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To cite this article: C Prabukumar *et al* 2019 *Mater. Res. Express* **6** 085526

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Materials Research Express



PAPER

SnO₂ nanoparticles functionalized MoS₂ nanosheets as the electrode material for supercapacitor applications

RECEIVED
31 December 2018

REVISED
18 April 2019

ACCEPTED FOR PUBLICATION
16 May 2019

PUBLISHED
22 May 2019

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Keywords: supercapacitor, MoS₂ nanosheets, SnO₂ nanoparticles, nanocomposite, ligand exchange process

Abstract

Tin oxide (SnO₂) nanoparticles undergo the volume expansion during an electrochemical cycle. This volume expansion leads to discontinuities in the form of microcracks in the electrode material. The problem of charge transportation associated with this microcracking limits the application of SnO₂ in the energy storage application such as supercapacitors. The present work approached to solve this problem by incorporating the MoS₂ nanosheets along with the SnO₂ nanoparticles. The SnO₂ nanoparticles are functionalized onto the surface of the MoS₂ nanosheets by the ligand exchange process. The MoS₂ nanosheets act as the support material for the SnO₂ nanoparticles. The electrode material prepared using SnO₂ nanoparticles and nanocomposite of SnO₂ functionalized MoS₂ nanosheets are tested by cyclic voltammetry and galvanostatic charge-discharge measurements. The specific capacity of the MoS₂-SnO₂ nanocomposite is calculated to be 61.6 F g⁻¹ which is 4.4 fold higher than that of bare SnO₂ nanoparticles. The improvement in the electrochemical performance of SnO₂ is attributed to the high surface area and the charge transportation provided by the MoS₂ nanosheets.

1. Introduction

In recent years, most of the researches are focussed on to improve the performance of the energy generation and storage devices. The supercapacitors are being evolved as a promising energy storage device for the future. The supercapacitors possess some superior qualities than their peers, namely batteries. Unlike batteries, the charging and discharging process is faster for the supercapacitors. With the higher power density and longer life time than the batteries [1], the supercapacitors will play a major role in the field of energy storage in the coming years. Recently, the metals oxides (Sn, Mn, Ti, Co) are being explored for such applications [2–5]. Tin oxide (SnO₂) is a potential electrode material in energy storage applications, due to its low cost, high theoretical capacity and high electron mobility. However, there is a concern associated with the SnO₂ electrode that needs to be addressed. The volume expansion of the SnO₂ particles during the electrochemical process hinders the performance of the SnO₂ electrode [6]. Charge transfer in the SnO₂ electrode is affected due to the discontinuity caused by the volume expansion [7]. This results in reduction in the capacitance value of the SnO₂ electrode. To address this issue, the two-dimensional materials like graphene are used along with SnO₂ nanoparticles to facilitate charge transportation [8].

Molybdenum disulfide (MoS₂) is a material having fascinating properties, like, high surface area, higher ionic conductivity than metal oxides [9] and good mechanical flexibility [10]. It is widely used in gas sensors, supercapacitors, batteries, hydrogen evolution reactions and electronic applications [11–15]. The MoS₂ nanosheets are synthesised either by bottom-up approaches, such as hydrothermal, chemical vapour deposition (CVD) or by top-down approaches, like, ball milling, mechanical exfoliation and liquid phase exfoliation [12, 16–19]. Among them, liquid phase exfoliation is the simple and high yielding route to prepare the MoS₂ nanosheets. The solvents such as ethanol, dimethylformamide (DMF), N-Methyl-2-pyrrolidone (NMP) are

used to exfoliate the bulk MoS₂ particles into nanosheets [19, 20]. The MoS₂ nanosheets are proved to improve the electrochemical performance of materials like Co₃O₄, polyethylene dioxythiophene (PEDOT), polyaniline (PANI) and Mn₃O₄ [5, 21–23]. So, it is presumed that the electrochemical stability of the SnO₂ could be improved by using MoS₂ nanosheets along with the SnO₂ nanoparticles.

There are very limited studies reported focussing on the supercapacitor application with the combination of MoS₂–SnO₂ phases as nanocomposite [24]. The hydrothermal method is the widely used technique to prepare MoS₂–SnO₂ nanocomposite [25]. But it requires the temperature as high as 220 °C to produce nanosheets of MoS₂. In the present work, we have used a ligand exchange process to prepare MoS₂–SnO₂ nanocomposite. Here, the SnO₂ nanoparticles are functionalized on to the surface of the MoS₂ nanosheets at room temperature. The route is expected to be energy saving and producing the MoS₂–SnO₂ nanocomposite which will serve as a good supercapacitor electrode material.

2. Experimental details

2.1. Synthesis of MoS₂ nanosheets

The MoS₂ nanosheets were prepared by ultrasonication-assisted liquid phase exfoliation method [26]. All the chemicals are of analytical grade purchased from reputed manufacturers. Firstly, 150 mg of polyvinylpyrrolidone (PVP, MW ~ 40,000) was dissolved in 10 ml ethanol. Then, 30 mg of bulk MoS₂ powder (Alfa Aesar, 325 mesh) was added into the prepared PVP solution. The mixture of MoS₂ and PVP in ethanol was ultrasonicated (Power = 50 W, f = 33 kHz) for 4 h to exfoliate MoS₂ nanosheets. After the exfoliation process, the exfoliated MoS₂ sheets which remained on top of the solution were transferred to the centrifugal tubes for washing. The product was mixed with acetone and centrifuged at 7000 rpm for 20 min. After removing the supernatant, isopropyl alcohol (IPA) was added to the precipitate. The product was again centrifuged at 8000 rpm for 20 min. Finally, the washed product was collected and suspended in IPA for storing.

2.2. Synthesis of SnO₂ nanoparticles

The SnO₂ nanoparticles were synthesised by following the work reported in the literature [27]. First, 1 g of tin chloride pentahydrate (SnCl₄.5H₂O) was dissolved in 10 ml of ethylene glycol (EG). It was followed by the addition of 250 mg of ammonium chloride, 250 mg of ammonium acetate and 30 ml de-mineralized (DM) water in a round bottom flask. Then, the flask was heated to 90 °C in the oil bath for 2 h. The synthesised product was washed by DM water and ethanol. Then it was dispersed in ethanol.

2.3. Functionalization of MoS₂ nanosheets with SnO₂ nanoparticles

The known density of MoS₂ nanosheets dispersion was added into a vial containing the known weight of SnO₂ nanoparticles dispersion. Then the two materials were well dispersed together by using ultrasonication bath. After the ultrasonication, the materials were allowed to get settled down at the bottom of the vial. Then the supernatant liquid was removed and the materials settled at the bottom were used for our study. Likewise, five different compositions of MoS₂–SnO₂ nanocomposites were prepared with varying the weight ratio of MoS₂ nanosheets (0.5%, 1%, 2.5%, 5% and 10%) to SnO₂ nanoparticles. The camera images of each step involved in this preparation process are shown in figure 1.

2.4. Materials characterization

The structural characterization and phase identification were performed by using x-ray diffractometer (XRD; JEOL, JDX 8) with Cu K_α (λ = 0.154 nm) radiation at the scan rate of 1° min⁻¹. The morphological features were studied by using field emission scanning electron microscope (FESEM; Carl Zeiss Sigma) and transmission electron microscope (TEM; JEOL, JEM-2100). The Raman spectra of bulk and exfoliated MoS₂ were obtained by using Raman spectrometer (Horiba Jobin-Yvon, labRAM HR) with an excitation wavelength of 532 nm. The surface chemistry of the materials was analysed by using x-ray photoelectron spectrometer (XPS; Thermo scientific, Multilab 2000) instrument (Mg K_α x-ray, 200 W as excitation source). The particle size of SnO₂ nanoparticles was determined by using particle size analyser (Malvern Zeta Sizer, NanoZS). The Specific surface area and pore size distribution were analysed by Brunauer–Emmett–Teller (BET) and Barrett–Joyner–Halenda (BJH) method using surface area analyser (Microtrac, BELSORP Max).

2.5. Electrochemical studies

Electrochemical studies were carried out with a standard three-electrode cell using SP-150 electrochemical workstation (BioLogic, France). Platinum wire and saturated calomel electrode (SCE) were used as the counter electrode and reference electrode, respectively. The working electrode was prepared as follows: 4.5 mg of nanocomposite was added into 250 μl water/ethanol (2:1 v/v) solution containing 15 μl Nafion (5 wt%). The

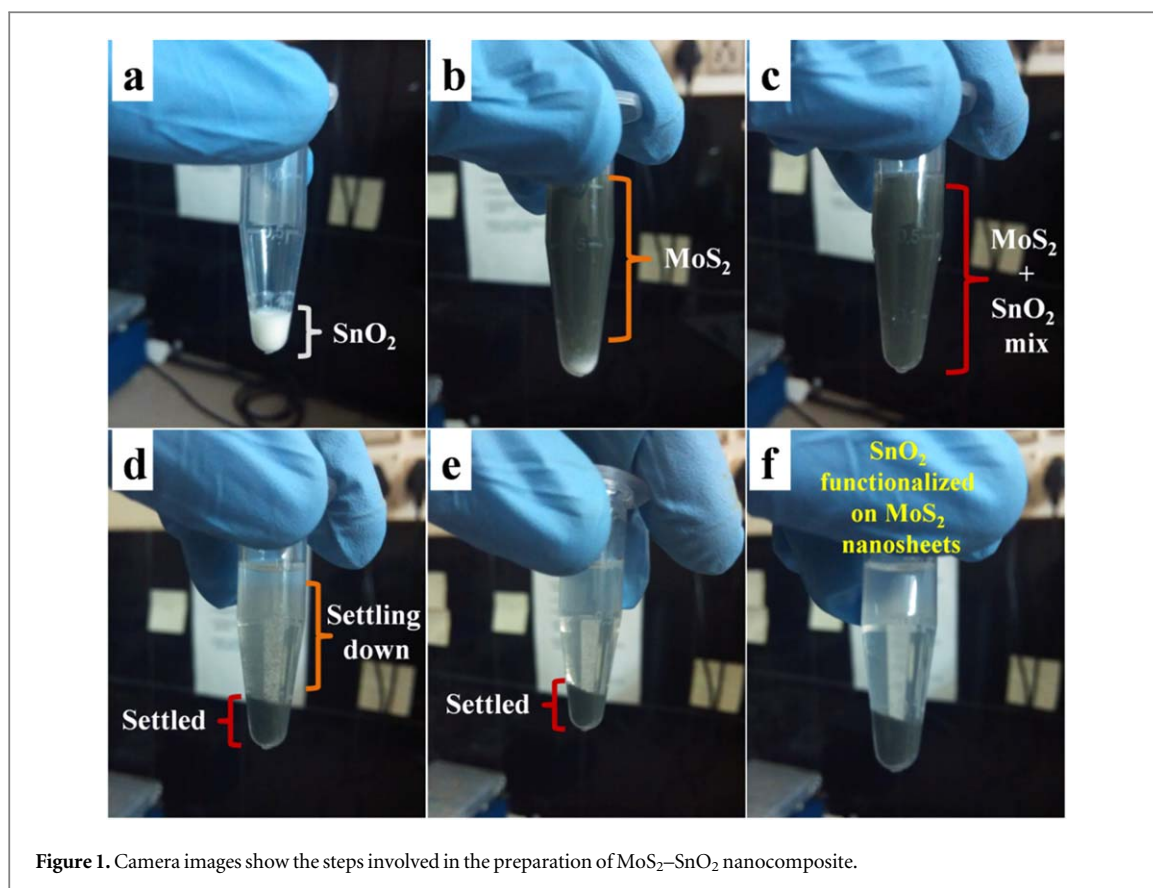


Figure 1. Camera images show the steps involved in the preparation of MoS₂-SnO₂ nanocomposite.

above mixture was ultrasonicated for 15 min to obtain a homogeneous mixture. 50 μl of the prepared mixture was drop cast on a graphite electrode (10 mm diameter rod). The calculated mass of the active electrode material was 1.1 mg cm^{-2} . The aqueous solution of 2 M KOH was used as the electrolyte.

3. Results and discussion

Figures 2(a), (b) show the FESEM micrographs of the bulk MoS₂ powder and exfoliated MoS₂ nanosheets. The bulk powder consisted of several layers of the MoS₂ stacked one above the other. The FESEM micrograph of exfoliated nanosheets revealed that the bulk MoS₂ powder was exfoliated into nanosheets. The synthesised SnO₂ nanoparticles (figure 2(c)) showed the spherical-like morphology with size in the range of 10–35 nm. Figure 2(d) shows the FESEM micrograph of the MoS₂-SnO₂ nanocomposite prepared by the ligand exchange process. The SnO₂ nanoparticles were functionalized over the surface of the MoS₂ nanosheets. The SnO₂ nanoparticles were firmly attached on to the surface of the MoS₂ nanosheets. Figure 3 shows the schematic diagram explaining the functionalization of MoS₂ nanosheets with SnO₂ nanoparticles by the ligand exchange process.

A thin layer of PVP molecules, from the exfoliation step, would still present onto the surface of the MoS₂ nanosheets even after washing process because of C-S and O-S bonding between PVP and MoS₂ [28]. Also, this PVP ligand attached with MoS₂ nanosheets kept nanosheets separated from each other. On the other hand, ammonium ions from ammonium salts during the synthesis process coordinated with the SnO₂ nanoparticles. These ammonium ions act as the surfactant to control the growth of the nanoparticles [27]. When the MoS₂ nanosheets and SnO₂ nanoparticles were mixed together (figures 1(b), (c)), the ammonium ions attached with the SnO₂ nanoparticles strip off the PVP molecules from the surface of the MoS₂ nanosheets. Subsequently, the vacant sites left by the stripped PVP molecules were occupied by SnO₂ nanoparticles. The ammonium ions present over the SnO₂ nanoparticles would help them to anchor onto the surface of the MoS₂ nanosheets [27]. Likewise, the SnO₂ nanoparticles were decorated or functionalized on the surface of the MoS₂ nanosheets. This functionalization process by exchanging the ligands is energy efficient compared to hydrothermal which involves high temperature and pressure [16].

Once the PVP molecules were replaced by SnO₂ nanoparticles from the MoS₂ surface, the MoS₂ nanosheets functionalized with SnO₂ nanoparticles were started to settle down as the precipitate (figures 1(e), (f)). The supernatant containing these PVP molecules was removed out and the obtained precipitate was used for our present studies.

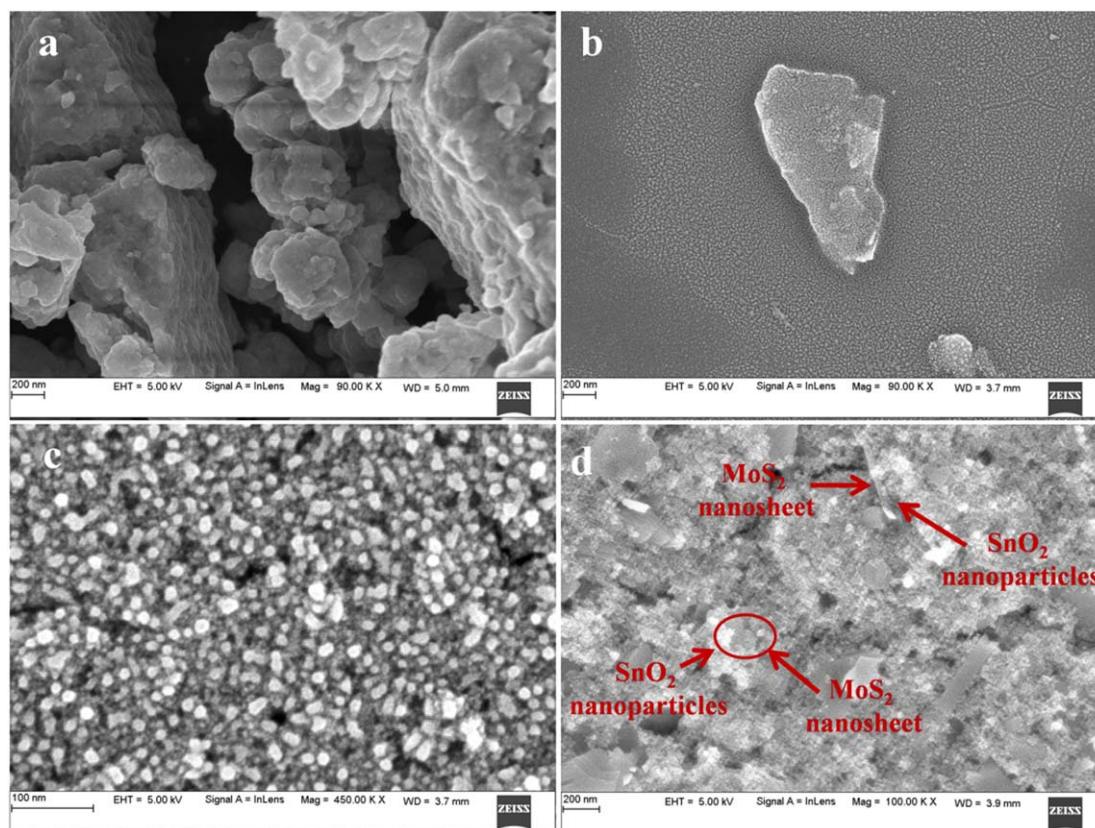


Figure 2. SEM micrograph of the (a) bulk MoS₂ powder, (b) exfoliated MoS₂ nanosheets, (c) synthesised SnO₂ nanoparticles, (d) MoS₂-SnO₂ nanocomposite.

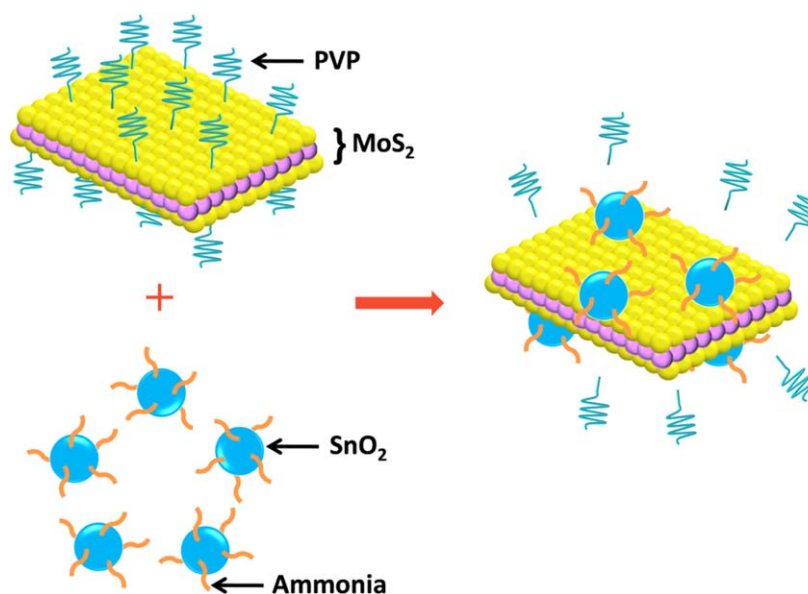


Figure 3. Schematic diagram explains the functionalization of MoS₂ nanosheets with SnO₂ nanoparticles.

Figure 4(a) shows the TEM micrograph of the exfoliated MoS₂ nanosheets. The exfoliated nanosheets contain only a few layers of MoS₂. The lattice distance of 0.27 nm is measured from the high-resolution TEM image shown in figure 4(b). It is attributed to (100) plane of MoS₂. The SAED pattern (inset figure 4(a)) revealed the hexagonal lattice structure of the MoS₂ nanosheets. The TEM micrograph of the synthesized SnO₂ nanoparticles showed in figure 4(c) affirmed the spherical-like morphology of the synthesised product. The lattice distances (interplanar spacing) in SnO₂ nanoparticles were measured to be 0.34 nm and 0.26 nm. These

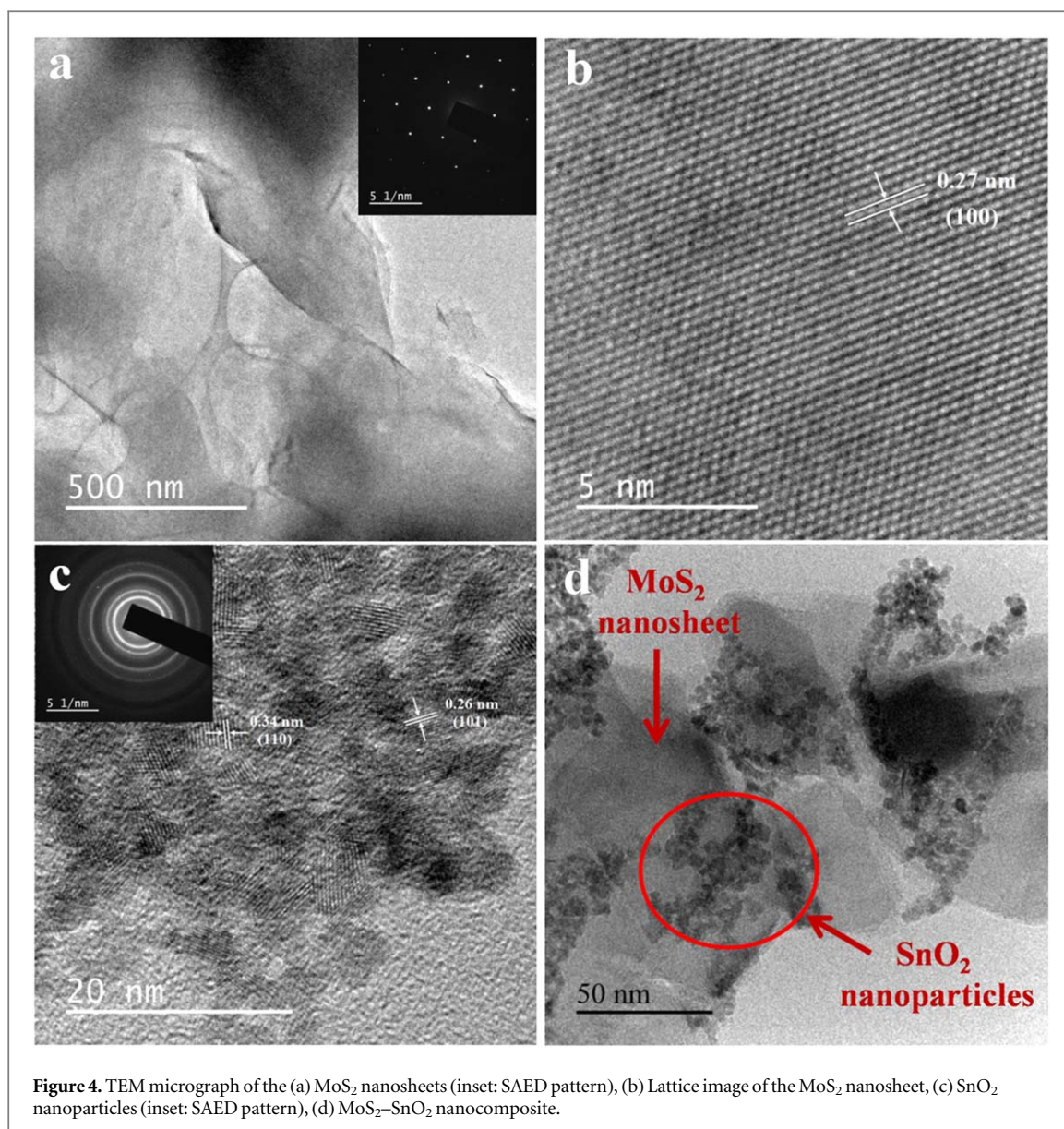


Figure 4. TEM micrograph of the (a) MoS₂ nanosheets (inset: SAED pattern), (b) Lattice image of the MoS₂ nanosheet, (c) SnO₂ nanoparticles (inset: SAED pattern), (d) MoS₂-SnO₂ nanocomposite.

values were well matched to (101) and (110) planes of the SnO₂, respectively. Figure 4(d) shows the TEM micrograph of the MoS₂-SnO₂ nanocomposite. The SnO₂ nanoparticles can be seen functionalized on the surface of the MoS₂ nanosheets.

The XRD patterns of bulk MoS₂ powder and exfoliated MoS₂ nanosheets are shown in figure 5(a). The XRD pattern of both materials belongs to the Molybdenite-2H phase (JCPDS #006-0097). The maximum intensity was observed for the peak belonged to (002) plane for both materials. But the peaks of (103) and (105) planes appeared for the bulk powder were absent for the exfoliated nanosheets. Also, the peak of (002) plane was broader for the exfoliated nanosheets compared to the bulk powder. This indicates the reduction in the thickness of the MoS₂ nanosheets after the exfoliation process. Figure 5(b) shows the XRD patterns of SnO₂ nanoparticles and MoS₂-SnO₂ nanocomposite along with MoS₂ nanosheets. Three peaks were observed at the angle of 26.2°, 33.8° and 51.2° for SnO₂ nanoparticles which are attributed to (110), (101) and (211) planes of SnO₂ (Cassiterite, JCPDS #041-1445). The XRD pattern of MoS₂-SnO₂ nanocomposite exhibited the peaks of both SnO₂ and MoS₂ phases.

The Raman spectra of bulk MoS₂ and MoS₂ nanosheets are shown in the figure 6. The characteristic Raman vibration peaks of E_{2g}¹ and A_{1g} for bulk MoS₂ are observed at 383 cm⁻¹ and 407 cm⁻¹, respectively [29]. When thickness of the MoS₂ sheet decreases, the E_{2g}¹ peak belonging to in-plane sulfur-molybdenum vibration shifts to higher frequency, whereas, the A_{1g} peak belonging to out-plane sulphur vibration shifts to lower frequency [29]. The exfoliated MoS₂ nanosheets exhibited E_{2g}¹ and A_{1g} peaks at 384 cm⁻¹ and 405.5 cm⁻¹, respectively. The frequency gap between two Raman peaks of the exfoliated MoS₂ nanosheets was reduced to 21.5 cm⁻¹ from 24 cm⁻¹. This is the indication that the bulk MoS₂ was exfoliated to nanosheets with few layers thickness [30].

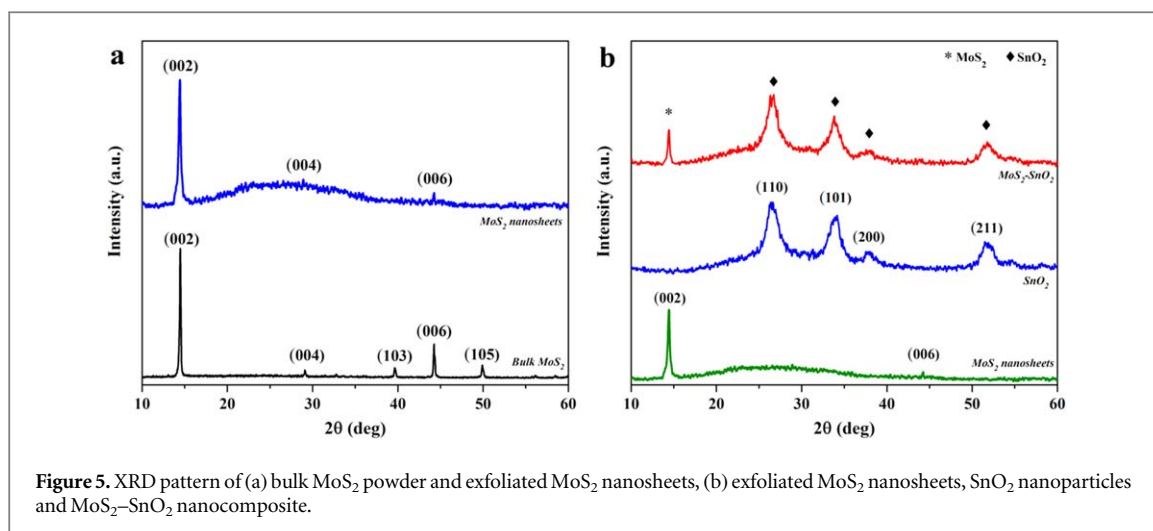


Figure 5. XRD pattern of (a) bulk MoS_2 powder and exfoliated MoS_2 nanosheets, (b) exfoliated MoS_2 nanosheets, SnO_2 nanoparticles and MoS_2 - SnO_2 nanocomposite.

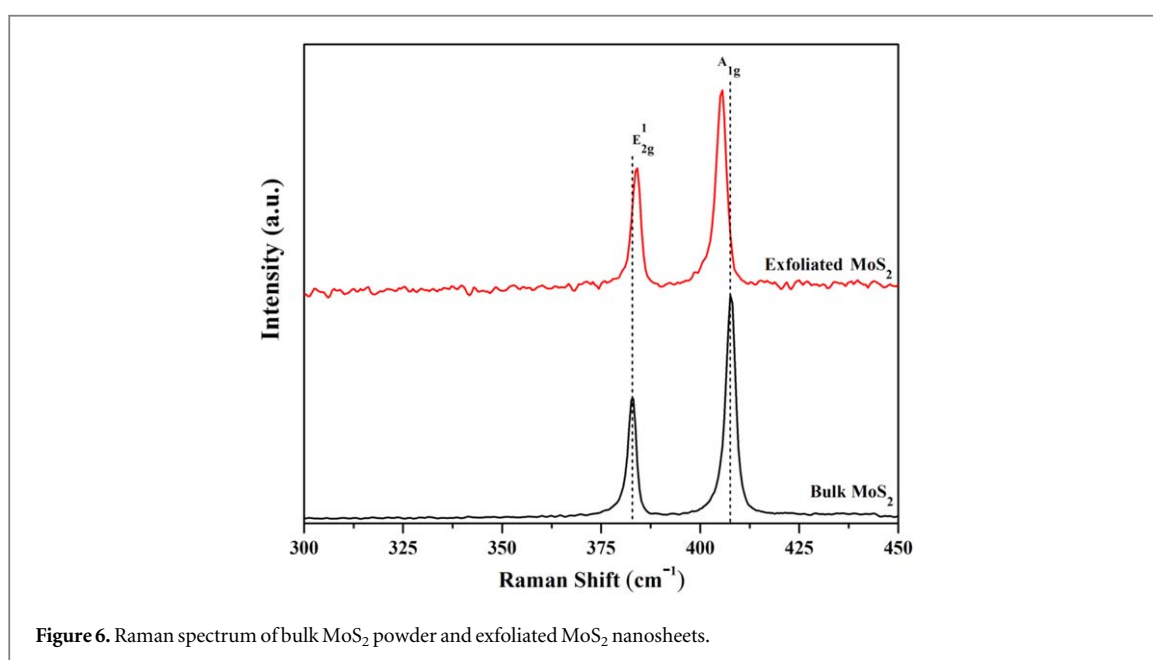
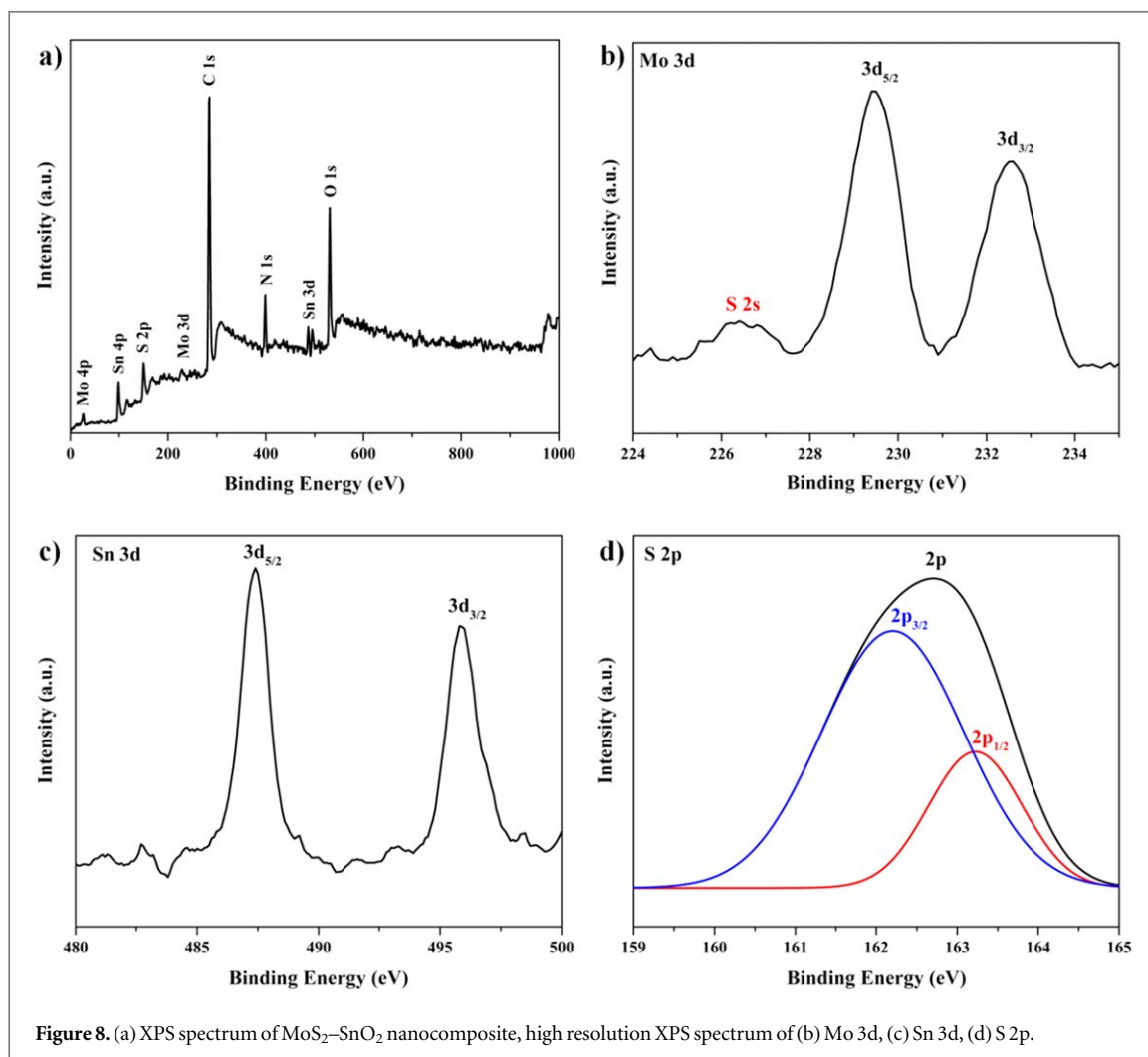
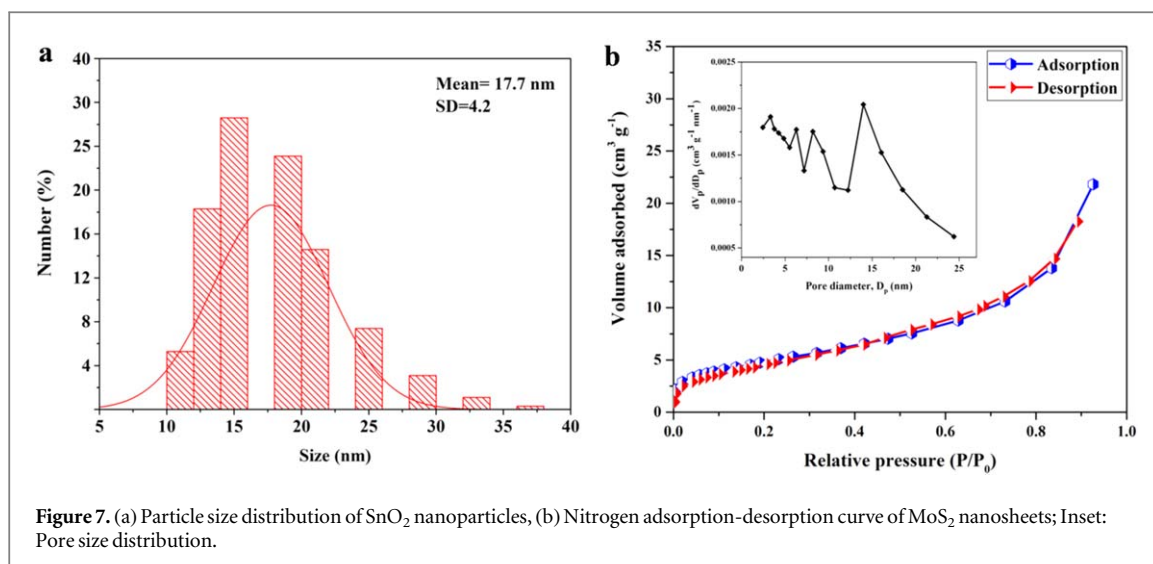


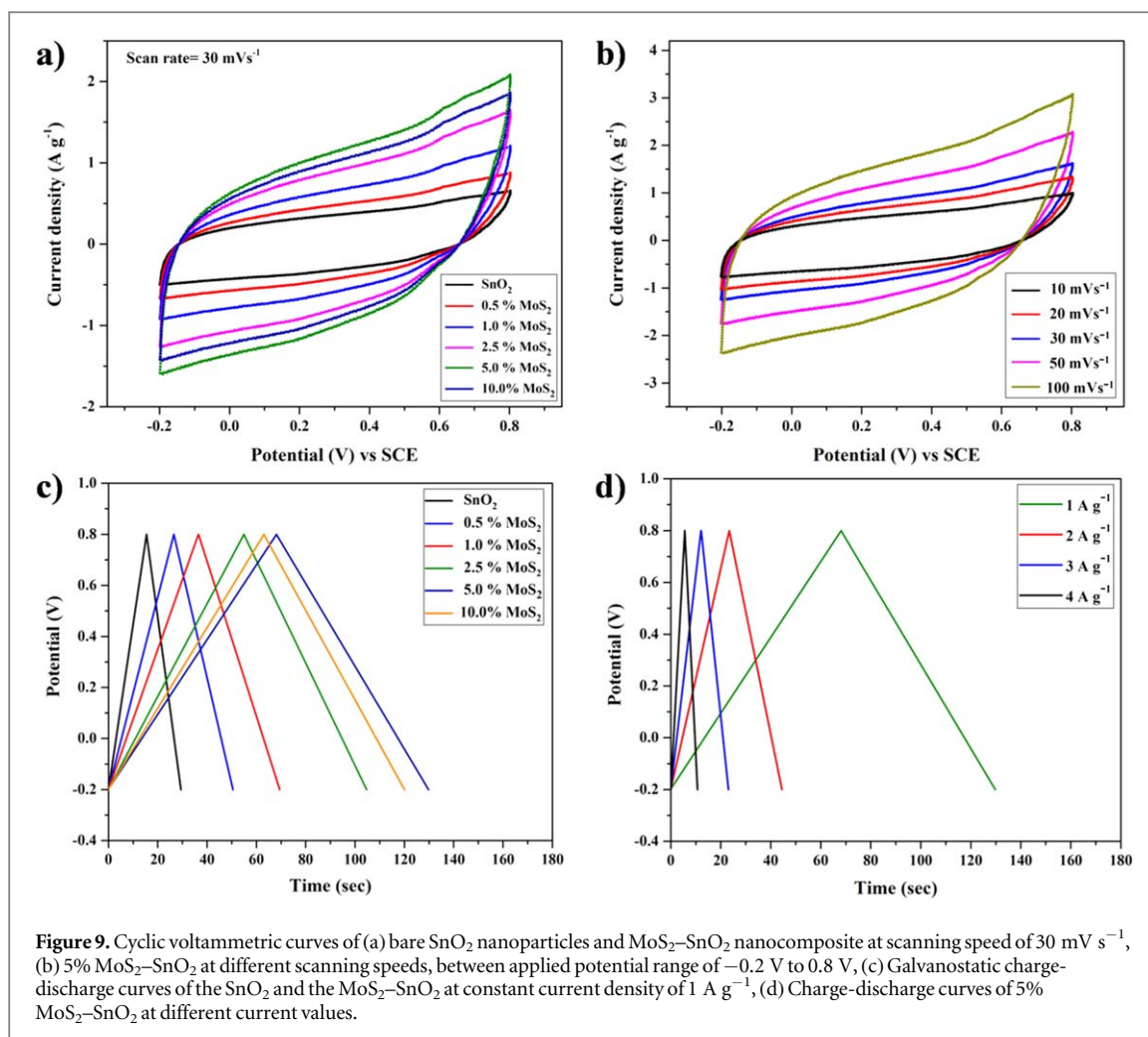
Figure 6. Raman spectrum of bulk MoS_2 powder and exfoliated MoS_2 nanosheets.

Figure 7(a) shows the particle size distribution chart of SnO_2 nanoparticles obtained from the particle analysis test. It is observed that the average size of the synthesised SnO_2 nanoparticles was calculated as 17.7 nm. Almost 90% of the particles fall in the size range of 10–21 nm with rest of the particles fall in the size range of 25–37 nm. Figure 7(b) shows the Nitrogen adsorption-desorption curve and pore size distribution (Inset) of the MoS_2 nanosheets. The adsorption-desorption isotherm with a small hysteresis loop indicates that the isotherm is a type IV isotherm as defined by the IUPAC [31]. The pore size distribution curve shows the peaks at the pore size of 6.2 nm, 8.2 nm and 14 nm with the mean pore size of 7.7 nm. This indicates the mesoporous nature of the dried MoS_2 nanosheets powder [31]. The specific surface area (S_a) of the MoS_2 nanosheets measured by BET method was $17.5 \text{ m}^2 \text{ g}^{-1}$. This S_a value of MoS_2 nanosheets was marginally larger than the previously reported values [31, 32].

In the XPS survey spectrum of MoS_2 - SnO_2 nanocomposite (figure 8(a)), the presence of peaks corresponding to Mo, Sn, S, O, N and C elements were observed. The high-resolution XPS spectrum of Mo 3d region (figure 8(b)) exhibited two peaks at binding energies 229.4 and 232.4 eV belonged to $3d_{5/2}$ and $3d_{3/2}$, respectively. These binding energies were corresponding to the +4 oxidation state of Mo. Also, a broad peak corresponding to S 2s was observed at 226.5 eV in the same spectrum. The high-resolution XPS spectrum of Sn 3d region is shown in figure 8(c). Two peaks at 487.4 and 495.8 eV were observed. These were belonged to $3d_{5/2}$ and $3d_{3/2}$ of Sn (+4 oxidation state), respectively. The XPS spectrum of S is shown in figure 8(d). The XPS spectrum of S 2s region was resolved into a doublet peak, with the binding energies 162.2 and 163.2 eV corresponding to $2p_{3/2}$ and $2p_{1/2}$ of S, respectively. The above results confirm the presence of MoS_2 and SnO_2 without any other oxidation products [25].



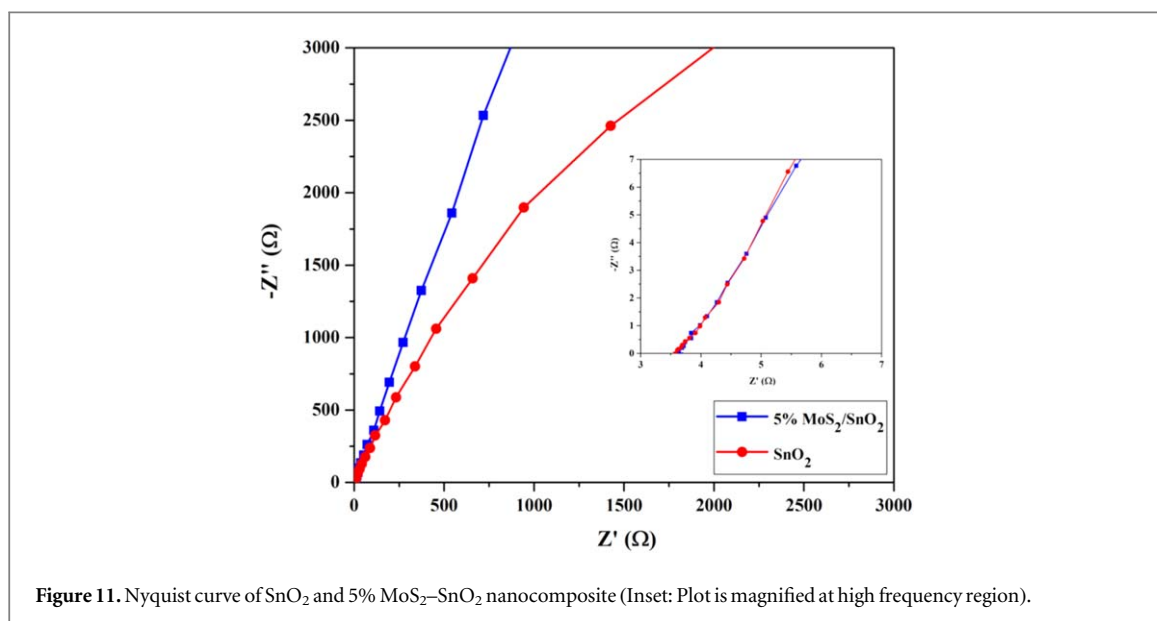
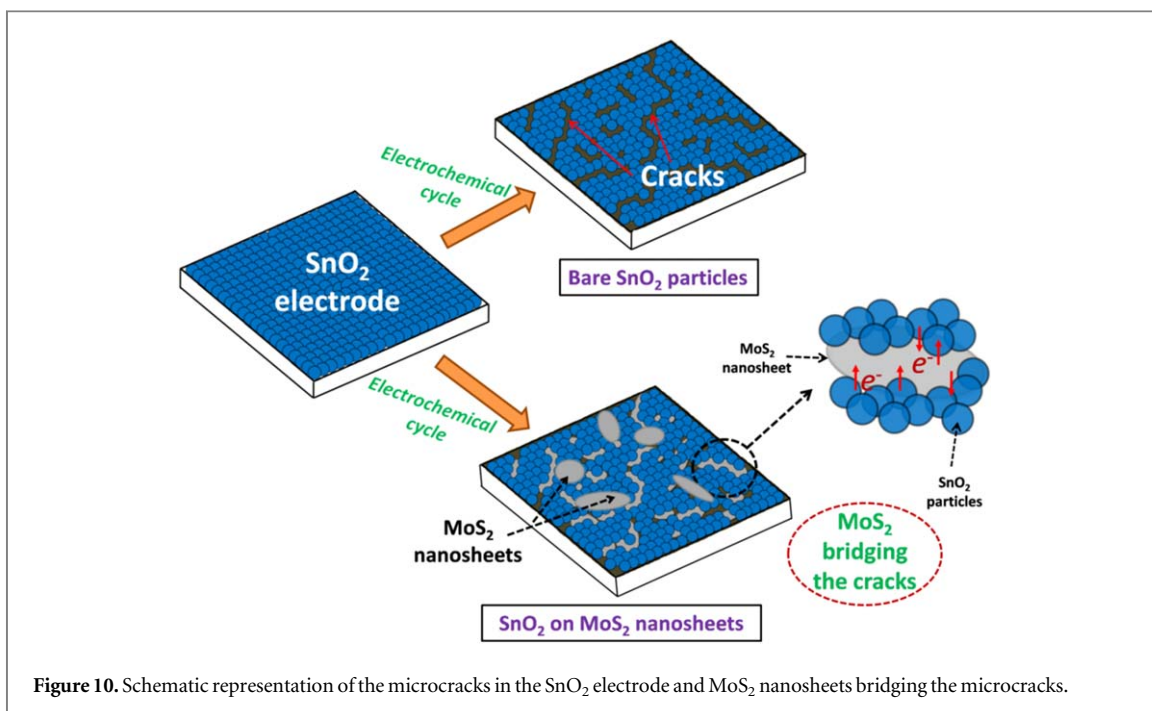
The cyclic voltammetry (CV) was performed for SnO₂ and MoS₂-SnO₂ between the potential value range of -0.2 V to 0.8 V. The CV plots are showed in figure 9(a). CV curves for all samples showed the double layer supercapacitor behavior with semi rectangular shape curve. This result was in accordance with the previously reported results [2, 12]. From figure 9(a), it was observed that the output current density of the MoS₂-SnO₂ nanocomposite is higher than the output current density of the bare SnO₂ nanoparticles. During the electrochemical process the SnO₂ nanoparticles undergo the volume expansion that lead to increase in the



particle size. The increase in the particle size leads to microcracks between the SnO₂ particles [6, 7]. The formation microcracks and reduction in the surface area were the reasons for the low performance of the SnO₂ nanoparticles. Addition of the MoS₂ nanosheets bridged the SnO₂ nanoparticles and provided the high surface area for the ion transportation between electrode and electrolyte. So, a gradual increase in the output current was observed with respect to increase in the MoS₂ to SnO₂ ratio. The 5% MoS₂-SnO₂ nanocomposite exhibited the highest current density amongst all the electrode materials. But the current density started to decrease when the MoS₂ ratio was increased to 10%. So it is concluded that the 5% is the optimum MoS₂-SnO₂ ratio for the supercapacitor application. Figure 9(b) shows the CV curves of 5% the MoS₂-SnO₂ nanocomposite at scanning speeds of 10, 20, 30, 50 and 100 mVs⁻¹ respectively. The obtained CV curves for all the scanning speeds are showing semi rectangular curve corresponding to double layer capacitance.

Figure 9(c) shows the galvanostatic charge-discharge curve of the SnO₂ and the MoS₂-SnO₂ nanocomposite. The charge-discharge study was performed within the potential of -0.2 V to 0.8 V at an applied current of 1 A g⁻¹. The obtained charge-discharge curves are linear and triangular in shape. This indicated the capacitance nature of the used electrode materials. The discharge time of the SnO₂ nanoparticles was 14 s, whereas, the MoS₂-SnO₂ nanocomposite showed longer discharge time. The discharge time was increased from 24 s for 0.5% MoS₂-SnO₂ to 61 s for 5% MoS₂-SnO₂.

As mentioned earlier, the SnO₂ particles would go through volume or size change during the electrochemical process [6, 7]. This would lead to microcracks in the SnO₂ electrode. The discontinuities formed by this cracks would affect the charge transportation. But, when the MoS₂ nanosheets were introduced with the SnO₂ nanoparticles, the nanosheets acted as a conductive pathway between the SnO₂ nanoparticles. This is explained schematically in figure 10. The MoS₂ nanosheets would anchor the SnO₂ nanoparticles within its surface and bridge the neighboring SnO₂ nanoparticles, electrically to help the charge transfer easier during the charge-discharge process [22]. The large surface area of MoS₂ nanosheets leads to effective interaction between the electrode and the electrolyte. Thus, the electrochemical performance (CV curve area and discharge time) was increased for the MoS₂-SnO₂ nanocomposite. But, it was noticed that the increasing the MoS₂ ratio beyond 5% the discharge time started to decrease. When the ratio of the MoS₂ was increased above the optimum value, the



discharge time started to decrease because of the reduction in active SnO₂ material. So it is concluded that the 5% is the optimum ratio in the MoS₂-SnO₂ nanocomposite. This is also in accordance with the cyclic voltammetry study.

The Nyquist plot obtained from the electrochemical impedance spectroscopy (EIS) is shown in figure 11. EIS was performed over the frequency range of 0.1 Hz to 100 kHz for bare SnO₂ and 5% MoS₂-SnO₂ are compared. A straight line was observed for both materials at lower frequency region. However, the slope of 5% MoS₂-SnO₂ nanocomposite was higher than that of the bare SnO₂ nanoparticles. This proves that the electronic resistance of the MoS₂-SnO₂ nanocomposite was lower than that of the bare SnO₂ nanoparticles. It indicated that the MoS₂-SnO₂ nanocomposite exhibited more ideal capacitor behavior than the bare SnO₂ nanoparticles. The high-frequency region was enlarged and shown as inset image. The curves tend to show a very small loop, the characteristics of charge transfer resistance, at higher frequency region. It is attributed to low charge transfer resistance of the electrode [33]. The solution resistance is the value at which the curve is intersecting with real axis. The values were almost equal for 5% MoS₂-SnO₂ nanocomposite and SnO₂ nanoparticles with 3.67 and 3.6 Ω respectively.

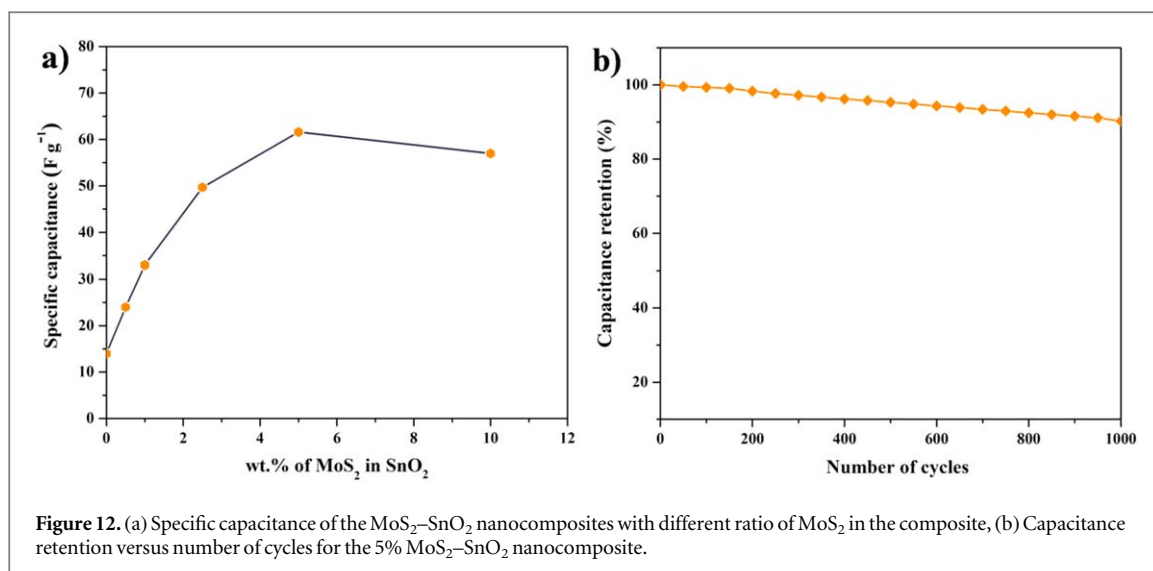


Figure 12. (a) Specific capacitance of the MoS₂-SnO₂ nanocomposites with different ratio of MoS₂ in the composite, (b) Capacitance retention versus number of cycles for the 5% MoS₂-SnO₂ nanocomposite.

The specific capacitance was calculated from the charge-discharge curve by the following equation [22],

$$C_s = (I \cdot t_d) / (m \cdot \Delta V) \quad (1)$$

where, ' C_s ' is the specific capacitance (F g⁻¹), ' I ' is the discharge current (A), ' t_d ' is the discharge time (s), ' m ' is the mass of the electrode material (g) and ' ΔV ' is the potential window. The values of C_s for SnO₂ and MoS₂-SnO₂ were plotted and shown in figure 12(a).

The specific capacitance (C_s) of bare SnO₂ nanoparticles was calculated to be 14 F g⁻¹. The specific capacitance value of the SnO₂ was increased after the addition of the MoS₂ nanosheets. The high surface area and easy the charge transportation between the electrode material and electrolyte were the reason for the increased value of specific capacitance. The 5% MoS₂-SnO₂ nanocomposite showed the highest specific capacitance value of 61.6 F g⁻¹ which is 4.4 times greater than that of bare SnO₂ nanoparticles.

The capacitance retention curve of 5% MoS₂-SnO₂ over the charge-discharge of 1000 cycles is shown in figure 12(b). The MoS₂-SnO₂ showed very good cyclic stability by retaining 90% of its original capacitance even after 1000 number of charge-discharge cycles. This indicates that the electrochemical performance of the SnO₂ nanoparticles is improved by the addition of the MoS₂ nanosheets with the SnO₂. The reasons for the improvement in the performance are, (1) The MoS₂ nanosheets provided good support and conductive path in the electrode material when the microcracks are formed due to volume expansion of SnO₂; (2) The high surface area offered to the electrode in the form of nanosheets made it easier for the ion transportation between electrolyte and electrode.

4. Conclusions

The MoS₂-SnO₂ nanocomposite was prepared by functionalization of SnO₂ nanoparticles onto the surface of the MoS₂ nanosheets by ligand exchange process. The specific capacitance of the bare SnO₂ nanoparticles calculated from galvanostatic charge-discharge was 14 F g⁻¹. The low specific capacity of SnO₂ was due to the discontinuity in the electrode material caused by the formation of microcracks. The problem of microcracks was solved by incorporating SnO₂ nanoparticles on the surface of the MoS₂ nanosheets. The MoS₂ nanosheets acted as a conducting bridge between the SnO₂ nanoparticles to help in charge transportation during the charge-discharge process. Also the nanosheets provided helped to improve the interaction between the electrolyte and electrode material. Thus, the specific capacitance of the MoS₂-SnO₂ nanocomposite was increased to 61.6 F g⁻¹. By improving the electrochemical performance of SnO₂, the prepared MoS₂-SnO₂ nanocomposite paves a way to develop low-cost energy storage devices for the future.

Acknowledgments

Prabukumar would like to thank NIT Karnataka for providing financial support in the form of Institute fellowship. The author is very thankful to Dr Shashi Bhusan Arya, Assistant Professor, Dept. of MME, NITK for

providing the facility to carry out the electrochemical characterization. The author wants to thank Mr Prashant Huilgol, Research Scholar, Dept. of MME, NITK for helping in TEM images.

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