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# Electrochemical supercapacitor properties of highly porous sponge-like selenium thin films

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## ABSTRACT

The porous nanostructured material is a prime requirement of energy storage devices, as it contributes maximum surface area for charge storage. In present paper, a simple and cost effective electrodeposition route has been adopted to prepare highly porous sponge-like selenium thin films. The selenium surface displays porous nanostructure with specific surface area of  $35.9 \text{ m}^2 \text{ g}^{-1}$  and suitable pore size, providing auspicious trails for transportation as well as penetration of electrolyte ions. The structural study confirms the formation of trigonal structure. The electrochemical properties of selenium films are reported. The film exhibits maximum specific capacitance ( $C_s$ ) of  $29.25 \text{ F g}^{-1}$  at  $5 \text{ mV s}^{-1}$  scan rate in  $1 \text{ M Na}_2\text{SO}_4$  electrolyte along with electrochemical stability of 91% after 1000 cyclic voltammetry (CV) cycles. This investigation implies that selenium thin films in composite form may be useful for charge storage applications.

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## Introduction

Selenium is a non-metal with intermediate properties between chalcogen of sulfur and tellurium. It exhibits important applications such as manganese electrolysis as the electro-winning of manganese in electrolysis cells [1], alloys making with bismuth in brasses to replace more toxic lead [2] and as a solar cell absorber material in copper indium gallium selenide ( $\text{CuIn}_{1-x}\text{Ga}_x\text{Se}$ ) [3]. The small amount of organo-selenium is used to modify the vulcanization catalysts in the production of rubber [4]. Because of photovoltaic and photoconductive properties of selenium, it has been used in photocopying application [5]. Selenium is also widely used in selenium

rectifiers and power DC surge protection, where the larger energy capabilities of selenium suppressors create them more appropriate than metal oxide varistors. The zinc selenide is first time used for blue LED fabrication and cadmium selenide has played an important role in the fabrication of quantum dots [6,7]. The sheets of amorphous selenium alter X-ray images to patterns of charge in xeroradiography, crystallography and single wavelength anomalous dispersion phasing [8,9]. The photo conducting properties of selenium thin film are studied by Serdouk and Benkhedir [10] using Laplace-transform method.

Insulator consisting of  $\text{Se}_8$  rings having amorphous (a-Se) or red selenium, polymeric rings having vitreous or black selenium (v-Se) and trigonal gray selenium (t-Se) are three

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distinct states of selenium [11]. Aal et al. [12] prepared macroporous selenium thin films by electrodeposition route. Lai et al. [13] reported selenium electrodeposition onto SnO<sub>2</sub> coated glass substrate. The voltammetric electrochemical behavior of electrodeposited selenium (IV) material on gold and platinum electrodes in sulfuric acid, perchloric acid and potassium chloride media is investigated by Beni et al. [14]. Also, Maranowski et al. [15] studied electrochemical properties of electrodeposited selenium. Zeng et al. [16] prepared carbon-selenium composite nanofibers with superior electrochemical performance for Li–Se/Na–Se batteries. Luo et al. [17] studied electrochemical properties of selenium/mesoporous carbon microsphere composites for battery and achieved an initial capacity of 485 mAh g<sup>-1</sup>. Patil et al. [18] prepared selenium thin films by simple chemical bath deposition method for supercapacitor application. This work reported the specific capacitance of 21.98 F g<sup>-1</sup> with specific surface area of 30.5 m<sup>2</sup> g<sup>-1</sup> for selenium electrode material. Literature survey shows that there is no more work on electrochemical supercapacitor properties of electrodeposited selenium thin films. Additional to capacitors, modified ultracapacitors or supercapacitors (SCs) generated enhancing power density and electrical storing capacity. Inexpensiveness of SCs individually depends on method of electrode preparation, availability of electrode material and device fabrication cost [19,20]. The charge storing mechanism of SCs at an interface of electrode-electrolyte, divides SCs into two types as electrochemical double layer capacitors (EDLCs) and pseudocapacitors, while combination of both is a hybrid capacitor [21,22]. The storage of charge without faradaic charge transfer reaction between conductor electrode and electrolyte solution over the potential range of operation is denoted as EDLCs, and Helmholtz double layer is developed due to charge accumulation [23]. In EDLCs, carbon materials such as graphene or carbon aerogel are used as electrode material [24–27]. Metal oxide, metal sulfide as well as conducting polymers (polyaniline and polypyrrole) are pseudocapacitive electrode materials which undergo redox reactions to store the charges [28,29].

In present work, selenium thin films are deposited on stainless steel (SS) substrate using potentiostatic mode of electrodeposition at room temperature (300 K). Selenium thin films are characterized by X-ray diffraction (XRD), Raman spectroscopy, contact angle measurement, field emission scanning electron microscopy (FE-SEM), energy dispersive spectroscopy (EDS), atomic force microscopy (AFM) and Brunauer, Emmett and Teller (BET) techniques. Further, electrochemical storage properties such as cyclic voltammetry (CV), galvanostatic charge discharge (GCD) and electrochemical impedance spectroscopy (EIS) are investigated using battery cyler and electrochemical workstation.

## Experimental

The analytical grade selenium dioxide (SeO<sub>2</sub>, 99%) is used without further purification. The SS substrate (5 × 1 cm<sup>2</sup>) was polished with zero grade polish paper, and washed with detergent in double distilled water (DDW). Initially, 0.1 M SeO<sub>2</sub>

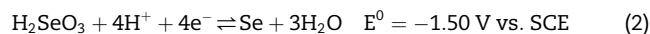
powder was dissolved in 30 ml DDW by continuous stirring. The potentiostatic electrodeposition (–1.5 V/SCE) was carried out using three electrode system consisting of saturated calomel electrode (SCE) as a reference electrode, graphite as a counter electrode and SS substrate as a working electrode. Within 15 min, the thin selenium film is deposited on SS substrate. The electrodeposition time is major factor affecting on thickness of selenium thin film. Red-brown colored selenium thin film of thickness 750 nm was developed on SS substrate.

## Material characterization

The X-ray diffraction (XRD) technique was employed for crystal structure determination of selenium thin film using Bruker AXS D8 advanced Model with copper radiation (K<sub>α</sub> of wavelength = 1.54 Å) unit. Raman spectrometer (Bruker MultiRAM, Germany Make) supported with Nd:YAG laser source was used to phase confirmation. The surface morphological properties and composition were investigated by FE-SEM and EDS techniques (JEOL JSM 6390). AFM, Model-INNOVA 1B3BE unit was employed for three dimensional profile of film surface. Rame Hart instrument with drop image advanced software unit was used for wettability test. The specific surface area of electrodeposited selenium material was calculated by BET model Quantachrome Instruments v11.02. The thickness of selenium thin film was measured using XP-1 Stylus surface profiler unit. The electrochemical properties such as CV and GCD were carried out using automatic battery cyler unit (WBCS3000). The electrochemical behavior of selenium thin film was evaluated by electrochemical workstation (ZIVE SP5).

## Results and discussion

The deposition of selenium thin film was carried out using potentiostatic mode of electrodeposition. The discriminating reduction and deposition of selenium in an acidic medium resulted as development of thin film on SS substrate. The aqueous solution of 0.1 M SeO<sub>2</sub> forms selenious acid (H<sub>2</sub>SeO<sub>3</sub>). The pH of precursor solution is about 4.3 (±0.1). Further, applying negative potential (–1.5 V/SCE) to SS electrode due to reduction process, selenium film is deposited on SS substrate [30]. This process of selenium deposition according to the reactions looks like the four-electron reduction when the concentration of H<sub>2</sub>SeO<sub>3</sub> is high enough [31].



The selenium thin film was red-brown colored as well as adherent to SS substrate. However, the color of selenium thin film was changed to gray-black due to aging, denoting formation of thermodynamically more stable hexagonal state of selenium [32].

The structural analysis was carried out to confirm the phase formation in selenium thin film. Fig. 1(A) shows XRD pattern of selenium thin film on SS substrate. The high

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