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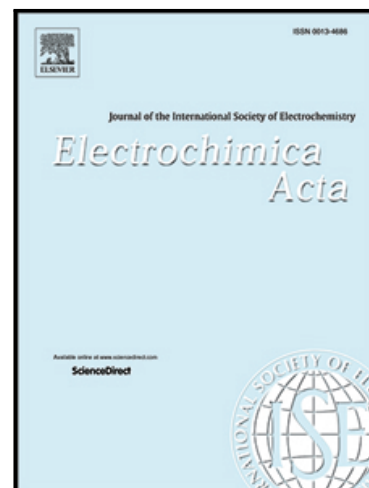
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Exploring 1T/2H MoS₂ Nano Coral Structured Active Electrodes for Enhanced Pseudocapacitive Supercapacitors: A Rigorous Evaluation and Characterization Study

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Highlights

- Cost effective synthesis of nano coral structured dual phase 1T/2H MoS₂ electrode material prepared via facile one step hydrothermal approach.
- The electrochemical tests were examined through both three and two cell configurations.
- The as prepared A-36 MoS₂ working electrode exhibits a high specific capacitance of 1550.86 F/g at current density of 1 A/g manifests an excellent rate capability (96.5% capacity retention after 1000 cycles).
- The two-cell asymmetric configuration demonstrated 97% of capacitive retention even after 5000 cycles at 10A/g.
- This device made the red LED lit up for 3 minutes, which conclude that this material can be used in miniature level energy storage devices.

Exploring 1T/2H MoS₂ Nano Coral Structured Active Electrodes for Enhanced Pseudocapacitive Supercapacitors: A Rigorous Evaluation and Characterization Study

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Abstract

This article optimizes the synthesis parameters of nano coral structured (NC-S) molybdenum disulfide (MoS₂) using hydrothermal method and enhances its physiochemical properties for high-performance supercapacitors (SC). This nano coral architecture of transition metal dichalcogenides has garnered significant attention for efficient pseudocapacitive charge storage in SC. Herein, this work demonstrates the fabrication of dual phase MoS₂ by tailoring the reaction time of the NC-S, furthermore this effect has been explained with the help of several analytical techniques. The synthesized 1T/2H MoS₂ NC-S phase was affirmed by powder X-ray diffraction, Raman spectroscopy and X-ray photoelectron spectroscopy studies. The field emission scanning electron microscope and transmission electron microscope images unveiled nano corals like morphology with numerous active sites. Impressively, the as prepared 24, 36 and 48 h MoS₂ NC-S entrenched as working electrode endows the achievement with a greater specific capacitance of about 761.82, 1550.86 and 1036.28 F/g at 1 A/g of current density

manifests an outstanding rate capability of 94.5, 96.5 and 95.3% capacitive retention even after 1000 cycles. Among them, the 36 h (A-36) synthesized NC-S MoS₂ possess high specific energy (43.61 Wh /kg) and power (225.50 W/kg) for this application. Additionally, it demonstrates a commendable capacitive retention of 97% for the two-cell asymmetric setup, even after 5000 cycles at 10 A/g of current density. This inventive configuration of a two-cell device is employed using a Swagelok setup, where the 1T/2H phase MoS₂ NC-S//AC exhibit outstanding storage stability and sustained up to 180 sec. The promising results validate that this material can significantly enhance performance in energy storage applications.

Keywords: Supercapacitor, Specific capacitance, Capacitive retention, Swagelok setup, Transition metals

1. Introduction

Current electrical vehicles (EV's) stupendously mark and capture the transportation sector, which signifies a positive sign for our planet, earth as there will be some reduction in carbon emissions. Hence, the contributions from researchers, and industrialists are intertwined and moulded into a single cohen line as "net zero carbon emission" [1,2]. The substantial subject of concern in this current biosphere is energy storage, as there is a cumulative energy demand. Later, to attain and accomplish this as a goal it requires a transition from usage of renewable energy sources to electrochemical energy sources. To manifest this demand, there is a need for devices, which can perform as a storehouse of energy. The available energy storehouse devices are fuel cells, batteries, and supercapacitors. Among these devices, supercapacitors have grabbed the attention of burgeoning storage devices as they have the potential possibility of high power and energy density, possess eco-friendly characteristics compared to conventional

batteries and capacitors. In these devices, the energy is retrieved from the chemical reaction with the applied potential [3]. Supercapacitor (SC) achieves a capacitance that of several orders larger than traditional capacitors by using nanomaterials with electrodes having enormous surface area and thin dielectric electrolytes [4-6]. The density of energy and power of SCs remain as the most predominant attributes. A quantity per unit mass or per unit volume for either measurement is expressed as density. A capacitor's stored energy (E), varies directly with its capacitance and square of its voltage [4]. In respect of the energy storage mechanism and the electrodes used, they are classified into three types of ultra-capacitors or SCs: Electrochemical double layer capacitors (EDLC), Pseudo supercapacitors (pseudo-SC) and Hybrid capacitors (HC) [7]. They are also portrayed as faradaic, non-faradaic and blends of these two types. The electrode - electrolyte charge transfer is the aspect of the faradaic process whereas in the non-faradaic technique, a redox chemical reaction does not take place [8]. The EDLC capacitors store charge electrostatically through a non-faradaic process, using the carbon and carbon-based nanomaterials as their electrodes and the pseudo-SC store charge electrochemically through a faradaic process use metal oxides and conducting polymer as their electrodes [9-11].

It is best to select an electrode material with a novel structure of crystal, high porosity, as well as enriched electrochemical active sites for an amazing energy storage device [12-14]. Until now, the electrode materials used for these applications are of carbon materials (activated carbon, graphene oxide, reduced graphene oxide (rGO)) and transition metal oxides (i.e., Fe_3O_4 [15], V_2O_5 , TiO_2 , etc.). Quite a lot of materials, particularly phosphates, oxides, sulphides, and polymers are currently under development for use of electrode materials [16,17]. Among them, phosphates, transition metal oxides (TMO's), and phosphides are the ubiquitously used as active materials, yet these materials express

some short comes like poor conductivity and short lifetime which limit their usage [18]. Recently, the emergence of transition metal dichalcogenides (TMDs) has garnered attention as promising candidates for energy storage devices. This is due to their potential characteristics, including high anisotropy, excellent chemical stability, solid mechanical stability, high electrical conductivity, and significant surface area [19]. Given this scenario, the considered electrode materials fall under the umbrella of TMDs. They are the compounds composed of transition metals and the chalcogens elements (selenium, sulphur, and tellurium). TMDs have number of crystalline layers, each coupled by weak forces (Van der Waals) like those in graphite and the compounds are analogues to their structure [20]. These materials possess polymorphs and have multiple oxidation states. The atoms present in a TMDs family are bonded covalently as X-M-X layers where M represent transition metal and X represents chalcogenide group. In recent years, the electrode materials such as WS_2 , WSe_2 , MoS_2 , MoSe_2 , NiS_2 , V_2S have been explored in SC at forefront. Among these, MoS_2 has distinct physical, electrochemical characteristics, intriguing sheet-like structure, more-number of oxidation states from +2 to +6 and defined layered structure [21]. Mostly, sulphides are chosen over selenides as they are highly available and less toxic when compared to selenides [22].

In MoS_2 , there are typically three phases: metallic (1T) phase and semiconducting phase (2H, 3R) characterized by their hexagonal (H), trigonal (T), and rhombohedral (R) crystal structures, with each phase indicating the layer count within the unit cell [23]. In the above-mentioned phases, metallic (1T) and semiconducting (2H) phase are the most synthesised for various applications. The phase 2H is stable and the phase 1T is thermodynamically unstable. 1T- MoS_2 exhibits high conductivity (10-100 S/cm), with high active sites of edge and the results gets overlooked by 2H phase in tremendous electrochemical activity. The electrochemical performance of 1T and 2H phase

synthesized individually declines the cyclic stability of the electrode due to their intrinsic low electrical conductivity. So, the mixed phase with a combination of 1T and 2H exhibits improved electrical conductivity and enhances electrochemical activity compared to the single phases of MoS₂ [24]. Mixed phase MoS₂ nanoparticles exhibit improved stability compared to single phase MoS₂ due to the presence of the more stable 2H phase and the stabilizing effect of the metallic 1T phase domains. This enhanced stability extends the self-life and operational lifespan of mixed-phase MoS₂ nanoparticles in various applications. Therefore, fabrication of mixed phase MoS₂ is suitable for the energy storage applications [25]. The electrochemical properties of MoS₂ have a high theoretical specific capacitance (2000 Fg⁻¹), received considerable attention because of its wide spread in nature as molybdenite and hence, it has been one of the most studied layered structures in TMDs [26]. Soon et al. [27] was the first one to exhibit the super-capacitive behaviour of MoS₂. The 2H-MoS₂ nanoparticles (NPs) for the solid-state SC was examined by Shanu et al [28]. Further, Vikraman et al. [29] had also engineered MoS₂ as the electrode material for hydrogen evolution reaction (HER) and energy storage application. MoS₂ and WS₂ was generally used for the energy applications by Wu et al., and Xuyen et al. was the first to publish a report on the microwave technique to create the dual phase MoS₂ using a straight forward microwave technique, along with its electrochemical performance for SC [30]. Feng et al. used a one pot hydrothermal technique to fabricate MoS₂ micro flowers [31]. Jiang et al. discovered that, by adjusting the hybridization phases of 1T and 2H in MoS₂ monolayer, they could enhance the SC capacitance. Saber et al. reported obtaining 1T-MoS₂ by using ammonium molybdate as a precursor, attributes it to the intercalation of foreign species (NH₄⁺) ions. Various synthesis techniques including mechanical exfoliation, chemical vapour deposition, microwave-assisted hydrothermal, ion intercalation, and hydrothermal method can be adopted. All the above discussion

corroborates that, the synthesis of MoS₂ through hydrothermal technique is a way more convenient method, produces high yield and inexpensive [32]. However, the weak conductivity and limited catalytic sites hinder their presence in practical applications [33]. Further, to expand the electrochemical properties, specific capacitance and electrical conductivity of the dual phase material, researchers focused on tuning the reaction time, molybdenum (Mo) to sulphur (S) ratio, reaction temperature, the addition of foreign materials, transition from one phase to other phase, and adjusting the percentage content of the phases, etc [34]. In addition to all these, the tuning of morphology also matters the most. The morphology of nanoparticles be in nanoflowers, nanoflakes, nanospheres, nano-corals and nanorods etc. Among all these, the nano-coral morphology foreshadows all the other morphology for bringing out the best performance of the material in electrochemical studies. The surface chemistry of the nano-coral structures plays a critical role in determining their electrochemical behaviour. These structures enhance the charge transfer capabilities by facilitating electron and ion transport during the electrochemical reactions. This type of intricate morphology increases the number of active sites for charge accumulation, leading to energy storage capacity and supports rapid ion diffusion during charging and discharging cycles. Nano-coral structure morphology and their surface chemistry play a significant role in electrochemical stability. These structures maintain the performance over the extended periods. An example of successful material of nano-coral morphology in supercapacitor application, materials like ZnCo₂S₄ with a coral-like hierarchical architecture demonstrate promising performance in storing energy through both electric double-layer capacitance and pseudo-capacitance. This dual mechanism allows for greater overall energy storage, which is beneficial in various high-energy applications [35]. These kinds of architecture are explored as innovative electrode materials in lithium-ion

batteries. Their unique structure enhances lithium-ion diffusion and provide efficient pathways for charge transport, which is critical in achieving high-rate performance [36,37].

This work presents the optimization of synthesis parameters for the fabrication of (1T/2H) MoS₂ NC-S through hydrothermal method which involves fine tuning of reaction conditions such as synthesis duration to achieve the formation of mixed phase MoS₂. The structural, morphological, and electrochemical techniques of dual phase MoS₂ NC-S were interrogated by various techniques. These findings gave an upshot of specific capacitance about 1550.86 F/g at 1 A/g of current density, capacitive retention, and a long cycle life for a bare dual phase 1T/2H MoS₂ due to the nano coral morphology, which can trait to many active sites. Upon with these results, the research introduces the innovative asymmetric device with dual phase MoS₂ NC-S, in combination with activated carbon was fabricated and made to illuminate a red LED power source up to 180 s. This configuration demonstrates the storage stability and sustained performance of the material. The novelty of this study lies in its facile synthesis method for achieving a mixed phase of MoS₂, which was accomplished solely by adjusting the reaction conditions, without the need for any additional reagents beyond the precursors.

2. Experimental Section

2.1. Materials

AMTH (Ammonium molybdate tetrahydrate, (NH₄)₆Mo₇O₂₄·4H₂O-98%, SRL), Thiourea (CH₄N₂S-99%, SRL) were used as molybdenum and sulphur source respectively. Potassium hydroxide pellets (KOH) were purchased from SRL, hydrochloric acid (HCl, 35-37%) from Molychem, ethanol from Hayman. The chemicals employed are of research grade and is used without further purification in this study.

2.2. Material Characterization

The investigation on the structural analysis of MoS₂ NC-S was performed using Powder X-ray Diffractometer (XRD) (PANalytical Aeris) with ($\lambda = 1.5409 \text{ \AA}$) Cu K α radiation with the accelerating voltage and applied current of 40 kV, 15 mA in the 2θ range of $5 - 70^\circ$ with a step size of 0.02° . Morphology of 1T/2H MoS₂ NC-S were analysed by field emission scanning electron microscope (FESEM) (7610F Plus /JEOL) and high-resolution transmission electron microscope (HRTEM) (JEOL Japan, JEM-2100 Plus). The surface functionalities were examined using fourier transform infrared spectroscopy (FTIR) (JASCO-4700) in the wavenumber ranging $4000\text{-}500 \text{ cm}^{-1}$. Raman spectra were detected by (Lab spec 6 -HORIBA Scientific) with 532 nm laser excitation. The presence of different element chemical state was confirmed by X-ray photoelectron spectroscopy (XPS) (Physical electronics, PHI Versaprobe III). The measurements of specific surface area were carried out in the Quanta chrome NovaWin instrument by using pure N₂ gas as absorbate at a temperature of 77.35 K. The surface area was determined using Brunauer Emmett Teller (BET) method and the distribution of pore size was assessed with Barrett-Joyner-Halenda (BJH) calculation. The electrochemical measurements of three-electrode (3E) and two-electrode (2E) fabrication were measured by CH instrument (CHI6097E).

2.3. Synthesis of MoS₂ nano corals

The nano corals like MoS₂ were prepared via hydrothermal technique. Briefly, 1 M of ((NH₄)₆Mo₇O₂₄·4H₂O) ammonium molybdate tetrahydrate (AMTH) and 2 M of (CH₄N₂S) Thiourea was dissolved in a 40 mL distilled water separately for 30 min under vigorous stirring at 350 rpm. Subsequently, the prepared thiourea solution was added gradually into the AMTH solution. Further, the homogeneous solutions were kept stirring for 3 h at room temperature (RT) of 33°C . Then the prepared mixture was transferred into a Teflon

lined autoclave of 100 mL and subjected at 200°C (5°C.min⁻¹) in hot air oven for different reaction durations (24, 36, and 48 h) were labelled as A-24, A-36, and A-48 respectively, cooled naturally to the RT. Thereafter, the black precipitate was collected by centrifuging and then washed with both ethanol and deionised water for six times to get rid of unwanted impurities. Finally, the black precipitate was dried at 80°C for 12 h in air and then the powder of MoS₂ NC-S was obtained.

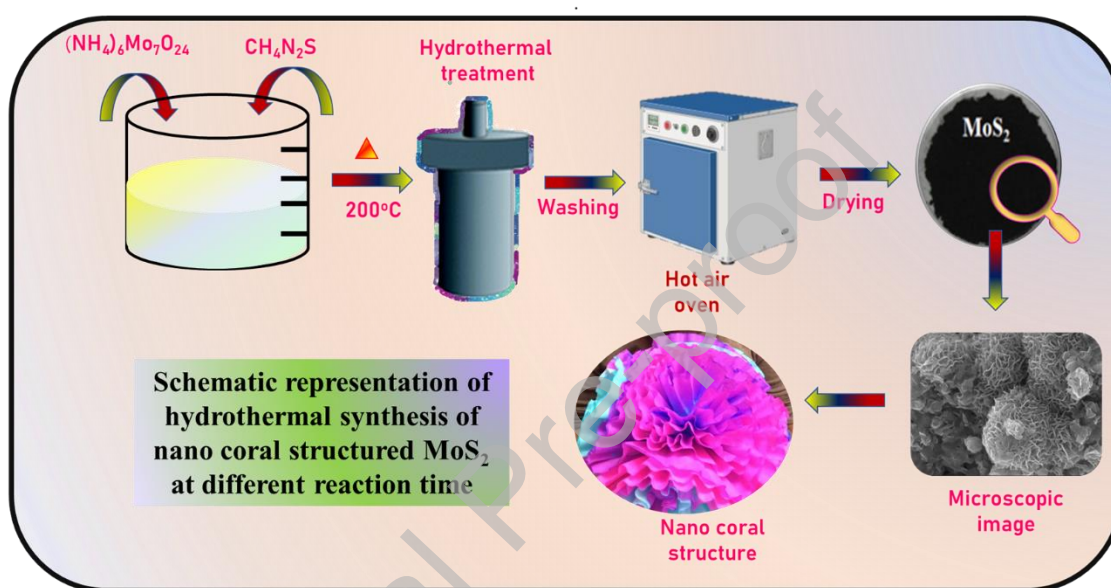


Figure 1. Schematic representation of synthesized 1T/2H MoS₂ NC-S.

2.4. Evolution of MoS₂ Nano Coral Formation

The conceivable growth development of MoS₂ NC-S was assembly of stacked nanostructures that are described based on crystallographic structure as well as detailed morphological investigations with respect to the process conditions. The growth development takes place as three phases in the synthesis process; Phase I - accelerated nucleation, Phase II - aligned aggregation, and Phase III - self-assembly. Initially, at ambient temperature, water containing (NH₄)₆Mo₇O₂₄·4H₂O and CH₄N₂S as the Mo⁴⁺, Mo⁶⁺ and S²⁻ sources contact elastically to form a transparent homogenous solution. Within the temperature range from the boiling point to the critical temperature of water

at 200°C, synthesis of MoS₂ nano corals were carried out. Further CH₄N₂S was hydrolysed to H₂S gas during hydrothermal treatment, a high pressure was created that causes the creation of MoS₂ centre, which develop into the order of molybdenum in middle of sulphur atoms (X-M-X) layers. Due to excessive stress, orientated primary nuclei aggregate, and this causes the formation of nanostructures [38]. These nanostructures act as the fundamental units for self-assembly process, leading to the formation of nano corals. This hierarchical arrangement is attributed to various parameters including van der Waals forces, hydrophobic and ionic interactions, hydrogen bonds and synergistic effects. When autoclave temperature increases, the process of nucleation becomes more prominent, favouring the growth of MoS₂ NC-S crystals with better quality and larger sizes, as seen in the structural and morphological analysis (Figure 1. Figure 2 and Figure 5).

2.5. Preparation of MoS₂ Nano Coral Electrodes for Electrochemical Measurements

The electrochemical performances of MoS₂ NC-S at different reaction time were supported out using three electrode configurations in an electrochemical workstation. The sludge made with the active material was used as a working electrode (WE), silver/silver chloride and platinum wire were subjected as the reference electrode (RE), and counter electrode (CE) in 1 M KOH electrolyte solution. The nickel foam was used as substrate with purity 99.9%, thickness 1.5 mm, dimension is about 1.0 cm x 2.0 cm and porosity are of 95% - 98% for working electrode to coat the active material and it was pre-cleaned with distilled water, ethanol and 10% HCl in distilled water under sonication for 30 min before usage and dried at 60°C. The sludge of the working electrode comprises of active material (1T/2H MoS₂), with 40 µL of Nafion[®] D-520 dispersion, 5% w/w in water and 1-propanol, Alfa Aesar and 20 µL of N-Methyl pyrrolidone (NMP), 99.5%, SRL. Then, the sludge is coated on to the substrate of 1x1 cm² using doctor blade method and

dried out at 80°C in an oven for about 12 hours. The cyclic voltammetry (CV) was measured from 0 to 0.8 V vs RE by varying sweep rates (5, 10 to 100 mV/s). The chronopotentiometry (CP) profiles were measured by varying the current densities from 1 to 5 A/g. The specific capacitance (C_{sp}) was attained from both the CV and CP profiles using the equations (1) and (2) for three electrode configurations.

$$C_{sp} = \frac{\int Idv}{v * m * \Delta V} \text{ F/g} \quad (1)$$

$$C_{sp} = \frac{I * \Delta t}{\Delta V * m} \text{ F/g} \quad (2)$$

where I stands for the current (A), Δt represents the discharge time (s), v indicates the sweep rate (mV/s), m represents the mass of the active material (g) and ΔV signifies the potential window (V). In addition to this, electrochemical impedance spectroscopy (EIS) was too evaluated at frequencies ranging from 0.001 - 100 kHz using an open circuit voltage of 0.025 V.

For 2E configurations, the cathodic electrode was carbon coated and the anodic electrode was coated with mixed phase MoS₂ NC-S with a filter paper as separator impregnated with 1 M KOH electrolyte that was placed between the electrodes in a Swagelok device setup. Notably, this device has been utilized to fabricate the area of 1x1 cm² cell device. The CV measurements were done in the potential range from 0 to 1.8 V by varying the sweep rates from 5 to 100 mV/s. The charge discharge profiles were measured with current densities ranging from 1 to 10 A/g. The most important parameter in asymmetric cell configuration is the balance of mass loading and it is determined by using the formula,

$$m_+ / m_- = C_- * nV_- / C_+ * nV_+ \quad (3)$$

where the m_+ and m_- denotes the mass loaded in the positive and negative electrode, ΔV_+ , ΔV_- and C_+ , C_- signifies the potential window and the specific capacitance of the positive and negative electrode respectively from 3E configuration. Both the electrodes

were balanced in 1:7 ratio (Activated carbon:1T/2H MoS₂ NC-S) according to the above-mentioned formula. The sludge of the nanoparticles was coated in nickel foam (substrate) the same way as it was done in 3E configuration. The specific capacitance was also calculated from CV and CP profiles of the device using the equations (4) and (5),

$$C_{sp} = \frac{\int I dv}{v \times 2 \times m \times \Delta V} \text{ F/g} \quad (4)$$

$$C_{sp} = 4 * \frac{I \times \Delta t}{\Delta V \times m} \text{ F/g} \quad (5)$$

In this 2E configuration, the 'm' represents the total mass of the active material coated on the both sides of the cell, I stands for the current (A), Δt represents the discharge time (s), v indicates the sweep rate (mV/s), and ΔV signifies the potential window (V).

The specific energy (E) and specific power (P) can be calculated using the following equations (6) and (7),

$$E = \frac{C_{sp} * V^2}{2 * 3.6} \text{ Wh/kg} \quad (6)$$

$$P = \frac{E * 3600}{\Delta t} \text{ W/kg} \quad (7)$$

Where, E is specific energy (Wh/kg), P is specific power (W/kg), V is the potential window, Δt is the discharge time and C_{sp} is the specific capacitance (F/g).

3. Results and Discussions

3.1. X-ray diffraction (XRD) Analysis

The powder XRD has been used to analyse the crystallographic and structural phase of as synthesized pristine MoS₂ NC-S with respect to reaction time, and the diffraction patterns that are exhibited in Figure 2. The characteristic diffraction peaks centred at $2\theta \sim 14.0^\circ$, 32.0° , and 59.0° corresponds towards the crystal planes of (002), (100), and (110) that were well agreed with the described JCPDS card no:75-1539 attributed to the 2H hexagonal phase. The diffraction peak lower than the standard peak imply the presence

of few layered nanosheets of MoS₂, development of metallic 1T phase and the distance between the interlayers of (002) plane enlarges [39]. The lower angle shift of (002) diffraction plane, obviously indicates the formation of 1T phase labelled as (002)_{new}. Li et al. [40] published that the shift towards lower angle diffraction was due to the ammonium ion intercalation between the Mo and S layers and implies large interplanar spacing [41]. The d spacing of (002) plane was calculated using Bragg's law and reckoned to be nearly 0.93, 0.92, and 0.96 nm for A-24, 36 and 48 was notably greater than the usual d spacing value (0.615 nm) of semiconducting 2H phase [42] and corresponds to an enlargement in the interlayer spacing which coincides with the above reference. The electrolyte ions may now access the plane more easily owing to structural expansion which supports in enhancing the electrochemical capacity. The absence of additional peaks in the patterns indicates minimal impurity in the synthesized MoS₂ NC-S. The broad region of 33.02° in the A-36 NC-S is due to the coexistence of both 1T/2H phase composition. Further, the broadening the (100) plane is caused due to the dominance of 1T/2H phase and structural disorder of the NC-S. The broadness of (100) plane results in higher angle shift leads to compression strain. [43]. The plane (110) indicates the 2H phase of MoS₂ that is significant in defining its layered structure and relates to their crystallographic arrangement. The average crystallite sizes for A-24, A-36, and A-48 are 10.70 nm, 5.07 nm, and 18.73 nm, respectively. The difference in crystallite size is ascribed to variations in reaction time during the synthesis process. As reaction time increases, nucleation and growth of the particles occur, and the reaction kinetics for each particle take longer to achieve complete growth or aggregation. Given the saturation levels of these parameters, their performance in energy storage applications is exceptional.

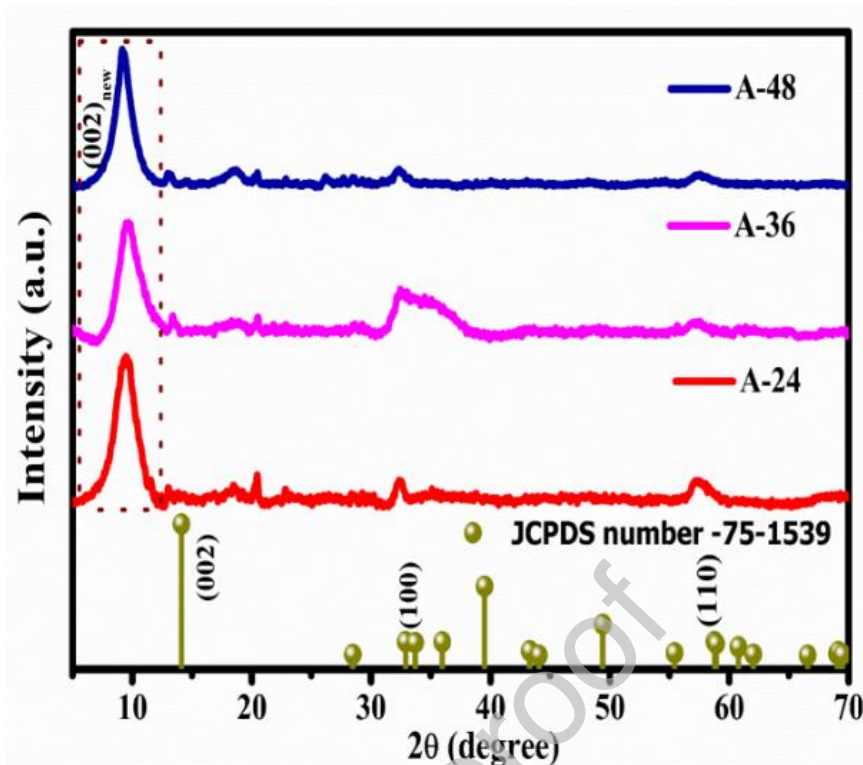


Figure 2. XRD patterns of MoS₂ NC-S for different reaction time.

3.2. Raman Spectroscopy Analysis

To determine the phase, composition, and vibrational modes of the MoS₂ NC-S, Raman spectroscopy was employed and their spectra were exhibited in Figure 3. Yuhui Li, et al. [44] observed the two typical peaks for 2H phase indexed at 380 and 413 cm⁻¹ correspond to E_{12g}, A_{1g} mode respectively and evidently reported that the emergence of several new vibration peaks at lower order Raman shift, indicates the presence of metallic 1T phase [31]. Er, et al. [45] discussed the three notable peaks which are second order spectrum labelled as J₁, J₂ and J₃ explains the formation of 1T phase. In these MoS₂ NC-S, the spectra indexed at 378 and 281 cm⁻¹ are the activation modes are attributed to E_{12g}, E_{1g} of 2H-MoS₂ is due to the vibration of in-plane and out-plane of molybdenum-sulphur atoms [46]. The existence of metallic (1T) phase of MoS₂ were indexed at 147 (J₁), 199 (J₂) and 400 (J₃) cm⁻¹ which are longitudinal acoustic phonon modes represented by the

second order peaks that confirm the existence of superlattice. The absence of (A_{1g}) mode is caused due to the dominance projected by the peaks of 1T phase. As the reaction time increases the intensity of the peaks has been enhanced. Sanghyan et al. [47] discuss the vibrational modes of both semiconducting and metallic phase of the electrode material that reflects with the obtained Raman spectrum. Based on above analyses, the as-prepared MoS_2 possess the hybrid 1T/2H phase which is well corroborated with XRD analysis.

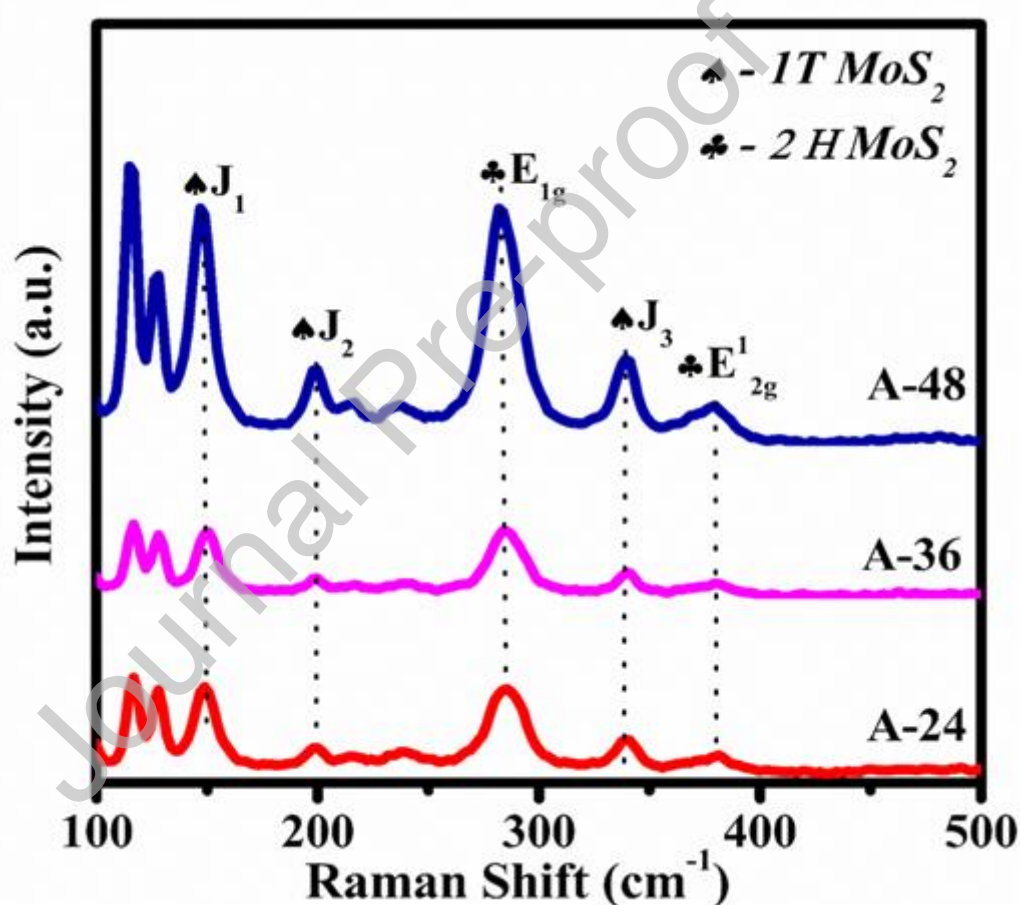


Figure 3. Raman spectra of MoS_2 NC-S for different reaction time.

3.3. Fourier Transform Infrared Spectroscopy (FTIR) Analysis

The chemical conformation of the MoS_2 NC-S was detected by FTIR spectroscopy and the Figure 4 depicts their range from 500 to 4000 cm^{-1} . The two distinctive peaks

observed at 1634 and 3432 cm^{-1} in the FTIR spectrum, was attributed to the bending vibration of O-H bonds in the absorbed water molecule. Lee, et.al, [47] and Song, et.al, [48] also discoursed about this indexed peak. It obviously shows that, the existence of the absorption peak indexed at 884 cm^{-1} attributes to the vibration of S-S atoms and the spectra indexed at 1350 cm^{-1} discloses the vibration of Mo-S atoms [49]. The peak at 1100 cm^{-1} belongs to C-O bond and the peaks indexed at 1850 cm^{-1} represent the bending of C=O bonds. The absorption spectra in the 2400 – 3000 cm^{-1} range were attributed to OH groups. Further to that, all the bonds related to MoS₂ NC-S were detected in the FTIR spectrum, strongly indicated the existence of 1T/2H phase.

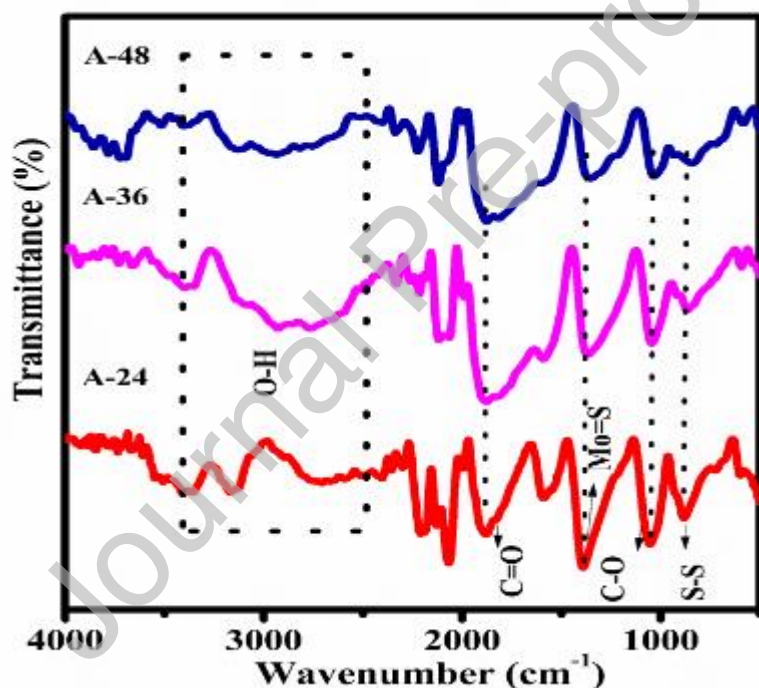


Figure 4. FTIR spectra of MoS₂ NC-S for different reaction time.

3.4. FESEM and HRTEM Analysis

The morphology of the as-synthesized 1T/2H MoS₂ NC-S was scan and imaged by Field emission scanning electron microscope (FESEM) and the morphological insights of the NPs are displayed in Figure 5. There is a transition of morphology from stacked

nanosheets to nano corals for pristine dual phase MoS₂, as the reaction duration increases. The nano coral is composed of lots of intercalated stacked nanosheets [50] and these nano corals were prominently noticeable in all the various synthesis duration. Figure 5 (a-f) exhibits the FESEM images at low and high magnification of MoS₂ NC-S obtained at 200°C in various reaction time. At low magnification, it is apparent that the NPs are densely arranged, forming a uniformly agglomerated NC-S. The dimensions of these nanostructures on average were around 0.455, 0.228 and 0.319 μm in diameter for A-24, 36, and 48 respectively. The FESEM image taken at high magnification divulged that the MoS₂ NC-S consists of ultrathin stacked nanosheets of about 10~15 nm in thickness as shown in Figure 5 (g-i). The coral like morphology was formed due to the agglomeration of stacked nanosheets like structures. Furthermore, the morphology of the hierarchical nanostructures described in this paper bears a resemblance to the typical nano coral morphology [51-54]. The obtained nano coral morphology enhances the number of electroactive sites which will increase the electrode-electrolyte interface. The high-resolution transmission electron microscope (HRTEM) images of A-36 NC-S is depicted in Figure 6 (a-c). The HRTEM analysis exhibits the formation of analogous sheet-like structures. These structures revealed well-defined lattice fringes and the morphological characterizations was in corroborated with FESEM observations, thereby validating the sheet like morphology. In conjunction with HRTEM imaging, the selected area electron diffraction (SAED) pattern showed diffused rings. Such patterns typically indicate the presence of poorly crystalline structures as showed in Figure 6 (d). This observation is in line with findings reported by Gajot.et.al [55], who noted similar diffraction pattern in their results. For MoS₂, common lattice spacings corresponds to the (002) planes, which are typically around 0.62 nm apart for 2H phase. The interplanar spacings of the lattice fringes observed in the HRTEM images were measured and found

to be in good agreement with the values obtained from XRD analysis. This concurrence between the HRTEM and XRD results further substantiates the structural analysis.

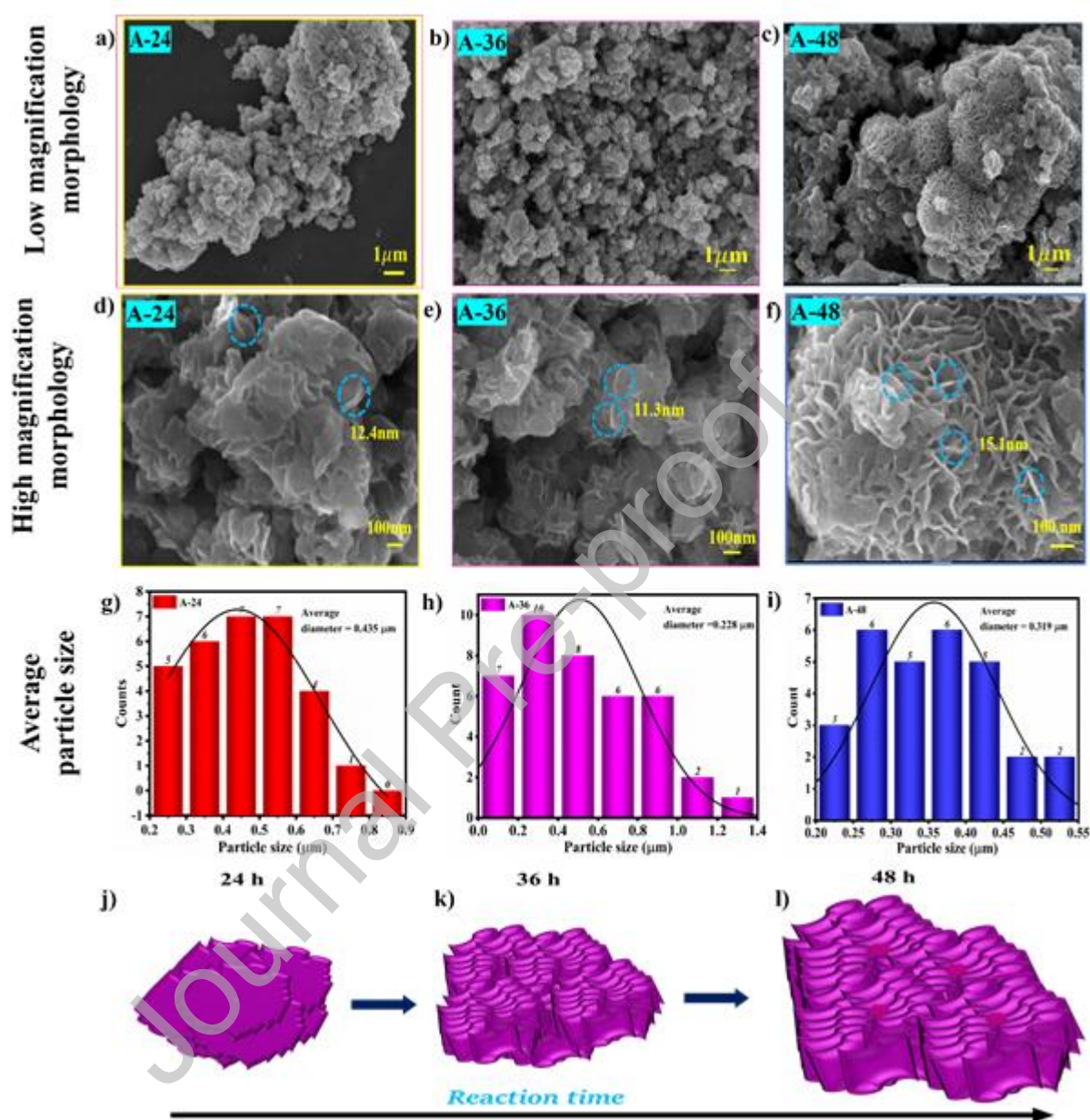


Figure 5. (a-c) Images of FESEM at lower magnification, (d-f) Images of FESEM at higher magnification, (g-i) average particle size distribution of the MoS₂ NC-S, and (j-l) Evolution of nanocoral structures with respect to reaction time.

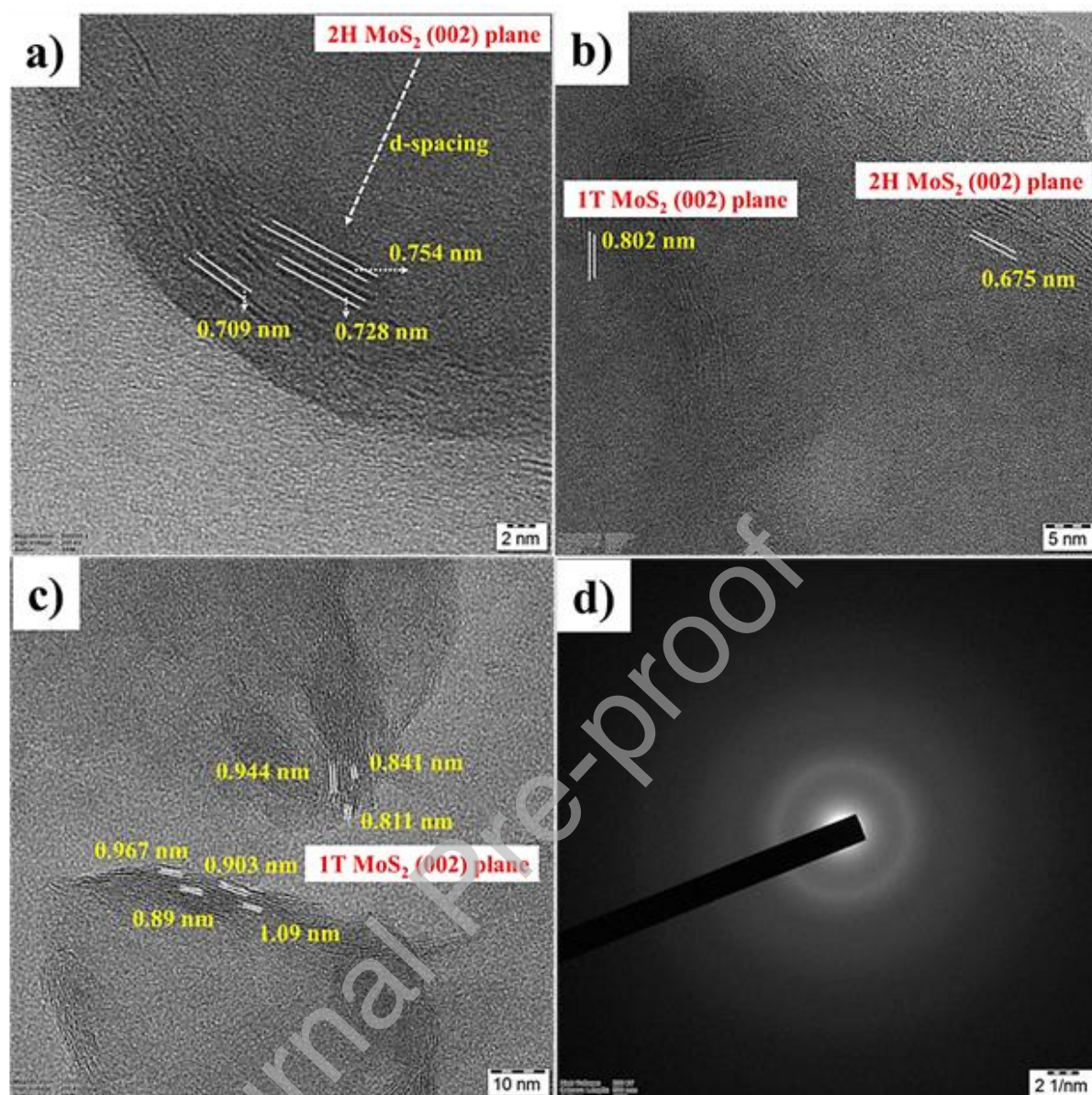


Figure 6. (a-c) HRTEM images of A-36 MoS₂ NC-S at different magnification, d) SAED pattern of A-36 MoS₂ NC-S.

3.5. BET Analysis

To measure the surface area of the MoS₂ NC-S at different reaction time, N₂ adsorption – desorption curves (BET) were employed. The adsorption and desorption isotherms were recorded using BET method and size of the pore is estimated using BJH method. As shown in Figure 7 (a,b), all the NC-S exhibits type IV isotherm which concludes the presence of mesoporous structure. The values of the specific surface area (SSA) was recorded to be

14.61, 10.77 and 11.74 m²/g for A-24, 36, and 48 MoS₂ NC-S respectively. The average pore diameter for the A-24, 36, and 48 MoS₂ NC-S were 14.609, 14.728 and 14.930 nm respectively. The pore size distribution confirms the existence of well-developed mesopores. These significant results confirmed that the presence of mesopores, potentially with high surface area which enables faster ion transfer to improve the electrochemical behaviour and enhances the exposure of electrochemically active sites and specific capacitance in the supercapacitor performance.

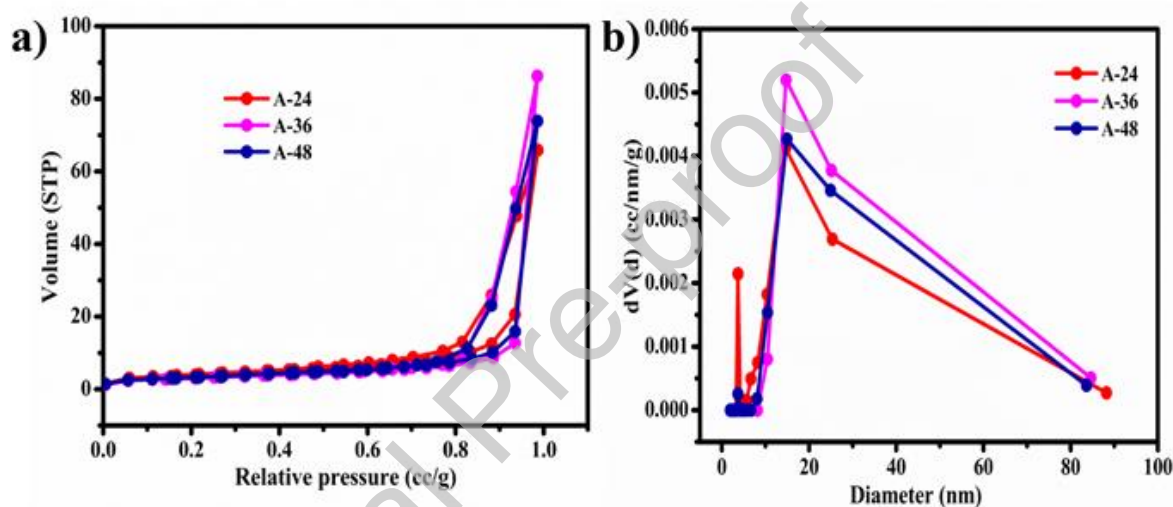
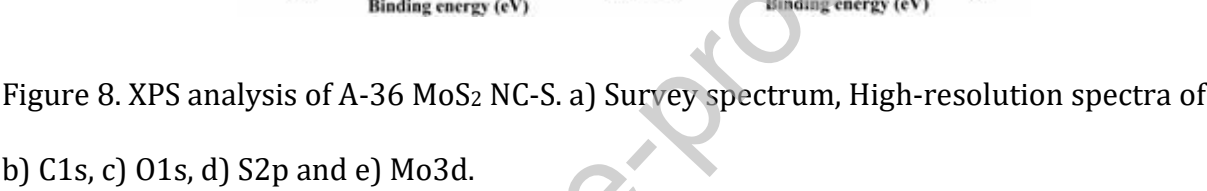


Figure 7. a) BET - N₂ adsorption/ desorption isotherm and b) BJH pore size distribution plot for all the A-24, 36, and 48 MoS₂ NC-S.

3.6. X-ray Photoelectron Spectroscopy Analysis

The presence of different elements chemical states such as Mo 3d, S 2p, O 1s, and C 1s in the A-36 MoS₂ is assessed through X-ray photoelectron spectroscopy (XPS) as displayed in Figure 8. The spectra were evaluated in CasaXPS using a linear type background. The total survey scan of as prepared A-36 MoS₂ NC-S indicates the occurrence of all the elements Mo, S, C and O is in full agreement with the composition of the material as shown in Figure 8 (a). The XPS signals of elements Mo 3d, S 2p, O 1s and C 1s were observed

which manifest the existence of molybdenum and sulphur in the A-36 MoS₂ NC-S. The carbon was used for calibration by setting the C 1s peak to 284.4 eV. The C 1s spectrum as shown in Figure 8 (b) is divided into three peaks at 284.8, 287.53 and 286.04 eV, which are associated with C-C, O-C=O, and C-O-C respectively [24]. The surface functional groups like C-O (533.6 eV) and OH (532 eV) were present thorough analysis of the O 1 s spectra depicted in Figure 8 (c). On the other side, the S 2p core level spectrum displayed in Figure 8 (d) shows six peaks, where the two peaks at 162.1 and 163.1 eV assigned as sulphur doublet 2p_{3/2} and 2p_{1/2} corresponding to the 1T phase of MoS₂ and the peaks at 163.6 and 164.5 eV corresponds to S²⁻ oxidation states reliable to 2p_{3/2} and 2p_{1/2} with the 2H phase of MoS₂ and the other two peaks at around 168.7 and 169.7 eV belongs to the chemical state of the metal sulphate. Furthermore, the Mo 3d spectra of A-36 MoS₂ NC-S demonstrates the coexistence of Mo⁴⁺ and Mo⁶⁺ oxidation states providing insights into the oxidation and valence states of Mo atom as shown in Figure 8 (e). The Mo 3d spectra were broke into six peaks in which the two characteristics peaks at around 229.4 and 232 eV corresponding to the doublet Mo 3d_{5/2} and Mo 3d_{3/2} for Mo⁴⁺ oxidation state consistent with the 2H phase of MoS₂, the other two peaks in consistent with 1T phase of MoS₂ are at around 229 and 232.4 eV corresponding to the doublet Mo 3d_{5/2} and Mo 3d_{3/2}. There is a pair of peaks at higher binding energy side of Mo 3d region at 233.08 and 236.4 eV corresponds to Mo 3d_{5/2} and Mo 3d_{3/2} of Mo⁶⁺ oxidation states, respectively. The presence of side peak at lower binding energy at around 226.5 eV is identified as S 2s core level [56]. So, the binding energy positions and the spin orbit splitting are in accordance with the earlier reports [57,58]



b) C1s, c) O1s, d) S2p and e) Mo3d.

3.7. Electrochemical Analysis of MoS₂ Nano Coral Electrodes

3.7.1. Cyclic voltammetry

For SC application, obtained MoS₂ nanocorals were coated on to the nickel foam. At first, the cyclic voltammetry (CV) of MoS₂ was examined in 1 M KOH electrolyte executed in a 3E configuration. The type of exhibition of the charge storage performance of the electrode materials, can be inferred from these curves. Lee et al. [47] suggested two dissimilar charge storage mechanism for an active electrode material. The former one is non-faradaic reaction and the latter is faradaic reaction. The charges accumulate on the electrode/electrolyte interface by electrostatic interaction on EDL (electric double layer) is called non faradaic reaction. When the charges accumulate through a oxidation and reduction transfer reaction, its called faradaic reaction. Kour et al. [49] reported that the anodic and cathodic redox peaks occur only in faradaic process of charge storage and

these redox peaks demonstrates the pseudocapacitive nature of the electrode material. Figure 9 (a-c) displays the CV curves of A-24, 36, and 48 MoS₂ NC-S electrodes at different scan rates respectively. The comparative CV curves of all the electrode materials at a sweep rate of 50 mV/s is depicted in Figure 9 (d). The profile of the CV curves reveals that the mechanism of charge storage in MoS₂ NC-S electrode is distinct than that of the electrochemical double layer capacitor behaviour, which was in shape of rectangle. With reference to the above trend, the well distinct redox peaks suggests the pseudocapacitive behaviour of the electrode materials and this inferees the electron transfer process in a reversible manner on the surface of the MoS₂ NC-S electrodes. Li et al [44], discuss that as the sweep rate increases from 5 to 100 mV/s, transition of oxidation/reduction peaks shift has been observed. The faster scan rates, enhances the peak currents but lowers the specific capacitance as electrolyte ions don't have necessary time to intercalate the MoS₂ NC-S electrode and complete the reaction of redox process. As seen in Figure 9 (a-c), the peak shift indicates an increase in chemical composition polarisation and internal resistance with increase in scan rates. The C_{sp} of the MoS₂ NC-S is found from the CV curves using the formula (1) mentioned earlier. The specific capacitance of about 1530.40, 1678.96 and 1580.13 F/g is obtained for the A-24, 36, and 48 MoS₂ NC-S electrodes at a sweep rate of 5 mV/s. With reference to previous literatures, the CV studies shows that C_{sp} of material in WE decrease with increasing scan rate from 5 to 100 mV/s, since at lower sweep rate i.e, 5 mV/s, it allows to diffuse the ions into the interlayer between the active material, and therefore providing more active sites for the charge-transfer reactions [59-61]. The improved performance of MoS₂ NC-S electrodes can be attributed to the substantial contact area between the active material and the electrolyte, facilitating rapid ion diffusion and electron transfer. Notably, anodic, and cathodic peaks

currents were observed at + 0.5 V and + 0.2V respectively, corresponding to the oxidation and reduction process. resistance.

Additionally, it is worth mentioning that the shape of the CV curves undergoes minimal changes such as increase in the current value of the integrated area under the CV curve from 0.01 to 0.06 A for the sweep rates in the range from 5 to 100 mV/s. Further, MoS₂ NC-S electrode shows enhanced mass transportation and exhibits higher ionic conductivities, which can be inferred from EIS spectra where the resistance is low about 0.56 Ω , with respect to the general rule conductivity is inversely proportional to the resistance.

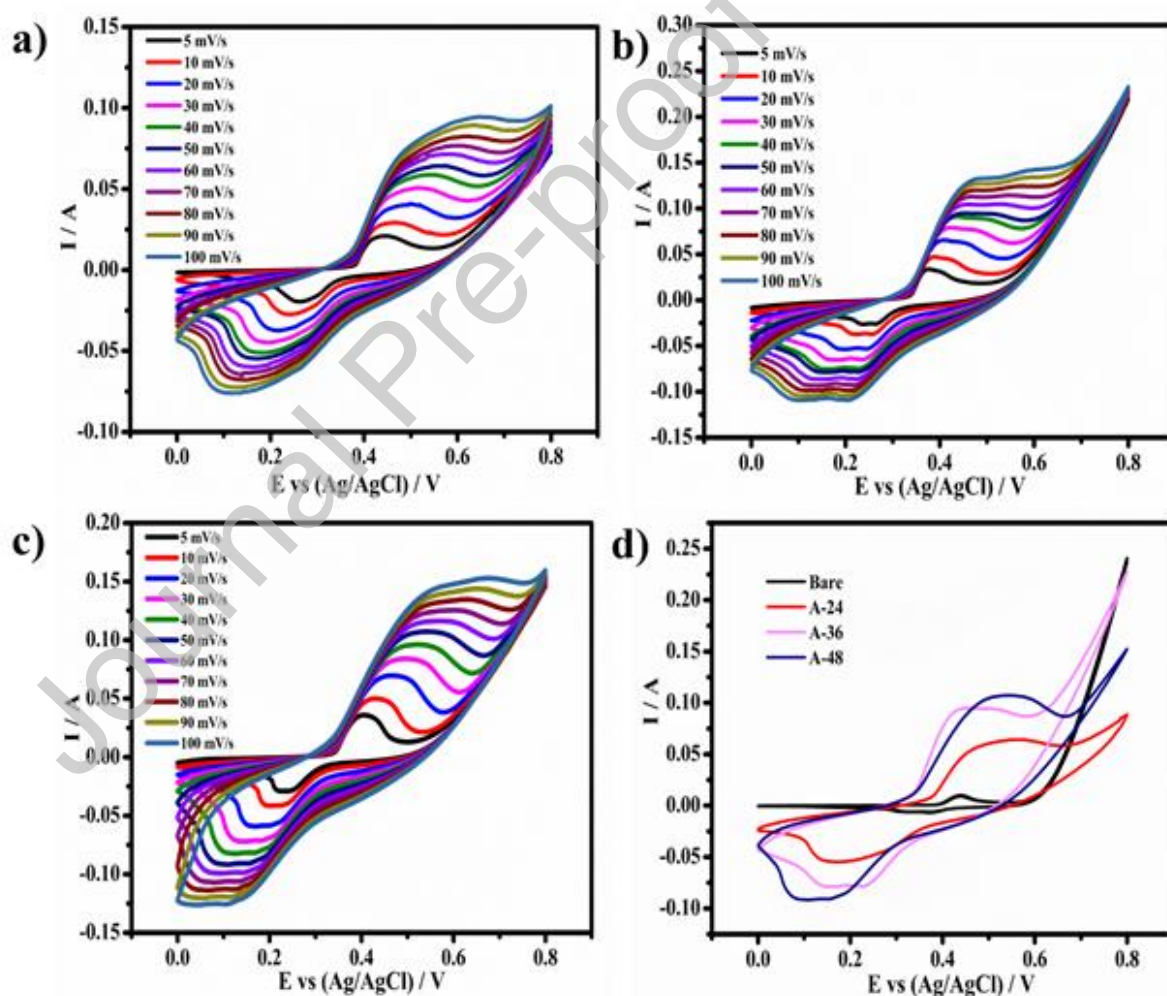


Figure 9. Cyclic voltammetry curves of the MoS₂ electrode material a) A-24, b) A-36, c) A-48 at various sweep rates from 5, 10-100 mV/s and d) Comparative cyclic voltammetry

curves of the electrode material in the potential range from 0 to 0.8 V at 50 mV/s sweep rate.

Figure 10 (a, g, d) illustrates the linear fit graph of Randle's- Sevcik plot (peak current vs $v^{1/2}$) of A-24, 36, and 48 MoS₂ NC-S electrodes shows a rectilinear trend of the peak currents as the sweep rate increases. The linear fit gives the value $b = 1$ for the redox peaks of all the three electrodes, which signifies the surface-controlled capacitive mechanism of the charge storage prevails. The b values obtained from the slope are 0.97, 0.98 and 0.91 for A-24, 36, and 48 MoS₂ NC-S electrodes respectively. It suggests that the charge storage process in all these electrodes is dominated by surface capacitive process. The effects of the capacitive mechanism are characterized by analysing CV curves at various sweep rates using the power law relation shown in equation (8) which is the graph against $\log(i)$ and $\log(v)$ from 10 to 100 mV/s for the oxidation peaks with sweep rates results in the expression,

$$i = av^b \quad (8)$$

where I is the current (A), v is the sweep rate (mV/s) and a , b are constants. By analysing $\log(i)$ vs $\log(v)$ for the oxidation and reduction peak currents, using Dunn's method [62]. The behaviour of the charge storage is defined by $b=1$, reveals the capacitive contribution and $b=0.5$ defines diffusion limited contribution. In addition, to the overall capacity, the ratio of contribution of the diffusion and surface-controlled process can be evaluated. The relative contribution of the different mechanism can be obviously calculated using Dunn's method which corresponds to the equation below;

$$i_p = k_1 v + k_2 v^{1/2} \quad (9)$$

which can be modified into

$$i_p/v^{1/2}=k_1v^{1/2}+k_2 \quad (10)$$

Where, k_1 and k_2 are constants. By calculating both k_1 and k_2 values, each process of the MoS₂ NC-S electrode can be identified. The linear fit of the $i/v^{1/2}$ vs $v^{1/2}$ yields the slope and intercept which is taken for k_1 and k_2 value. The surface-controlled contribution was high for all the three electrode materials compared to the diffusion effects which is shown in Figure 10 (b, e, and h). The contribution ratio of the surface-controlled process accounts as 2.26, 54.19, and 43.44% for A-24, 36, and 48 MoS₂ NC-S electrodes at 10 mV/s respectively. The prime capacitive contribution ratio of the A-36 MoS₂ NC-S electrode compared to the other two electrodes may be owing to its rich electroactive sites, over ion diffusion process. Therefore, the symmetrical redox peaks observed in the A-24, 36, and 48 MoS₂ NC-S electrodes reveal exceptional reversibility, while the expanded integrated area of the cyclic voltammetry curve indicates an enhanced redox reaction in the supercapacitor.

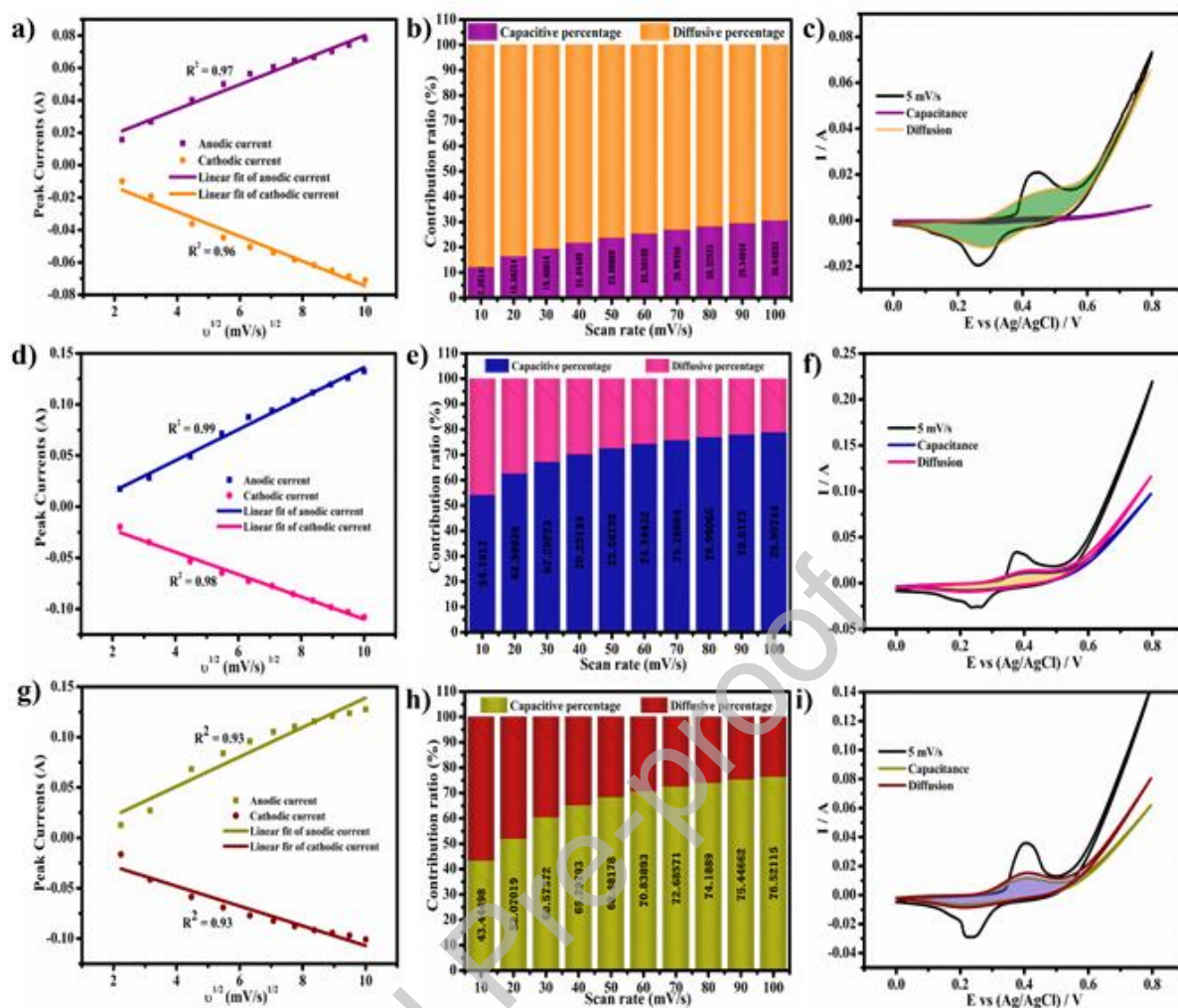


Figure 10. Randle's Sevcik plot and the relative contribution graph of the electrode materials (a-c) A-24, (d-f) A-36, and (g-i) A-48 MoS₂ NC-S at various sweep rates ranging from 10-100 mV/s.

3.7.2. Chronopotentiometry

Chronopotentiometry (CP) tests were performed on MoS₂ NC-S electrodes across a range of current densities from 1 to 5 A/g, while maintaining a potential between 0 to 0.45 V in a 1 M KOH electrolyte. As discoursed by Wang.et.al [63], the potential window achieved by the nickel foam during charge discharge is 0.45 V, which differs from the potential window in CV. This discrepancy is attributed to slower kinetics or capacitive limitations of the electrode material when subjected to constant current. The results are

presented in Figure 11 (a-c). There is a good correspondence between the CV curves and the charge – discharge curve provides further confirmation of the pseudocapacitive nature of the dual phase MoS₂ NC-S electrode material. The specific capacitance from CP profile was calculated using equation (2). Muhammad et al. [22] explains, electrode material having longer time of discharge exhibits a good charge storage behaviour and also states that the C_{sp} value diminishes, as the current densities increases [64]. Kour et al. [49] elucidates that the plateau region in the charge discharge curves correlates to the electrode-electrolyte interface developing in the redox reaction. The same trend has been observed in the as -prepared electrode material, whose discharge curves is used to compute C_{sp} respectively. At a very high current density (CD) of 5 A/g, the time of discharge is less (91.42 s) and in a lower CD of 1 A/g, better time of discharge (697.89 s) was detected. This could be attributed to the fact that at higher current densities, there interaction of active material with ions is limited. On the other hand, at lower current densities, the interaction of active material with ions is more. [65]. A C_{sp} of about 761.822, 1550.866, and 1036.288 F/g for the A-24,36 and 48 MoS₂ NC-S electrodes at 1 A/g of current density as depicted in Figure 11 (d). As stated in above reference, the electrode material that has longer discharge time has good capacitance value. An increase in the CD of about 2, 3, 4 and 5 A/g had resulted in low C_{sp} as shown in Figure 12 (b). Therefore, in comparison to all the electrode material, A-36 MoS₂ NC-S exhibits high C_{sp} with outstanding rate capability and longer discharge time. The obtained C_{sp} is higher than the reports (Table 2) of dual phase (1T/2H) MoS₂ NC-S. The resulted higher C_{sp} was mainly credited to the properties possessed by the nanostructures of MoS₂ NC-S with small amount of conductive binder (40 μ L of Nafion) which can help to decrease the contact resistance.

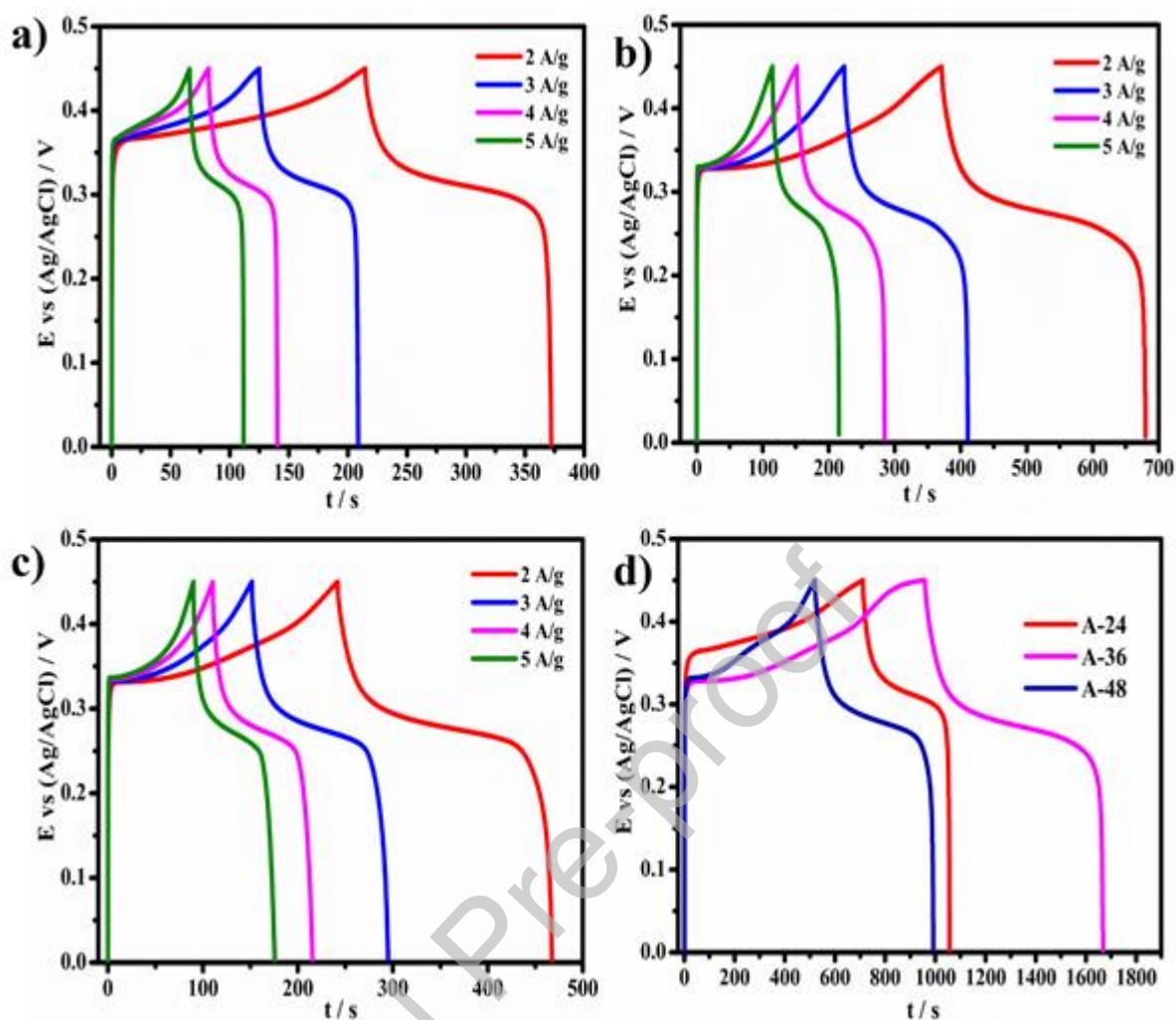


Figure 11. CP profiles of the electrode material a) A-24, b) A-36, c) A-48 MoS₂ NC-S at different current densities from 2 to 5 A/g, d) comparative CP profiles of the electrode material at current density of 1 A/g over the potential range of 0 to 0.45 V.

Figure 12 (a) depicts the specific capacitance with respect to scan rates ranging from 10 -100 mV/s of all the electrodes from the cyclic voltammetry curve and the graph of specific capacitance with respect to current density ranging from 1 to 5 A/g of the electrodes from the charge discharge curves is shown in Figure 12 (b). Moreover, cyclic stability tests were conducted for all three electrodes using charge-discharge profiles at a constant current density of 5 A/g over 1000 cycles. The results show a decrease in specific capacitance after 1000 charge-discharge cycles, with a retention of 94.5, 96.5, and

95.3 % of its initial specific capacitance as depicted in Figure 12 (c). Collectively, these studies provide evidence of the promising applicability of the prepared dual phase MoS₂ NC-S SC for electrochemical energy storage.

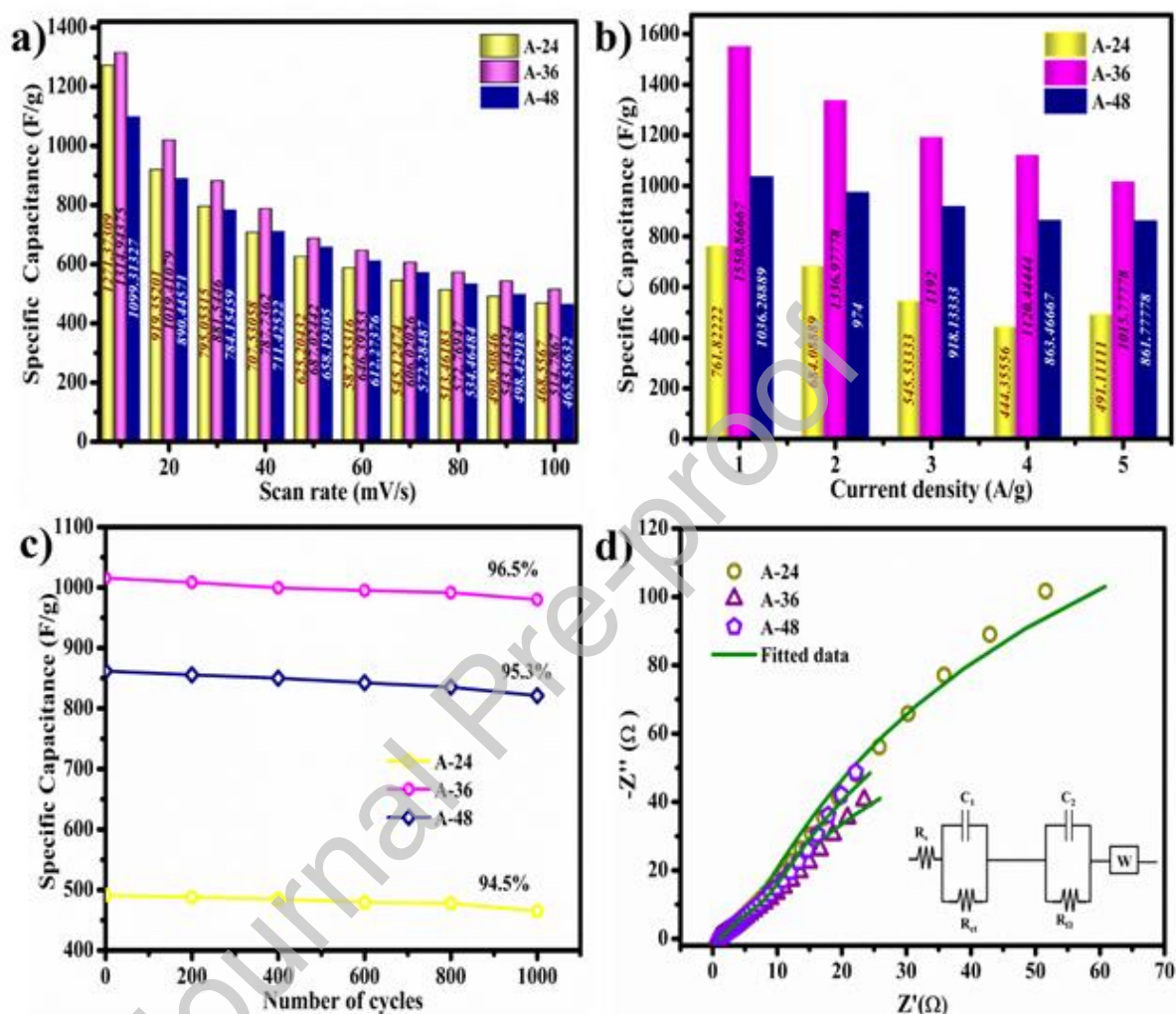


Figure 12. a) Effect of sweep rate on specific capacitance from CV profile, b) Effect of current densities on C_{sp} from CP profile, c) Specific capacitance vs number of cycles graph and d) The Nyquist plot with the fitted data of the electrode materials A-24, 36, and 48 MoS₂ NC-S. Inset image depicts the equivalent circuit of the electrochemical impedance spectra.

3.7.3. EIS

Electrochemical impedance spectroscopy (EIS) was conducted to gain insights into the fundamental behaviour, including charge transfer and capacitive nature of the prepared 1T/2H MoS₂ NC-S. Additionally, EIS was utilized to investigate the resistance and ion transport over a frequency range of 1 Hz to 100 kHz in a 1 M KOH solution. Figure 12 (d) displays the EIS data in a Nyquist plot with the fitted data using ZSimpWin software which is one of the important techniques for determining the electrode-electrolyte interaction and provide insights into the internal resistance of the active material. Nyquist plot was characterized by semicircle in a high frequency region and a straight line in a low frequency region, where the real part of the impedance is represented on x-axis and the imaginary part of the impedance depicted on the y axis. Muhammad et al. [22] explains that the ESR (equivalent series resistance) or the electrolyte resistance values can be found from the intersection point on x axis. Kour et al. [49] discuss that the charge transfer resistance (R_{ct}) is specified by semicircle diameter. The smaller diameter of the electrodes corresponds to lower resistance to the flow of ions, resulting in higher C_{sp} values. Conversely, larger diameters lead to higher resistance and lower C_{sp} values. Consistent with this trend, the Nyquist plot of MoS₂ NC-S electrodes displays a quasi-semicircle arc in the high frequency region followed by a straight line in the low frequency region. The presence of arc at high frequency region of 100 kHz, the region where lower real impedance value present is likely attributed to the R_{ct} caused by redox reactions occurring at the surface of the MoS₂ NC-S electrodes. At the low frequency region of 0.001 kHz, where the higher real impedance value lie is a parallel line running along the imaginary axis is observed, known as the Warburg line, indicating the capacitive behaviour of the as-prepared MoS₂ NC-S electrodes. The calculated values of solution resistance (R_s) and R_{ct} for the A-36 MoS₂ NC-S electrode are 0.5755 and 1.928 Ω respectively, indicating lower values compared to the other electrodes whose values of

R_s are 0.8953 Ω , 0.9825 Ω and R_{ct} are 3.119 Ω , 4.325 Ω for A-24 and 48 in the semicircle part as shown in Table 1. The presence of a 45° straight line, referred to as Warburg resistance (Z_w), is a consequence of the frequency – dependent ion diffusion kinetics of the electrolyte into the surface of the active material [66]. Therefore, the A-36 MoS₂ NC-S electrode has maximal ion diffusion rate that makes the migration of the ions in the electrolyte easier. EIS results evidently conclude that the A-36 electrode has a higher electrochemical conductivity as the A-36 electrode material possess low resistance value (0.57 Ω) compared to the other two electrodes whose values are 0.89 Ω and 0.98 Ω .

The equivalent circuit that corresponds to the Nyquist plot is depicted in Figure 12 (d). It includes R_s which represents solution resistance, R_{ct} which represents charge transfer resistance, Z_w which is Warburg impedance, R_o as ohmic resistance, C_1 associated to double layer capacitance and C_2 is linked to faradaic capacitance. The respective values for all the electrode material are tabulated below in Table 1.

Table 1. The Nyquist plot parameters of the equivalent circuit.

Electrode materials	Circuit parameters					
	R_s (Ω)	R_{ct} (Ω)	R_o (Ω)	C_1 (F)	C_2 (F)	Z_w (Ω)
A-24	0.8953	3.119	246.7	0.00023	0.00166	0.0143
A-36	0.5755	1.928	75.62	0.00352	0.00513	0.0214
A-48	0.9825	4.325	3.09	0.00411	0.00421	0.0243

The table is inserted under the section 3.7.3.

3.7.4. Ragone plot

In this study, the energy and power density of the dual phase MoS₂ NC-S electrodes were evaluated based on the CP profiles. The Ragone plot, shown in Figure 13 (b), illustrates the performance of the A-24, 36, and 48 MoS₂ electrode material. Among these, A-36 MoS₂ NC-S electrode exhibits the highest energy density of approximately 43.61 Wh/kg accompanied by a corresponding power density of about 225.50 W/kg was recorded at 1

A/g obtained from the CP profiles. As the CP profiles range from 1 to 5 A/g, there is a decrease in energy density, while the power density increases. Notably, the obtained energy and power density of the A-24, 36, and 48 MoS₂ NC-S electrodes significantly edge over the earlier reported values [67]. Future developments can be focussed on enhancing the energy and power density of the electrode material, by tailoring its compositions and morphologies [68].

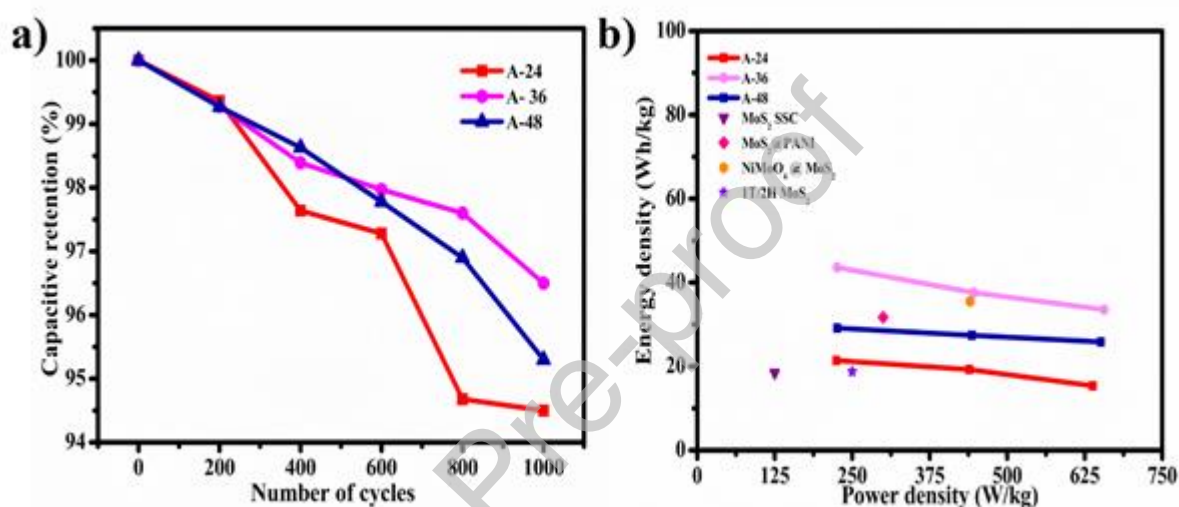


Figure 13. a) Capacitive retention graph and b) Ragone plot of A-24, 36, and 48 MoS₂ NC-S electrodes.

Table 2. Comparison of the capacitance, specific energy and power obtained in the present study with those of various metal sulphides nanostructures.

Electrode	Morphology	Electrolyte	Specific capacitance (F/g)	Energy density (Wh/kg)	Power density	Cycle Stability (%) / cycles	Ref.
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					(W/kg)		
Defect rich 1T-MoS ₂	Nanosheets	1 M KOH	379 @ 1 A/g	21.3	75	92 / 3000	[55]
Rare earth doped MoS ₂	Nanosheets and Nanospheres	-	231.38 @ A/g	-	-	81.50 / 5000	[56]
MoS ₂ -MnO ₂	Nanosheets	-	358 @ 5 mV/s	35.97	500	94.1 / 4000	[57]
Cu doped MoS ₂	Nanoarrays	-	353 @ 1 A/g	-	-	94 / 5000	[25]
MoS ₂ Nanodots	Nanosheets	6 M KOH	395 @ 1.5 A/g	22	100	86 / 1000	[58]
Biocarbon based MoS ₂	Nanospheres	2 M Na ₂ SO ₄	945 @ 0.5 A/g	74.9	3800	92 / 10000	[59]

1T/2H	Nano-	1 M KOH	1550.86 @	43.61	225.5	96.5 /	This
MoS ₂	corals		1 A/g		0	1000	wor
NC-S							k

3.7.5. Asymmetric cell configuration

The asymmetric SC device was fabricated by assembling 1T/2H MoS₂ NC-S as positive electrode to offer a high specific energy and activated carbon (AC) to perform as negative electrode for a source for high specific power [69]. A schematic illustration of the fabricated device is shown in Figure 14.

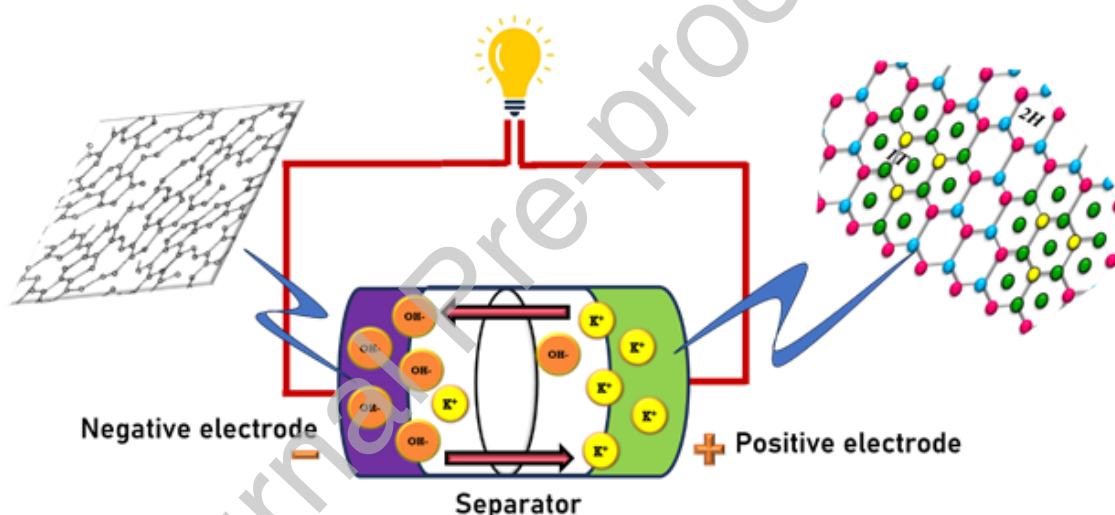


Figure 14. Schematic representation of the MoS₂ NC-S asymmetric two cell configuration.

The construction of the asymmetric device involves equalising the mass of the both positive and negative electrodes using equation 3. This process aimed to achieve a balanced charge distribution between the electrodes, thereby enhancing the energy storage performance. Figure 15 (a) shows the CV curves of the fabricated 1T/2H MoS₂ NC-S//AC device analysed under potential window of 0 to 1.8 V under various sweep rates ranging from 5 – 100 mV/s. Similarly, the CP profiles were also determined by different current densities in the potential range of 0 to 1.45 V shown in Figure 15 (b). At a modest

current density of 2 A/g, the asymmetric cell exhibited an almost symmetrical CP profile elucidating the efficient mass distribution of active materials between the two electrodes. The specific capacitance obtained was about 850.31 F/g at a CD of 2 A/g and the discharge time was about 154.12 s. The A-36 MoS₂ NC-S//AC were measured for the 'b' value using the Dunn's method. The resulting 'b' value was about 0.9, which showed a capacitive performance. As per the results obtained, the fabricated cell exhibited a pseudocapacitive charge storage mechanism by integrating both diffusive and capacitive charge storage. This device was examined for their contribution ratio at a lower scan rate of 5 mV/s and is illustrated in Figure 15 (c-e). The results revealed elevated capacitive rates and reduced diffusive rates. Moreover, the long-term cyclic stability of the asymmetric cell was assessed and illustrated in Figure 15 (f) through consecutive 5000 charge - discharge cycles at a constant current density of 10 A/g [70,71]. The device exhibited remarkable stability, retaining approximately 97 % of its initial capacitive performance and demonstrated favourable reversible characteristic.

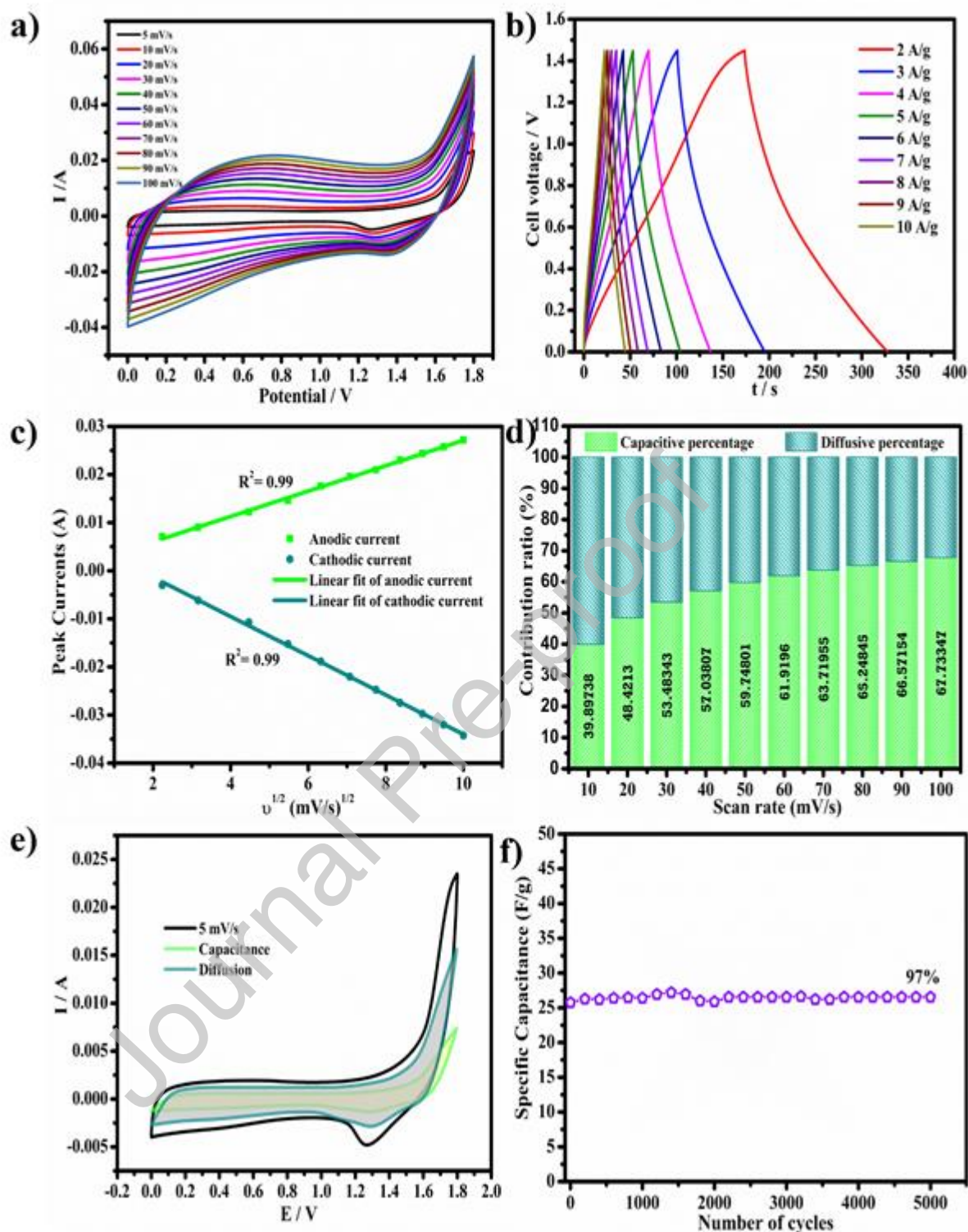


Figure 15. a) Cyclic voltammogram, b) Charge-discharge profile of the two-cell configuration, c) Dunn's method calculation R^2 graph, d, e) Contribution ratio graph, and f) Cycle stability of the 1T/2H MoS₂ NC-S//AC device.

The Nyquist plot of the asymmetric cell was measured before the cyclic stability of the 5000 cycles. The important parameters of an asymmetric cell are the specific energy and power which found to be 248.30 Wh/kg specific energy with specific power of 9881.65 W/kg at 2A/g of current density. Demonstrating the real time applicability of the asymmetric cell, the red-light emitting diode (R-LED) were powered by the high specific energy and power from the device. Figure 16 (a, b) demonstrates the efficient illumination of the R-LED for approximately 180 seconds.

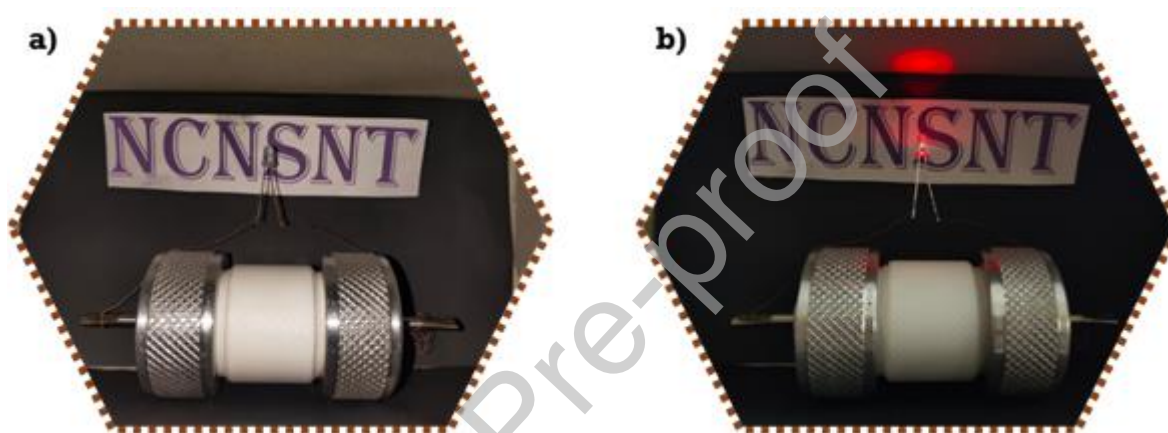


Figure 16. MoS₂ NC-S electrode sandwiched in Swagelok cell setup; a) Before charging, b) After charging.

4. Conclusion

In this study, dual phase (1T/2H) MoS₂ nano coral structures were successfully synthesized via a one-step hydrothermal technique using AMTH and thiourea. These structures were systematically investigated as electrode materials for supercapacitor applications. XRD analysis confirmed the presence of the dual phase, while FESEM revealed a nano coral-like morphology with an average particle size ranging from 0.3 to 0.7 μm in diameter. The comprehensive analysis of the electrochemical data evidently confirmed that the pseudocapacitive nature of the as-prepared MoS₂ NC-S electrode.

Among all the electrode materials, A-36 MoS₂ NC-S electrode material displays a remarkable C_{sp} of 1550.56 F/g as well as energy and power densities of 43.61 Wh/kg and 225.50 W/kg respectively. An asymmetric device was made with A-36 MoS₂, as they have outperformed other two electrodes, with exhibits specific capacitance of 850.31 F/g at a current density of 2 A/g, and possess 97% of retention ability even after 5000 cycles which remarks the sustained stability of the electrode material. Thus, it was made as a power source to illuminate a red LED. Collectively, these findings establish the excellent electrochemical properties of the dual phase MoS₂ NC-S and its potential applications. Therefore, the properties of the dual phase MoS₂ NC-S render it as a remarkable electrode material for the SC's and energy storehouse applications.

CRedit Authorship statement

M. N. Kavipriyah: Conceptualization, Methodology, Result Analysis, Data curation, Writing – Original draft and editing. S. Surender: Ideas, Review and Editing. S. Balakumar: Supervision and Investigation.

Availability of data

All generated or analyzed data during this current study are included in this published article.

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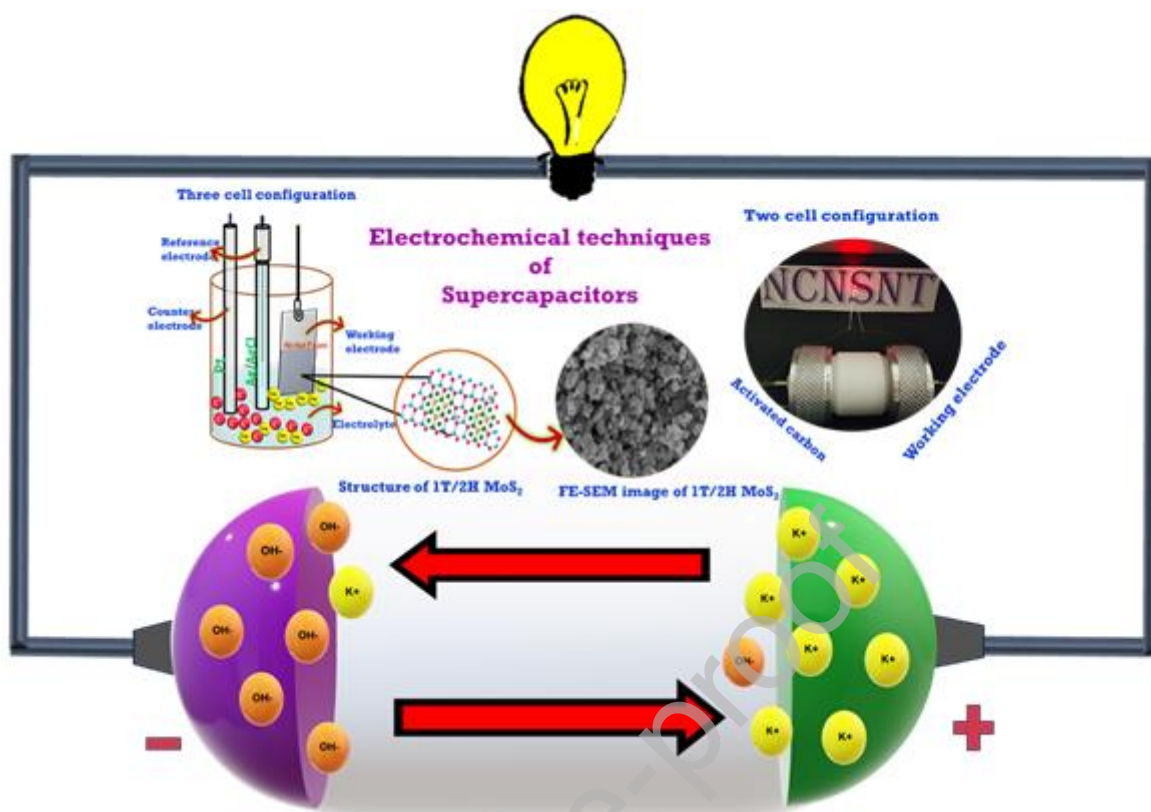
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Graphical Abstract



Graphical scheme of the prepared 1T/2H MoS₂ electrode for supercapacitor application

Declaration of interests

☒ 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.

☐ The authors declare the following financial interests/personal relationships which may be considered as potential competing interests.

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