Electronic Supplementary Material (ESI) for Sustainable Energy & Fuels.

Supporting Information (SI†)

Sonochemical synthesis of 2D-2D $MoSe_2$/Graphene nanohybrid electrode material for asymmetric supercapacitors

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Experimental Section

Materials used

All chemical reagents are analytical grade and deionized water was used throughout the process. Molybdenum Selenide ($MoSe_2$) and Poly(vinylidenefluoride) (PVdF) were procured from Sigma-Aldrich, USA. Natural graphite flakes (99.95%) was procured from Himedia, India. Isopropyl alcohol (IPA), Hydrogen peroxide (H_2O_2) (30 wt%) and N-methyl-2-pyrrolidone (NMP) were purchased from Merck, India. Black pearl carbon (Black Pearl®2000) (Activated carbon) was brought from Cabot Corporation, South Korea.

Physical characterization

The X-ray diffraction analysis (Rigaku, Ultima IV) was used to found the phase purity and structural properties of exfoliated $MoSe_2$ NS and $MoSe_2$/G nanohybrid in the range of 10° to 80° with nickel-filtered Cu-Kα radiation at an increment of 0.05˚. The confocal micro-Raman spectrometer (Renishaw RM 2000) was used to record the Raman spectra of $MoSe_2$ NS and $MoSe_2$/G nanohybrid under 10 mW Ar^+ laser at 785 nm. The field emission scanning electron microscopy (JSM, JEOL 7600F) was used to study the morphologies of few-layer $MoSe_2$ NS and $MoSe_2$/G nanohybrid and their elemental compositions were analysed by energy dispersive X-ray spectroscopy (EDX).

Electrochemical measurements

The electrochemical analyzer (VSP, Bio-Logic, France) was used to study $MoSe_2$ and $MoSe_2$/G nanohybrid electrodes performances in a three-electrode configuration at ambient temperature in 6 M KOH solution. The working electrode was fabricated by mixing 80% of the $MoSe_2$ NS (or) $MoSe_2$/G nanohybrid, 15% of black pearl carbon as the conductive carbon and 5% polyvinylidene difluoride in N-methyl pyrrolidine as the binder to form a homogeneous paste. This paste was coated on 1 cm^2 nickel strip and dried at 70 °C for 12 h in a vacuum oven. Platinum electrode and a saturated calomel electrode (SCE) were used as the counter and reference electrodes, respectively. The mass loading of both electrodes was ~5.0 mg. Cyclic voltammograms were recorded in the potential window of -0.2 to +0.4 V at various scan rates, viz., 5, 10, 25, 50 and 100 mV s^{-1}. Galvanostatic charge-discharge curves were recorded at various current densities viz., 1, 3, 5, 7 and 10 A g^{-1} in the potential range of -0.2 to +0.4 V. Electrochemical impedance measurements were recorded in the frequency range of 0.01 Hz – 100 kHz at the open circuit potential with an AC amplitude of 5 mV.

Fabrication of asymmetric supercapacitor (ASC) devices

The ASC devices were fabricated using $MoSe_2$/G nanohybrid as the positive electrode and activated carbon as the negative electrode and the electrospun PVDF polymer electrolyte membrane soaked in 6 M KOH as the electrolyte as well as the separator.

From the charge-discharge curves, the specific capacitance (C_{sp}) of ASC device was calculated using the following equation;

$$C_{sp} = \frac{i \times \Delta t}{\Delta V \times m} \tag{1}$$

where ΔV is the potential drop during discharge (V), Δt is the discharge time (s) and m is the mass of the active electrode material (g). To obtain the charge balance between the two electrodes, an optimum mass balances of both the positive and negative electrodes were calculated by using the following equation;

$$\frac{m_+}{m_-} = \frac{C_- \times \Delta V_-}{C_+ \times \Delta V_-} \tag{2}$$

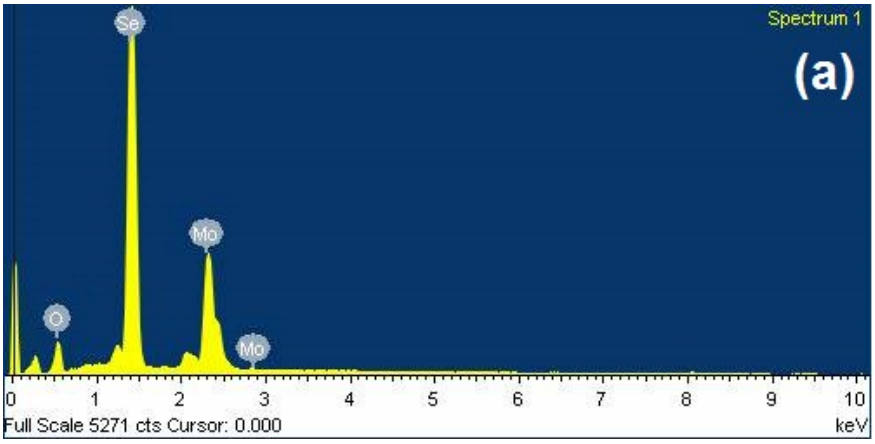
where C_+/C_- represents the specific capacitance of the positive and negative electrodes, m is the mass of active material, and $\Delta V_+/\Delta V_-$ is the potential window of the positive and negative electrodes in the three-electrode system.

The electrochemical studied such as cyclic voltammetry and galvanostatic charge-discharge studies were performed for the assembled ASC device. From the charge-discharge curves, the energy density (E) and power density (P) of the fabricated ASC devices were calculated using the following equation;

$$E = \frac{C_{sp} \times V^2}{2} \tag{3}$$

$$P = \frac{E}{t} \tag{4}$$

where, C_{sp} is the specific capacitance of ASC device (F g^{-1}), V is the potential window of ASC device, and Δt is the discharge time.



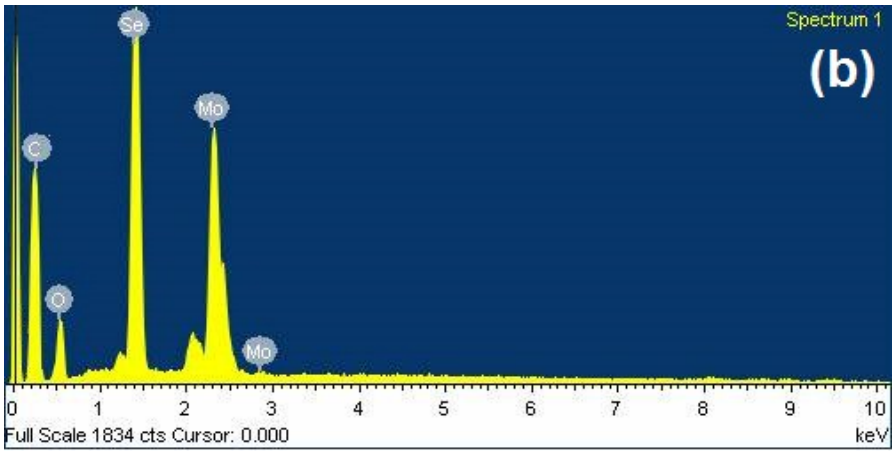


Fig. S1 EDX of (a) $MoSe_2$ NS (30 min) and (b) $MoSe_2$-G nanohybrid.

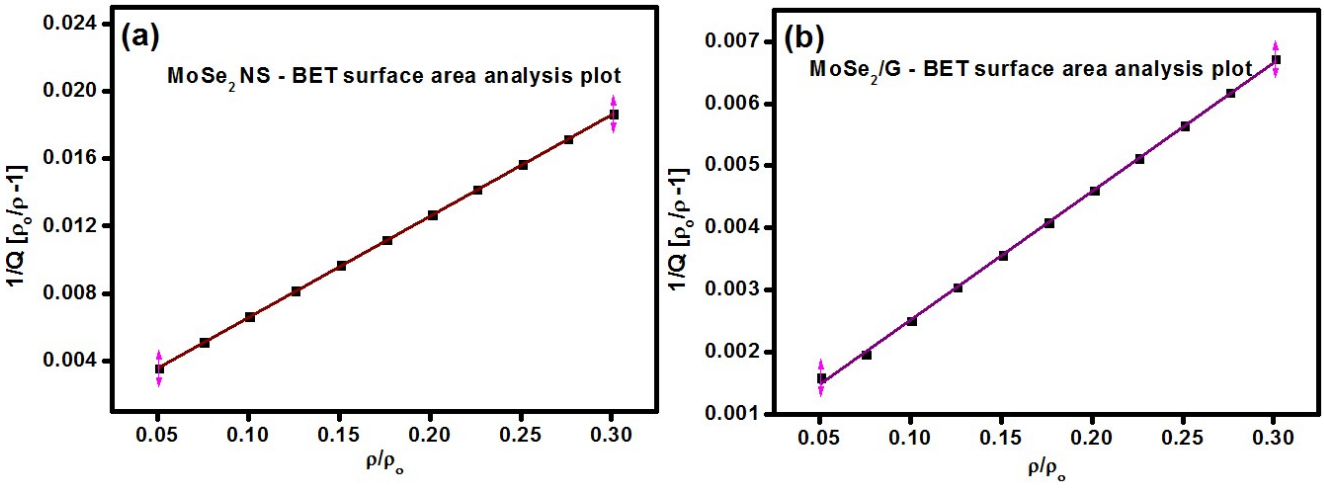


Fig. S2 BET surface area analysis plot for (a) $MoSe_2$ NS and (b) $MoSe_2$/G nanohybrid

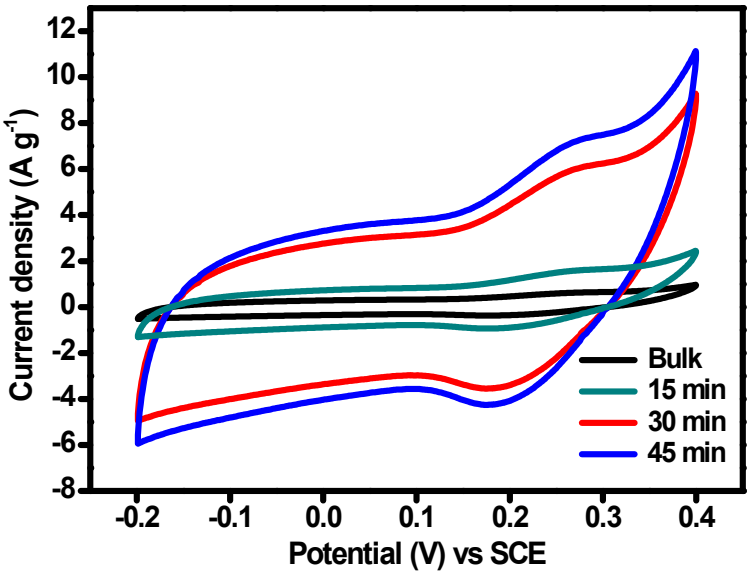


Fig. S3 CV curves of bulk $MoSe_2$ and exfoliated $MoSe_2$ under various sonication times at constant scan rate of 25 mV s^{-1}.

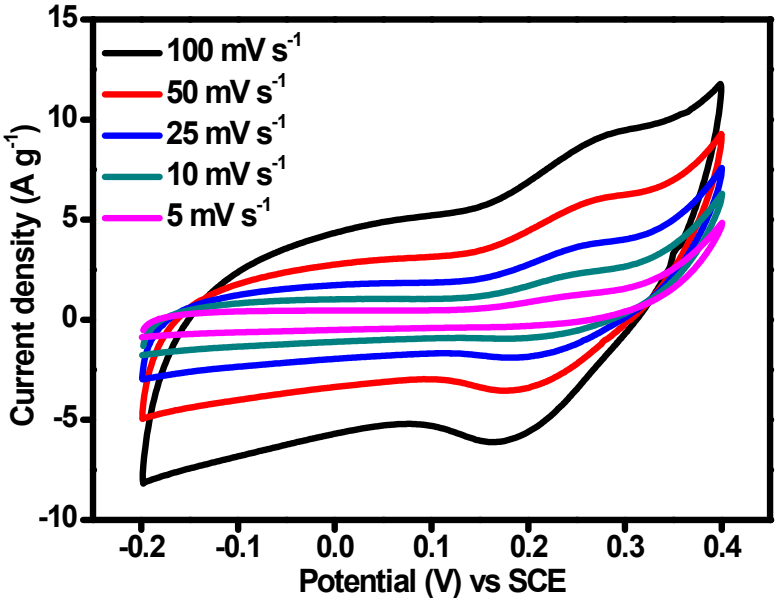


Fig. S4 CV curve of exfoliated $MoSe_2$ NS based electrode at various scan rates (5 to 100 mV s^{-1}) in the potential window of -0.2 to +0.4 V vs SCE.

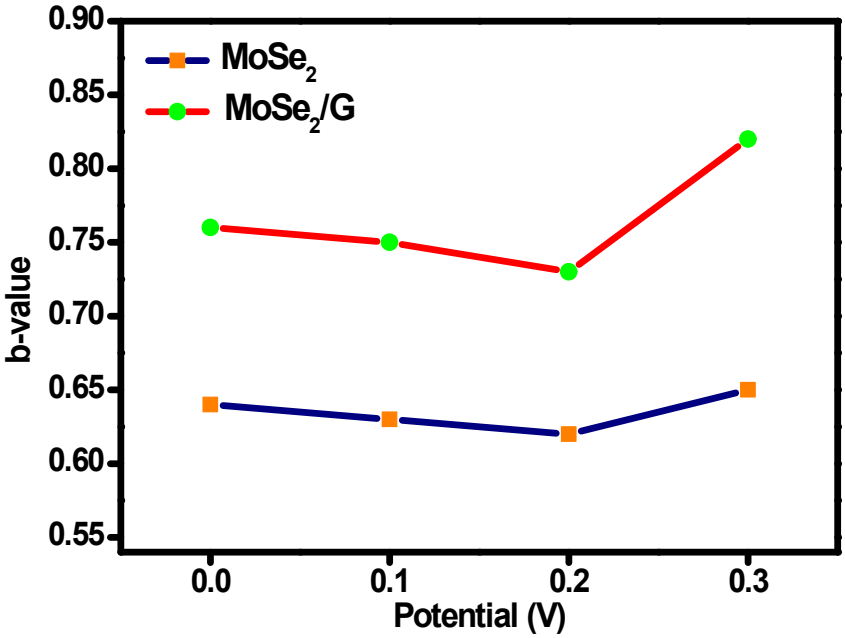


Fig. S5 b-values as a function of potential for $MoSe_2$ NS and $MoSe_2$/G nanohybrids.

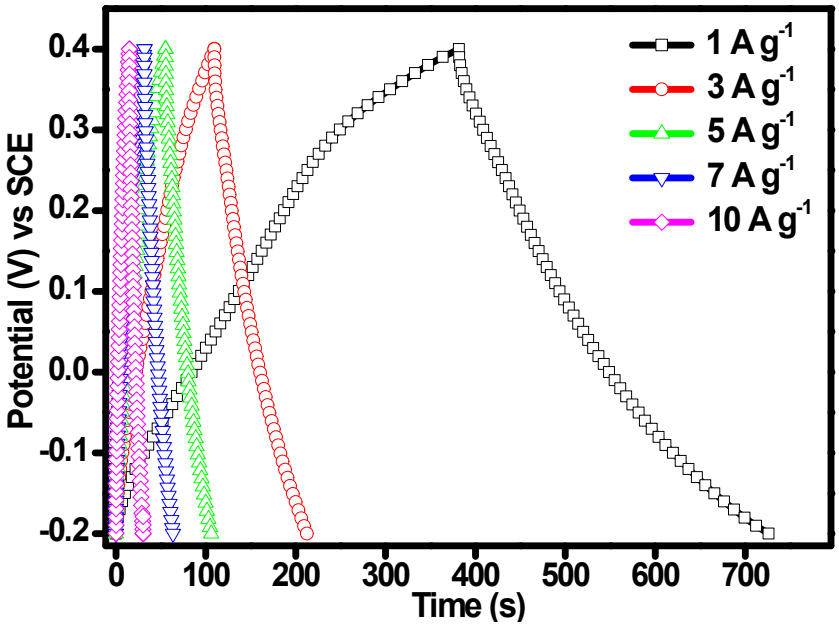


Fig. S6 Galvanostatic charge-discharge curves exfoliated $MoSe_2$ NS based electrode at various current densities (1 to 10 A g^{-1}).

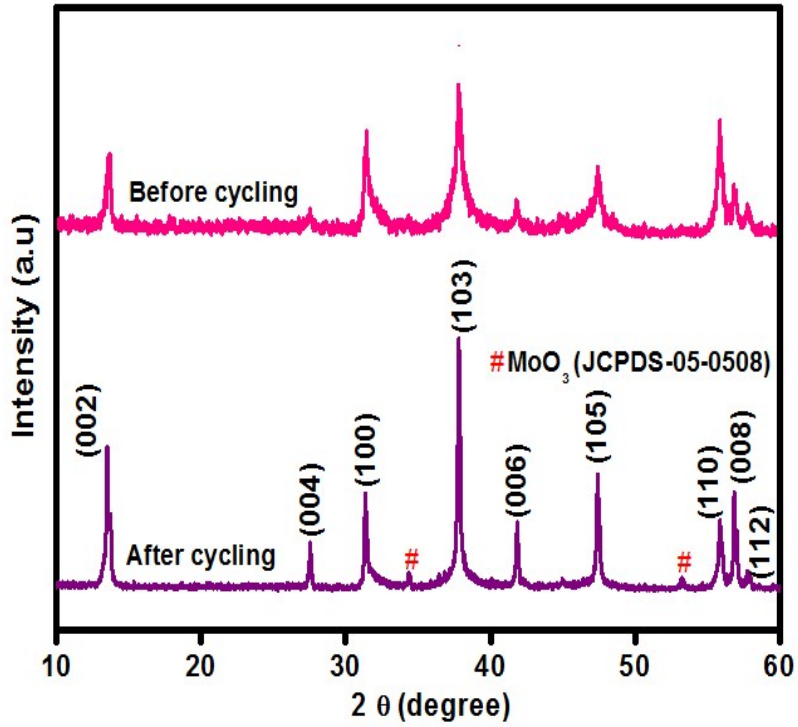


Fig. S7 XRD analysis of exfoliated $MoSe_2$ NS electrode before and after 3000 cycles.

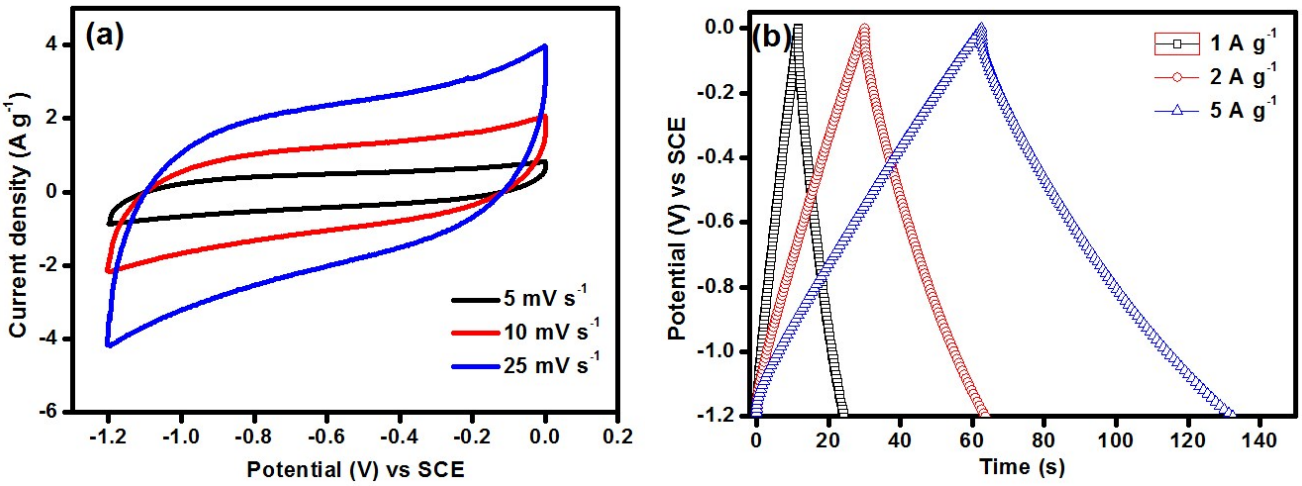


Fig. S8 (a) CV curves of AC at various scan rates, (b) Galvanostatic charge-discharge curves of AC at various current densities.

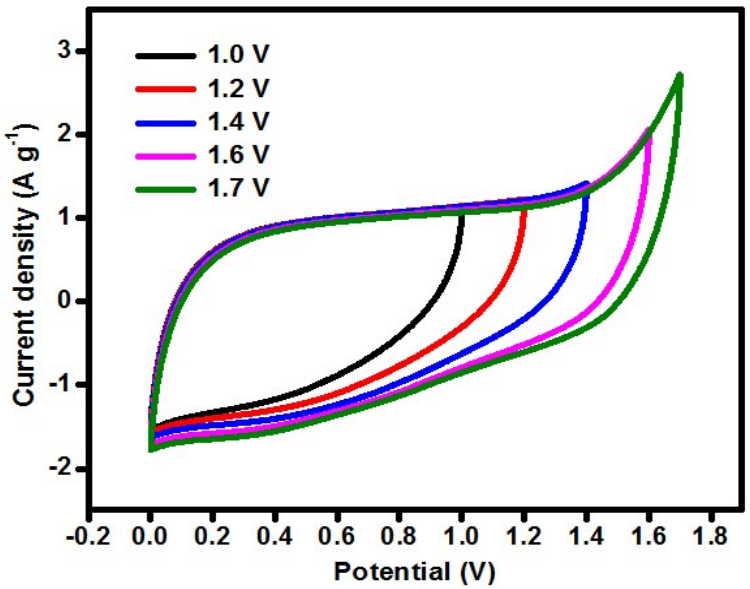


Fig. S9 CV curve of $MoSe_2$-G||AC ASC device at various working potential windows.

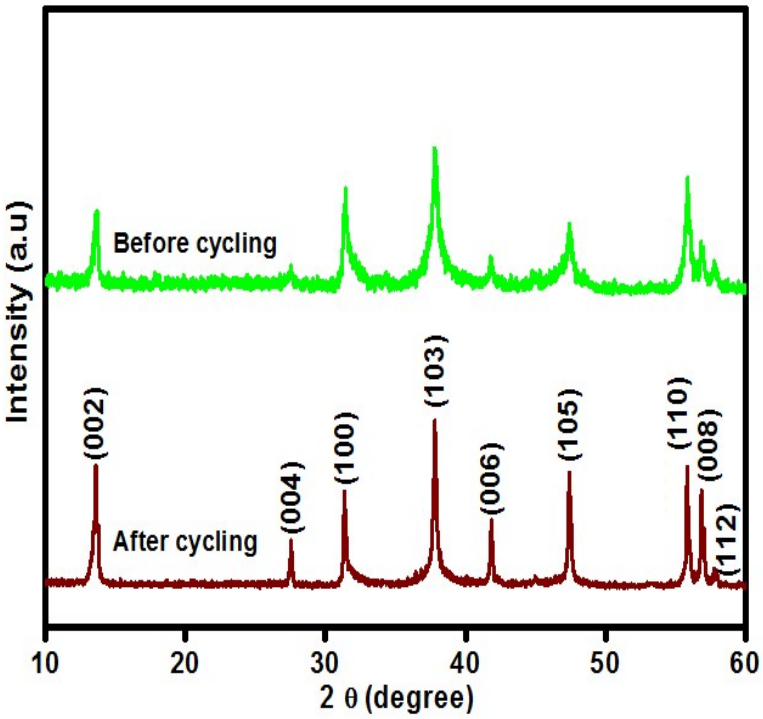


Fig. S10 XRD analysis of $MoSe_2$/G||AC device before and after 3000 cycles.