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MoS_2 incorporated carbon allotropes (activated carbon, graphene, MWCNT) as electrodes in symmetric supercapacitors

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ABSTRACT

Symmetric supercapacitor devices were fabricated from MoS_2 incorporated carbon allotropes such as activated carbon (AC)/MoS_2, graphene/MoS_2 and MWCNT/MoS_2. The device performance was evaluated using cyclic voltammetry (CV), galvanostatic charge-discharge (GCD), and electrochemical impedance spectroscopy (EIS). The electrochemical properties of the devices fabricated from carbon allotropes (activated carbon, graphene, MWCNT) were remarkably enhanced to above 50% by the incorporation MoS_2 phases. Out of the three fabricated devices, electrochemical performance of AC/MoS_2 as found to be superior. The specific capacitance and energy density of this device is 216 F/g and 6.2 Wh/Kg respectively with excellent higher rate capability and longer cyclic durability. The devices fabricated from graphene/MoS_2 and MWCNT/MoS_2 has exhibited a specific capacitance value of 202 F/g and 161 F/g with an energy density value of 5.68 Wh/Kg and 3.95 Wh/Kg respectively.

1. Introduction

The world economy demands the need of sustainable renewable energy technology for a clean development mechanism by reducing carbon footprint. Functional materials that can convert and store renewable energy have received a significant attention in recent years. The current technologies such as photovoltaics, wind turbines, geothermal power plants, etc. are highly viable for renewable energy conversion to electric energy; however the storage of this intermittent source of energy is the bottle-neck in sustainable energy management. Supercapacitors/Ultracapacitors has emerged as novel alternative for the conventionally using batteries and capacitors due to its high power delivery, quick charge-discharge capability, wide range of operating temperature and environmentally benign assembly, thus the device become popular in almost all fore-front areas of technological development [2].

Allotropes of carbon such as activated carbon, graphene, multi-walled carbon nanotubes are preferred as the electrode materials for supercapacitor applications owing to its high surface area, excellent conductivity, low density and porosity [3]. However the energy density and rate capability of the devices fabricated from these pristine carbon allotropes are found to be very small owing to it electrical double layer (EDLC) nature of charge storage mechanism. In the case of graphene and MWCNT the electrode fabrication process is very difficult due to aggregation and stacking of the materials, thus the electrochemical properties of the device deteriorate after each cycle [4]. Moreover the theoretical densities of these materials are quite less which hinders the higher mass loading across the electrodes. Recent advances in hybrid materials of carbon allotropes with pseudo-capacitive materials indicates that the energy density and electrochemical properties of activated carbon, graphene, multi-walled carbon nanotubes can be enhanced considerably by the introduction of synergism between EDLC and pseudo-capacitive nature [5]. Moreover the pseudo-capacitive phases can acts as a spacer in between the 2D layers and 1D tubes of graphene, MWCNT respectively which reduces the chance for agglomeration/stacking during electrochemical process [6]. Rakhi et al., reported an enhanced electrochemical performance of carbon nano coils (CNC) by hybridizing with nanocrystalline semiconductor oxides such as SnO_2, MnO_2 and RuO_2 [6]. In an another report Liu et al., recently developed oxygen vacancy rich Co_3O_4/graphene nano-structures with an excellent specific capacitance value of 978.1 Fg^{-1} at a current density of 1 Ag^{-1}in three electrode configuration [7]. The electrochemical properties of activated carbon were also modified by various methods by metal oxide incorporation.

Transition metal dichalcogenides such as MoS_2 have recently received great deal attention among pseudo-capacitive materials due to

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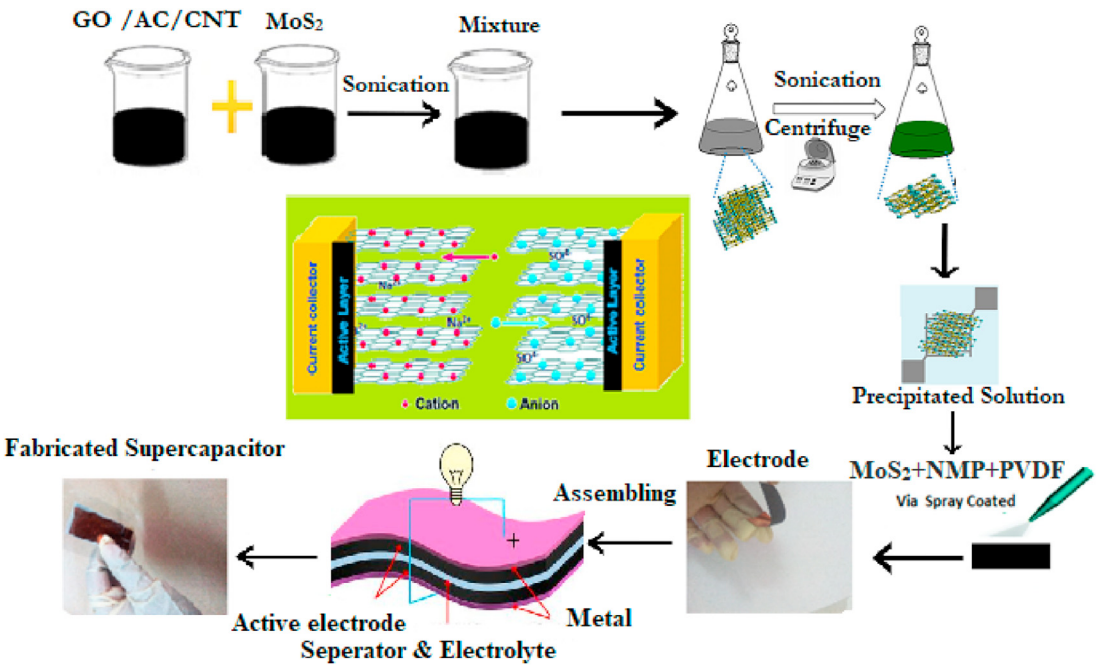

Scheme 1. Schematic representation of fabrication supercapacitor devices.

its 2D layer structure, high surface area and theoretical density [8]. Faradaic electron transfer from the change of oxidation states, ion intercalation between neighbouring layers, and ion adsorption at the surface with electric double-layer formation are the cited reasons for the substantial improvements in the electrochemical performance in MoS_2 [9,10].

In a recent report by Bissett et al., conductive graphene nano flakes were incorporated in solution exfoliated MoS_2 sheets to form a symmetric coin cell supercapacitor. The optimal combination of MoS_2/graphene composite sample to achieve a specific capacitance of 11 mF/cm^2 at a scan rate of 5 mV/s was found to be 1:3 [11]. A sandwiched supercapacitor device from graphene incorporated MoS_2 inks were prepared by Losic et al., they could achieve a specific capacitance of 392 F/g at 5 mV/s scan rate [12]. MoS_2@CNT/RGO network composites electrodes were developed for flexible supercapacitor device applications by Hao et al., and they could achieve a specific capacitance of 129 mF/cm^2 at 0.1 mA/cm^2 [13]. Despite of these attempts, few reports are available that focus to enhance the electrochemical properties of carbon allotropes by hybridizing with pseudo-capacitive oxides and sulphides that will mitigate the chance for agglomeration of the electrode materials during electrochemical process [13,14]. Moreover the supercapacitor device fabrication using carbon allotropes/MoS_2 nanocomposites were seldom reported in the literature [15]. In the present report, MoS_2 incorporated nano hybrid structures of carbon allotropes such as AC/MoS_2, graphene/MoS_2, MWCNT/MoS_2 was prepared by conventional ultra-sonication technique. The materials were used as electrodes in symmetric supercapacitor devices using Na_2SO_4 gel electrolyte.

2. Experimental techniques

2.1. Synthesis of MoS_2 nano flowers

MoS_2 nanostructures with flower shaped morphology were prepared by hydrothermal method reported elsewhere in the literature [16]. 1.2 g $Na_2MoO_4.2H_2O$ (Merk, India) and 1.6 g NH_2CSNH_2 (Merk, India) were dissolved in de-ionised water and stirred for about 30 min. The homogeneous solution so obtained was kept in a stainless steel hydrothermal flask at 200 °C for 24 h. After cooling, obtained black precipitate was washed several times with de-ionised water and the product was dried at 80 °C/6 h in a conventional hot air oven to get a dark green MoS_2 nano powder.

2.2. Preparation of AC/MoS_2, graphene/MoS_2 and MWCNT/MoS_2 nano-hybrid structures

MoS_2 intercalated nano-hybrid structures of AC, graphene (Hi-Media, India) and MWCNT (United Nanotech Innovations, India) having 2: 1 composition was prepared ultra-sonication method. The 0.2 g of carbon allotropes and 0.1 g of MoS_2 were separately weighed out into a 100 ml beaker containing 40 ml de-ionised water. The suspension was ultra-sonicated using a probe sonicator for 30 min. The precipitates were collected by centrifugation, washed and dried in a vacuum oven at 150 °C for 1 h.

2.3. Synthesis of PVA/Na_2SO_4 gel electrolyte

Polyvinyl alcohol (PVA)/Na_2SO_4 gel electrolyte was prepared by mixing 5.68 g of Na_2SO_4 crystals and 4 g of PVA powder in 40 ml water. The mixture was heated to 100 °C/30 min to get a uniform suspension. It is then cooled to room temperature to get PVA/Na_2SO_4 gel electrolyte.

2.4. Symmetric supercapacitor device fabrication

For the fabrication of electrodes, 1 g of active material was mixed thoroughly with 0.1 g of polyvinylidene fluoride (PVDF) in 5 ml n-methyl pyrrolidine (NMP) to get slurry. The slurry was coated uniformly on two copper foils (kept at 80 °C) having 1 mm thickness and 2 × 3 cm^2 area (Alfa Aser, India). The electrodes were kept at the same temperature for another 20 min to remove moisture and solvent molecules. Two electrodes with approximately 0.02 g of mass loading were then sandwiched on the PVA/Na_2SO_4 gel electrolyte film. After attaching leads on the electrodes entire assembly was sealed with thermal sleeves to avoid external contacts [16,17]. The schematic representation of the above procedure is illustrated in Scheme 1.

2.5. Characterization

Crystalline phase formation of the samples were recorded (10-60°)

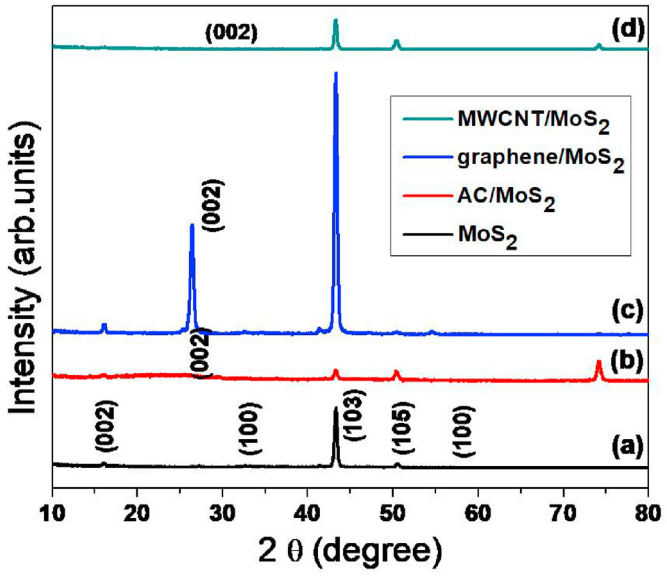

Fig. 1. XRD patterns of (a) MoS_2 (b) AC/MoS_2, (c) graphene/MoS_2 and (d) MWCNT/MoS_2.

using Rigaku X-ray diffractometer using Cu Kα radiation, $\lambda = 1.54178$ Å. Surface morphology of samples was analyzed with high-resolution field emission electron microscope (FESEM, Carl Zeiss Ultra55). Electrochemical measurements were done with an electrochemical workstation (Bio-Logic SP-150) followed by cyclic voltammetry (CV) in the voltage range of 0 V–1 V at different scan rates. The electrochemical impedance spectroscopy (EIS) measurements were performed over a frequency range from 1 MHz to 1 mHz with an alternating voltage of amplitude of 20 mV. Charge-discharge measurements were conducted from 0 to 1 V at a current density of 50 mA.

3. Results and discussions

3.1. Crystal structure analysis

XRD patterns of MoS_2, AC/MoS_2, graphene/MoS_2 and MWCNT/MoS_2 are given in Fig. 1. The peaks at 2θ angles 16, 32, 43, 51 and 59 correspond to the reflections from (002), (100), (103), (105) and (100) crystal planes of hexagonal phase of MoS_2 (JCPDS 37–1492) indicating the presence of pure MoS_2 [18]. For AC/MoS_2, graphene/MoS_2 and MWCNT/MoS_2 in addition to the diffraction peaks of MoS_2, emergence of additional peaks at 27° can be attributed to the diffraction from (002) planes of carbon allotropes (JCPDS 75–2078) [19].

3.2. Morphology analysis

The FESEM image of as synthesized MoS_2 is depicted in Fig. 2 (a) which shows a flower shaped morphology due to the aggregation of nanoplate like structures. Morphology of AC/MoS_2, graphene/MoS_2 and MWCNT/MoS_2 nano-hybrid structures were also represented in Fig. 2 (b), (c) and (d) respectively. The hybrid structures show a uniform distribution of MoS_2 nano flowers in their matrices. The porous structures in all the electrodes facilitate the effective transport of ions during electrochemical process.

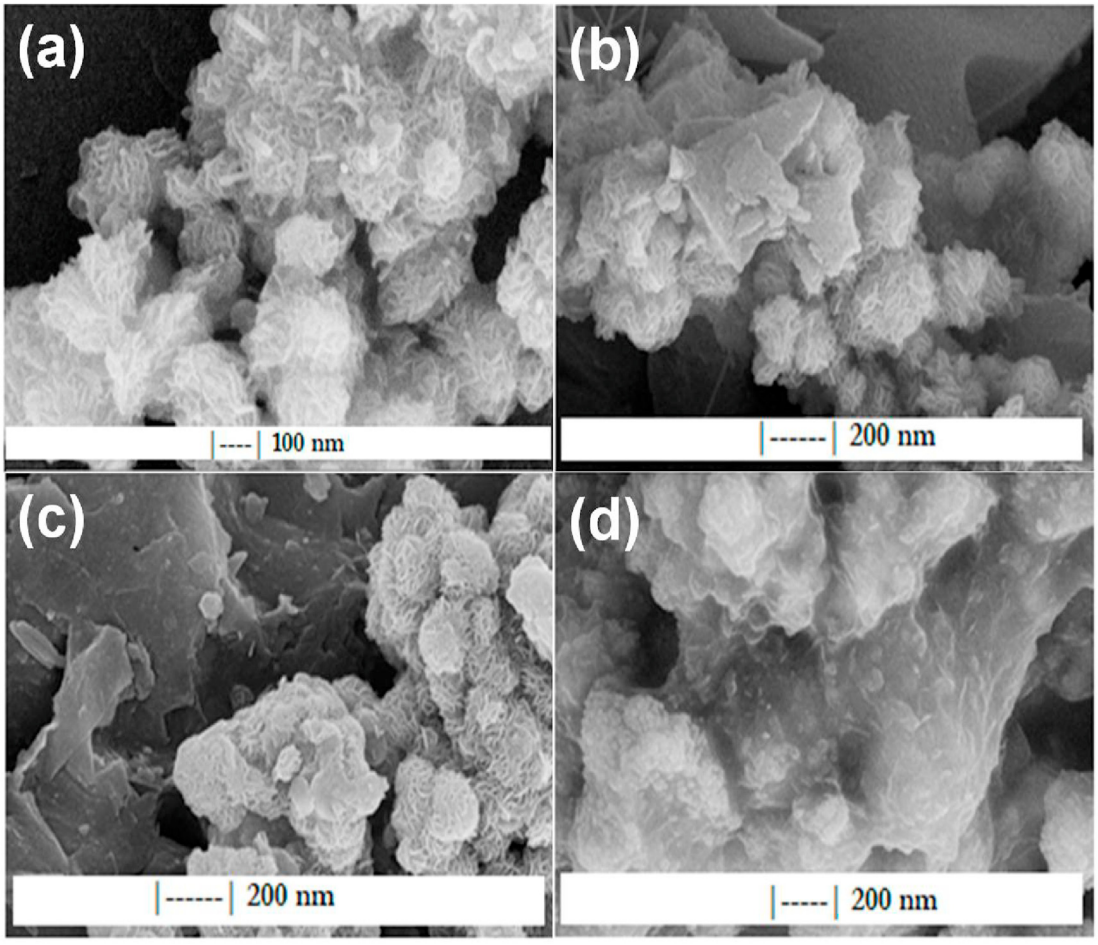

Fig. 2. FESEM images of (a) MoS_2 (b) AC/MoS_2, (c) graphene/MoS_2 and (d) MWCNT/MoS_2.

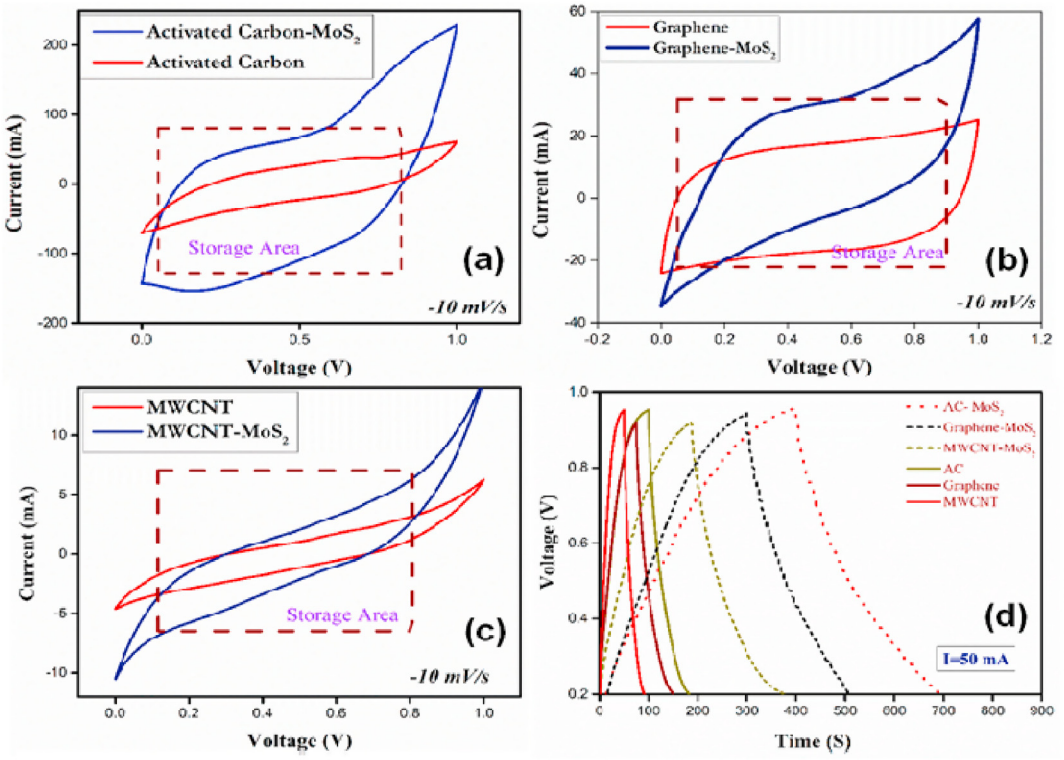

Fig. 3. Cyclic voltammograms of (a) AC/MoS_2, (b) graphene/MoS_2, (c) MWCNT/MoS_2 at a scan rate of 10 mV/s (d) Galvanostatic charge–discharge curves of AC/MoS_2, graphene/MoS_2, MWCNT/MoS_2 at a current density of 50 mA.

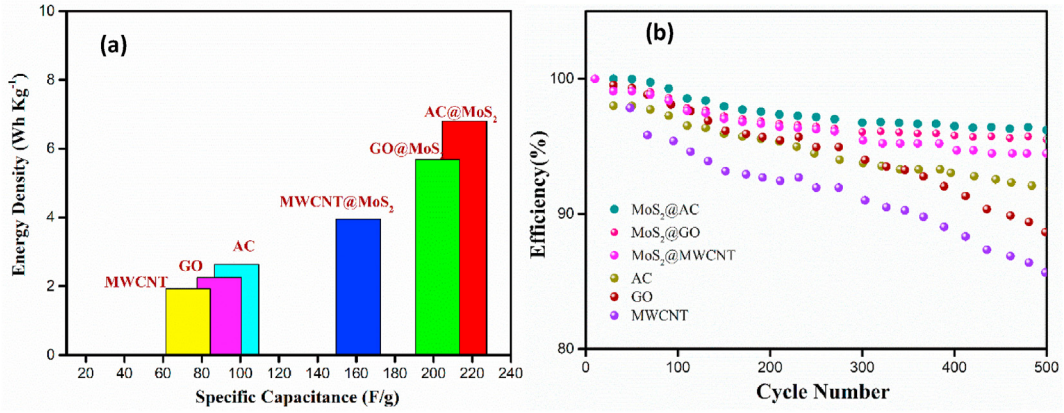

Fig. 4. (a) Ragone plot of (energy density vs specific capacitance) supercapacitor devices fabricated from AC/MoS_2, graphene/MoS_2 and MWCNT/MoS_2 were compared with corresponding carbon allotropes. (b) Cyclic performance of the various devices at a constant current density of 50 mA.

3.3. Cyclic voltametry

Electrochemical performances of the fabricated supercapacitor devices using AC/MoS_2, graphene/MoS_2 and MWCNT/MoS_2 nano-hybrid electrodes were examined by cyclic voltammetry (CV) measurements. Fig. 3 (a), (b) and (c) shows the CV measurements of fabricated supercapacitors at a scan rate of 10 mV/s. It is observable from figures that, compared to AC, graphene and MWCNT, their nanohybrid structures with MoS_2 shows almost rectangular type CV which is the characteristic feature of an ideal capacitor device [20,21]. Moreover the pseudocapacitive nature of the composite sample by hybridising with MoS_2 is clearly visible above 0.6 V in the CV diagram which can be atributed to the following electrochemical reaction between electrode and electrolyte molecule [11].

$$MoS_2 + Na^+ + e^- \rightarrow MoS\text{–}SNa \quad (1)$$

3.4. Galvanostatic charge-discharge curve (GCD)

GCD measurements are usually carried out to analyze the actual device performance of the electrodes. Constant current charge-discharge cycles of AC, AC/MoS_2, GO, GO/MoS_2, MWCNT and MWCNT/MoS_2 at a current density of 50 mA are shown in Fig. 3 (d). The GCD curves of composite samples exhibited a triangular and symmetric charge-discharge curve indicating a high capacitive nature of the electrodes. The initial voltage drop (IR drop) during the discharge was accredited to

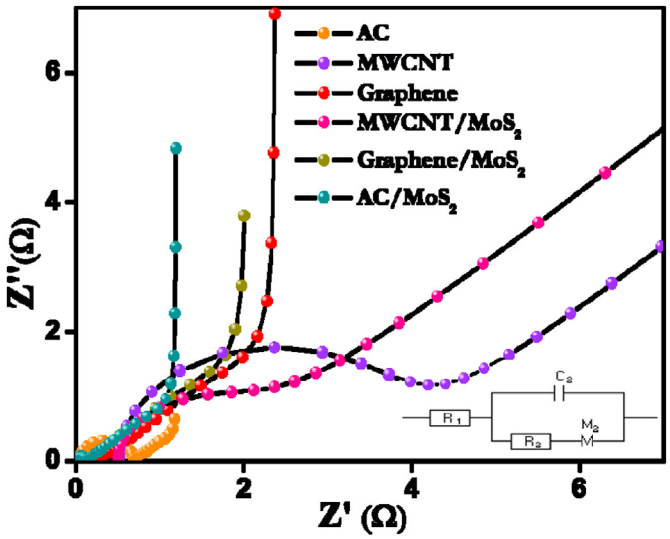

Fig. 5. Nyquist plots for AC, graphene, MWCNT, AC/MoS_2, graphene/MoS_2 and MWCNT/MoS_2 based supercapacitor devices at dc bias of 0 V with sinusoidal signal of 5 mV over the frequency range from 100 kHz to 1 MHz. *Z'*: real impedance. *Z'':* imaginary impedance.

the internal resistance of the materials [22]. Further, the small initial voltage loss indicated a low internal resistance and fast CV response of all the fabricated supercapacitors. The specific capacitance of fabricated supercapacitors were calculated from the following equation [23].

$$Csp = \frac{2I}{m}\,\Delta t \Big/ \Delta v \tag{2}$$

where *I, t, Δv* and m are the constant current (A), discharge time(s), total potential difference and weight of materials, respectively.*Csp* values obtained from GCD curves of AC, AC/MoS_2, GO, GO/MoS_2, MWCNT and MWCNT/MoS_2 are 98 F/g, 216 F/g, F/g, 89 F/g, 202 F/g, 73 F/g and 161 F/g respectively. As compared to the allotrpes of carbon discussed in the present work, the specific capacitance obtained for their MoS_2 intercalated electrodes exhibited a hike of 50–60%, resulted from the improved ion diffusion pathways created by MoS_2 nano-flowers. Moreover the surface modification might have decreased the chance for aggregation of the electrode material during the reaction.

The energy and power densites are calculated by the following equations [24].

$$E = \frac{I}{2}(CV^2) \tag{4}$$

$$P = E/\Delta t \tag{5}$$

where *C* is the capacitance of the two electrode system, *V* is the potential decrease in discharge, *E* is the energy density and *Δt* is the discharge time. The calculated energy densities of AC, AC/MoS_2, GO, GO/MoS_2, MWCNT and MWCNT/MoS_2 are 2.64 WhKg [1], 6.2 WhKg [1], 2.25 $WhKg^{-1}$, 5.68 $WhKg^{-1}$, 1.93 $WhKg^{-1}$ and 3.95 $WhKg^{-1}$ respectively. Fig. 4a presents a Ragone plot, comparing the specific capacitance and energy densities of prepared cells. From the plot it is clear that both the energy density and the Csp value of carbon allotropes have enhanced considerably by the incorporation of MoS_2 and we can conclude that this method is a promising step to modify the performance of the carbon allotropes. Moreover the cycle stability studies in Fig. 4b shows that the cycle life of the devices fabricated from composite electrodes such as AC/MoS_2, graphene/MoS_2 and MWCNT/MoS_2 have improved considerably than their bare carbon allotrope electrodes by the incorporation of MoS_2 phases.

3.5. Electrochemical impedance spectra (EIS)

The EIS analysis has been used as an essential tool to examine the charge transport behavior at the electrode-electrolyte interface of the supercapacitor device. Fig. 5 represents the Nyquist plots of the fabricated supercapacitors based on AC, AC/MoS_2, GO, GO/MoS_2, MWCNT and MWCNT/MoS_2 composite electrodes measured from high to low frequencies. The nyquist plot consists of a semi-circular arc at high frequency region followed by a straight line parallel to the y axis in the low frequency region [25]. The inset figure shows the equivalent circuit to fit the Nyquist plot which includes the internal resistance (R_1), The charge transfer resistance (R_2), the electric double layer capacitance C_2 and the Warburg impedance (M_2) [26]. The R_2 related to the ionic/intrinsic resistance of electrolyte and electrode, contact resistance of the electrode interface can be calculated from the high-frequency intersection of X-axis and R_2 is the span of semicircle along X-axis in the which attributed the process taking place at the interface of electrode/electrolyte caused by Faradaic reactions and electrochemical double layer capacitance [27]. The *ESR* is a factor that limits the specific power of a device which is the combined resistance of $R_1$and R_2. 45° slope of the curve represents the Warburg resistance which gives the frequency dependence of the device [28–30]. The calculated *ESR* of AC, AC/MoS_2, GO, GO/MoS_2, MWCNT and MWCNT/MoS_2 are 0.048 Ω, 0.023 Ω, 0.19 Ω, 0.14 Ω, 0.62 Ω and 0.53 Ω respectively. From the results, the MoS_2 attached materials show smaller *ESR* than the bare forms demonstrating improves charge transfer performance of the device by the addition of MoS_2.

4. Conclusion

MoS_2 intercalated nano-hybrid structures carbon allotropes such as AC/MoS_2, graphene/MoS_2 and MWCNT/MoS_2 were synthesized by conventional ultra-sonication method. Phase formation and homogeneity of the composites were analyzed from XRD and FESEM respectively. The materials were employed as electrodes in symmetric supercapacitor devices using PVA/Na_2SO_4 as the electrolyte. The performance of the devices analyzed from GCD shows that the introduction of MoS_2 to the carbon allotrope materials resulted in achieving a superior electrochemical performance. Device fabricated from AC/MoS_2 exhibited a specific capacitance of 216 F/g, energy density of 6.2 Wh/Kg, higher rate capability and longer cyclic durability. Other combination with graphene, and MWCNT also gave the same trend (202 F/g and 5.68 Wh/Kg for graphene/MoS_2, 161 F/g and 3.95 Wh/Kg for MWCNT/MoS_2). The results show that the MoS_2 intercalation to carbon allotropes is a promising technique to improve their device performance by minimizing the chance for aggregation/agglomeration during electrochemical process.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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