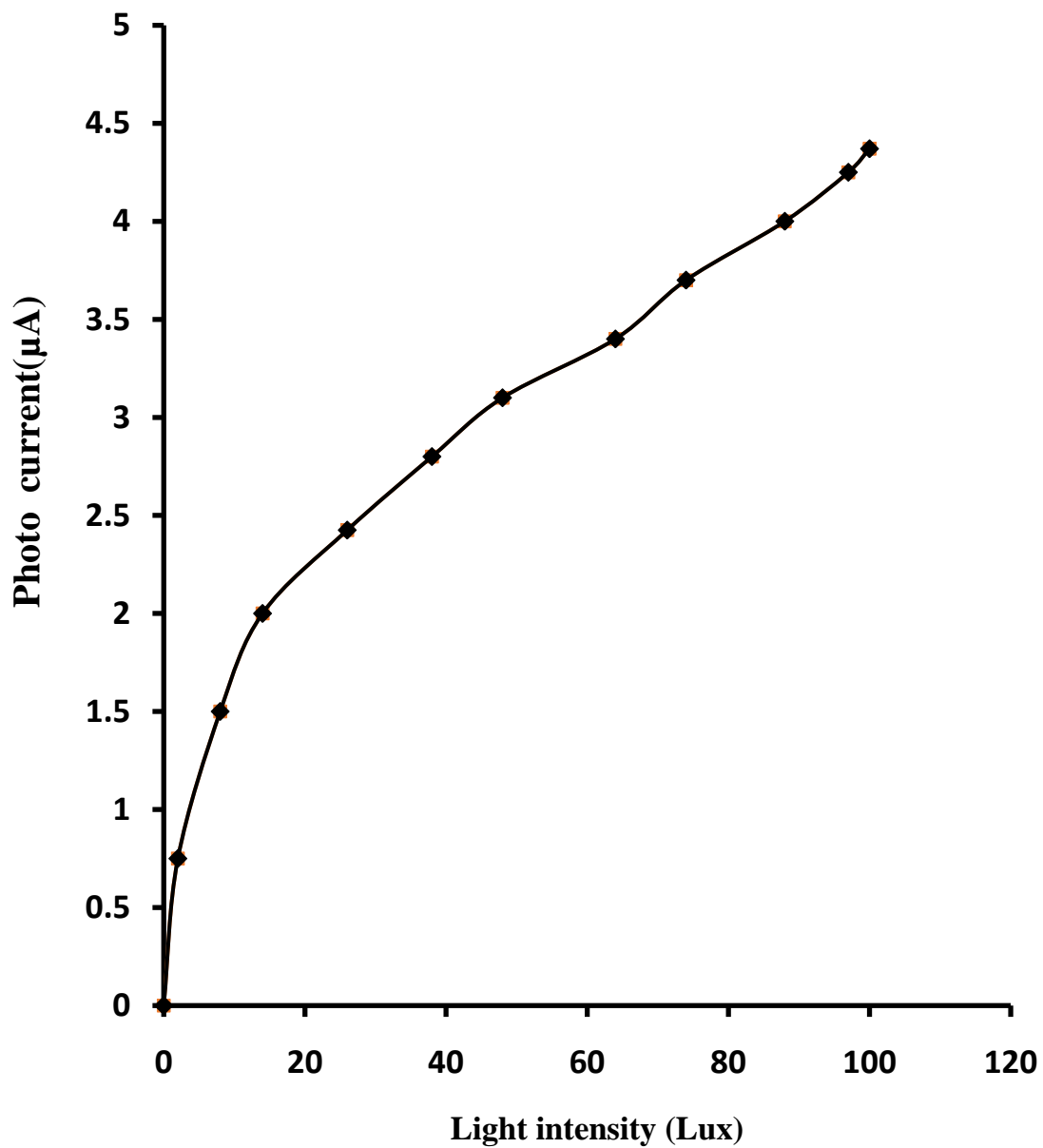


Fig. 3 Light intensity dependence on photo current for WSe₂ at $\lambda = 436$ nm

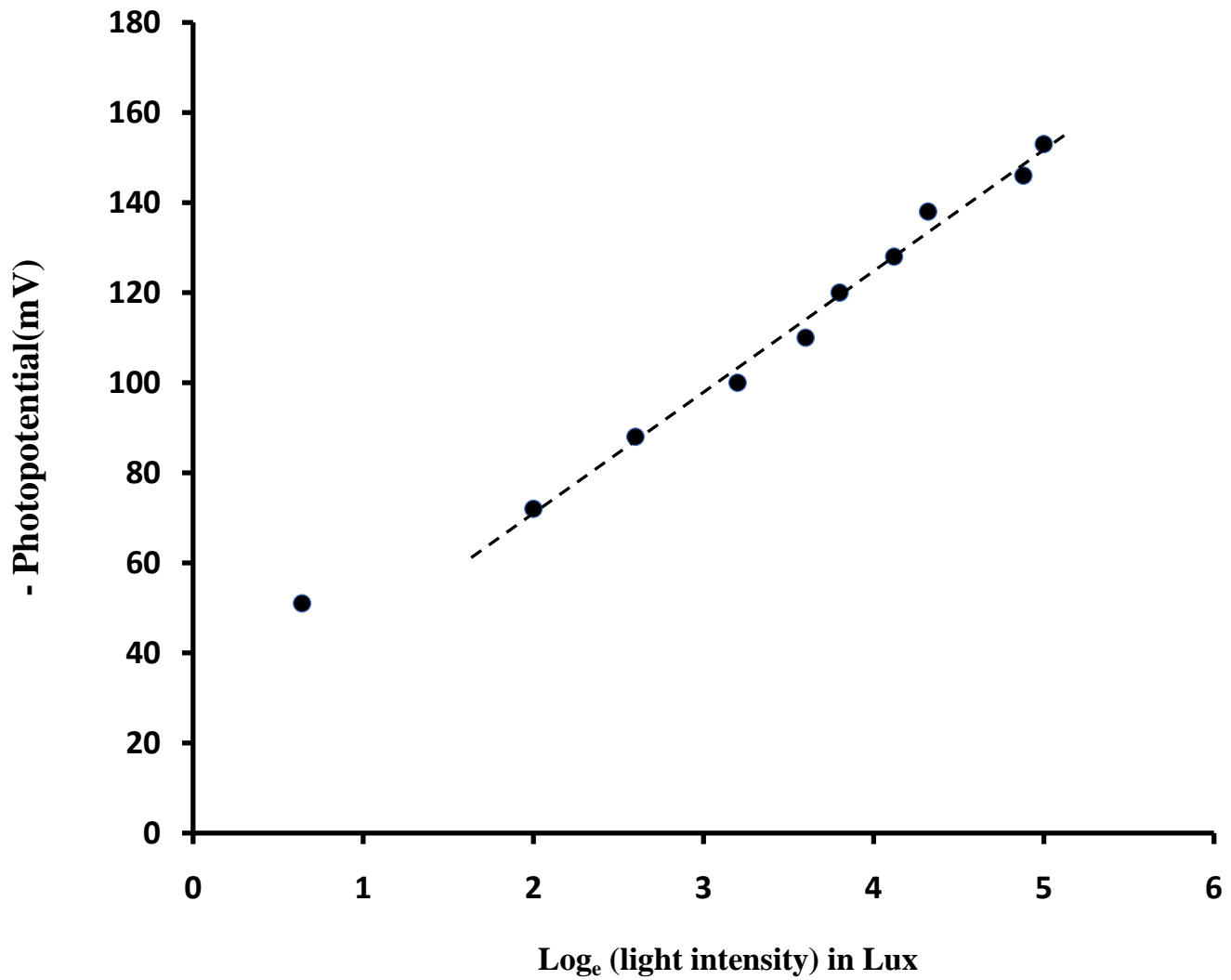


Fig. 4 Variation of log_e (Light intensity) with the photopotential obtained for WSe₂

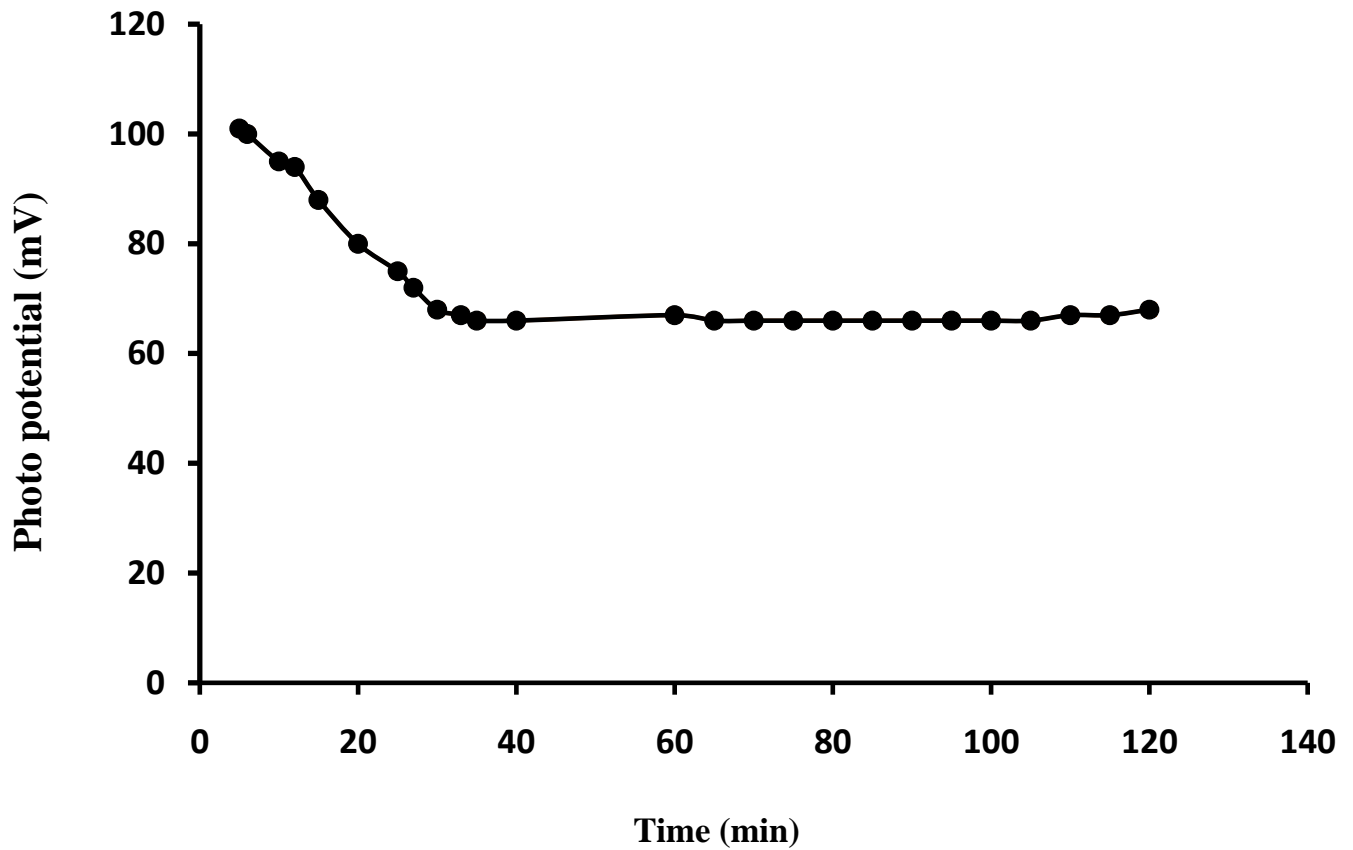


Fig.5 Photoactivity exhibited by a typical WSe₂ in redox solution on intermittent illumination