

Supporting Information

Ti₃C₂T_x MXene/Alginic Acid-Derived Mesoporous Carbon Nanocomposite as a Potential Electrode Material for Coin-Cell Asymmetric Supercapacitor

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Instrumental Characterization

The Ti₃C₂T_x/Mesoporous Carbon nanocomposites were characterized using various spectroscopic and microscopic techniques. Powder XRD patterns were recorded on an X'pert pro diffractometer, PANalytical using Cu K_α radiation ($\lambda = 1.5406 \text{ \AA}$, 40 kV, 40 mA) in the 2 θ range of 5-90° with the scan rate of 2° min⁻¹. Micro-Raman Spectrometer (LabRAM HR Evolution HORIBA France) was used for Raman analysis, using powder samples on a glass substrate. An oxixus laser of 633 nm wavelength (having max. power of 100 mW) was used throughout the complete measurement. The morphology of the prepared nanocomposites was investigated by using scanning electron microscopy (SEM) on a Quanta 200 FEG FE-SEM and transmission electron microscopy (TEM) on a JEOL-1010 transmission electron microscope operated at an acceleration voltage of 20 kV and 100 kV respectively. Whereas, high angle annular dark field-scanning transmission electron microscopy (HAADF-STEM) and elemental analysis mapping were conducted on an FEI make Tecnai TEM T-20 operating at 200 kV with LaB₆ filament fitted with Gatan Digital camera (bottom mount) with a resolution of 2K. The specific surface area of the sample was calculated by the Brunauer-Emmet-Teller (BET) method using a Quantachrome Nova-1000 surface analyzer. The pore size distribution was determined from the adsorption branches of isotherms through Barrett-Joyner-Halenda (BJH) method. Thermo Gravimetric Analysis (TGA) were done with a STA 2500 Regules instrument at a temperature range, RT-1100 °C in an inert atmosphere. X-ray photoelectron spectroscopy (XPS) measurements were done with a Thermo K-5 Alpha XPS instrument at a pressure better than 1×10^{-9} Torr with a pass energy of 50 eV, electron take-off angle of 60° and an overall resolution of ~1 eV using monochromatic Al K_α (source, $h\nu = 1486.6 \text{ eV}$). The spectra were fitted using a combined polynomial and Shirley-type background function.

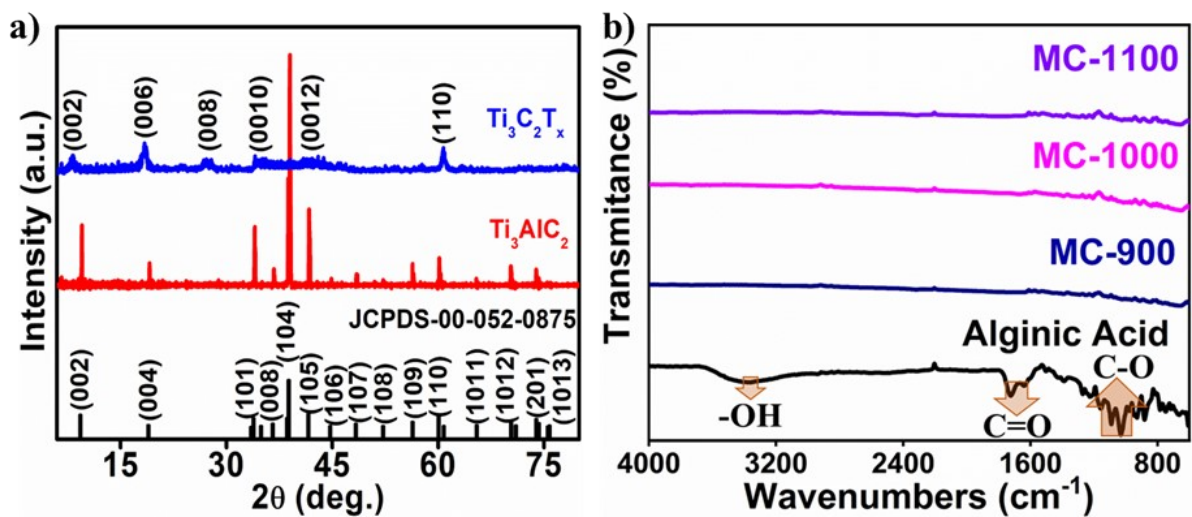


Figure S1. (a) XRD patterns of Ti_3AlC_2 and $Ti_3C_2T_x$; (b) FTIR spectra of the Alginic acid and MC-T.

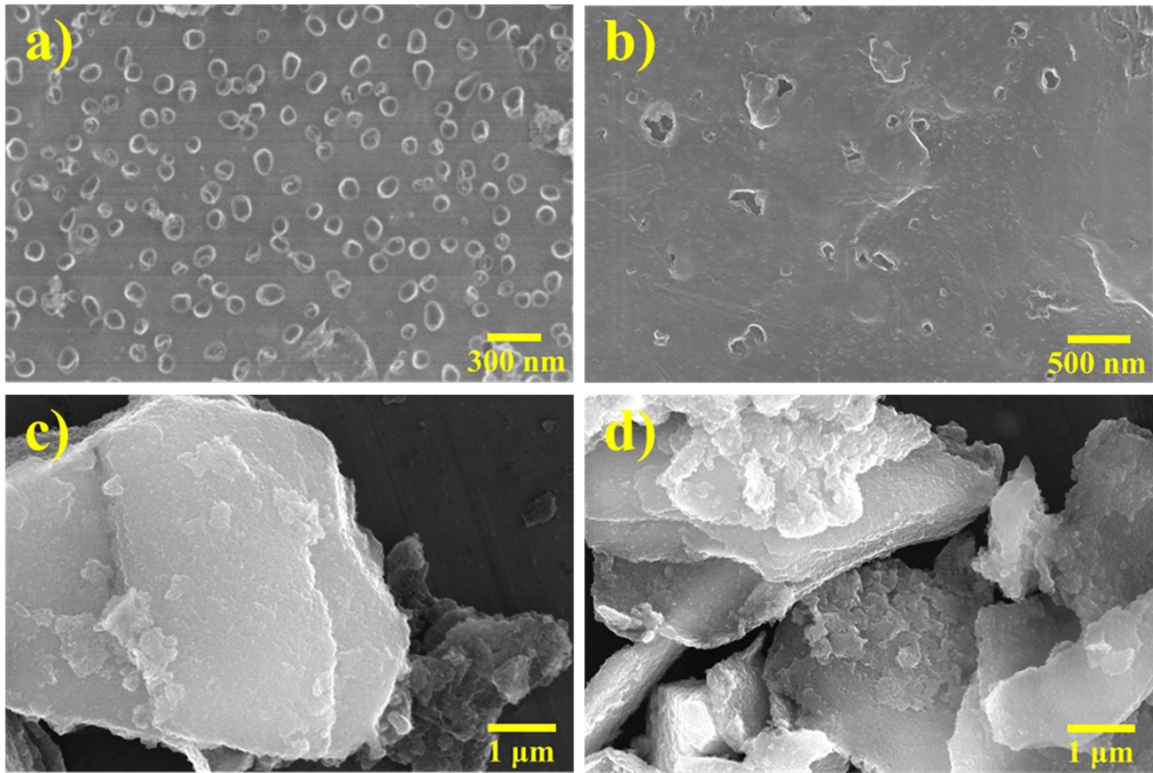


Figure S2. SEM images of (a) MC-900, (b) MC-1100, (c) Ti₃C₂T_x/MC-5 and (d) Ti₃C₂T_x/MC-1 nanocomposite.

Table S1. Titanium, Carbon, Oxygen and Fluorine content (wt% and at%) obtained from EDS analysis of all synthesized materials

| Materials | Carbon | | Oxygen | | Titanium | | Fluorine | |
|---|--------|------|--------|------|----------|------|----------|------|
| | Wt % | At % | Wt % | At % | Wt % | At % | Wt % | At % |
| Alginate Acid | 47.6 | 54.7 | 52.4 | 45.3 | | | | |
| MC-900 | 83.7 | 84.1 | 16.3 | 15.9 | | | | |
| MC-1000 | 88.5 | 88.7 | 11.5 | 11.3 | | | | |
| MC-1100 | 87.8 | 86.5 | 13.2 | 13.5 | | | | |
| Ti ₃ C ₂ T _x | 15.8 | 16.0 | 2.9 | 4.0 | 66.6 | 63.0 | 14.7 | 17.0 |
| Ti ₃ C ₂ T _x /MC-9 | 78.1 | 77.2 | 17.5 | 16.8 | 3.1 | 3.8 | 1.3 | 2.2 |
| Ti ₃ C ₂ T _x /MC-5 | 43.6 | 46.1 | 19.6 | 19.4 | 30.1 | 29.2 | 6.7 | 5.3 |
| Ti ₃ C ₂ T _x /MC-1 | 9.2 | 10.1 | 25.6 | 27.1 | 56.4 | 53.2 | 8.8 | 9.6 |

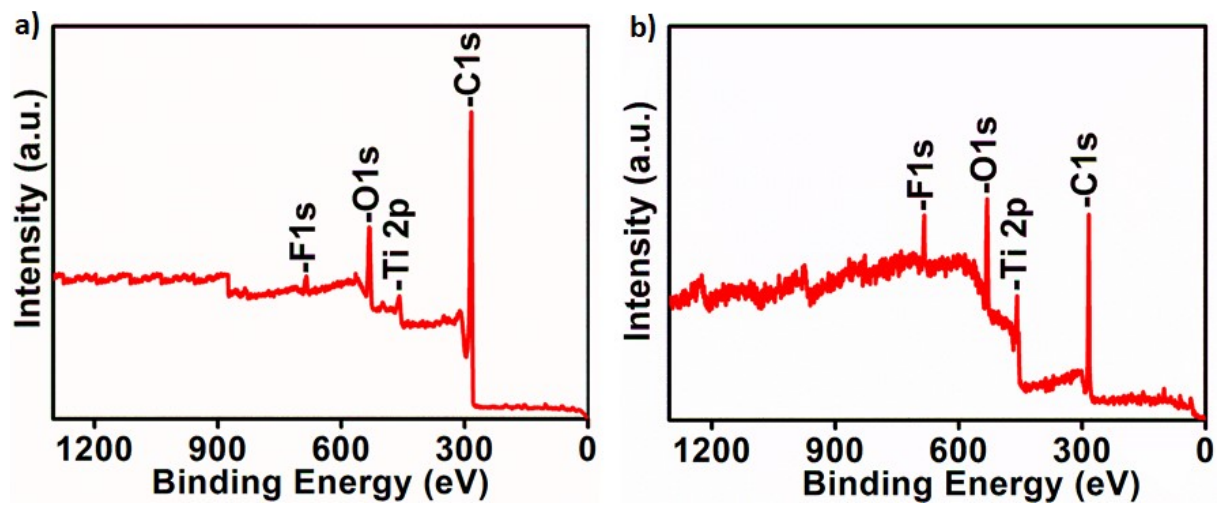


Figure S3. XPS survey spectra of $\text{Ti}_3\text{C}_2\text{T}_x/\text{MC-9}$ and $\text{Ti}_3\text{C}_2\text{T}_x$.

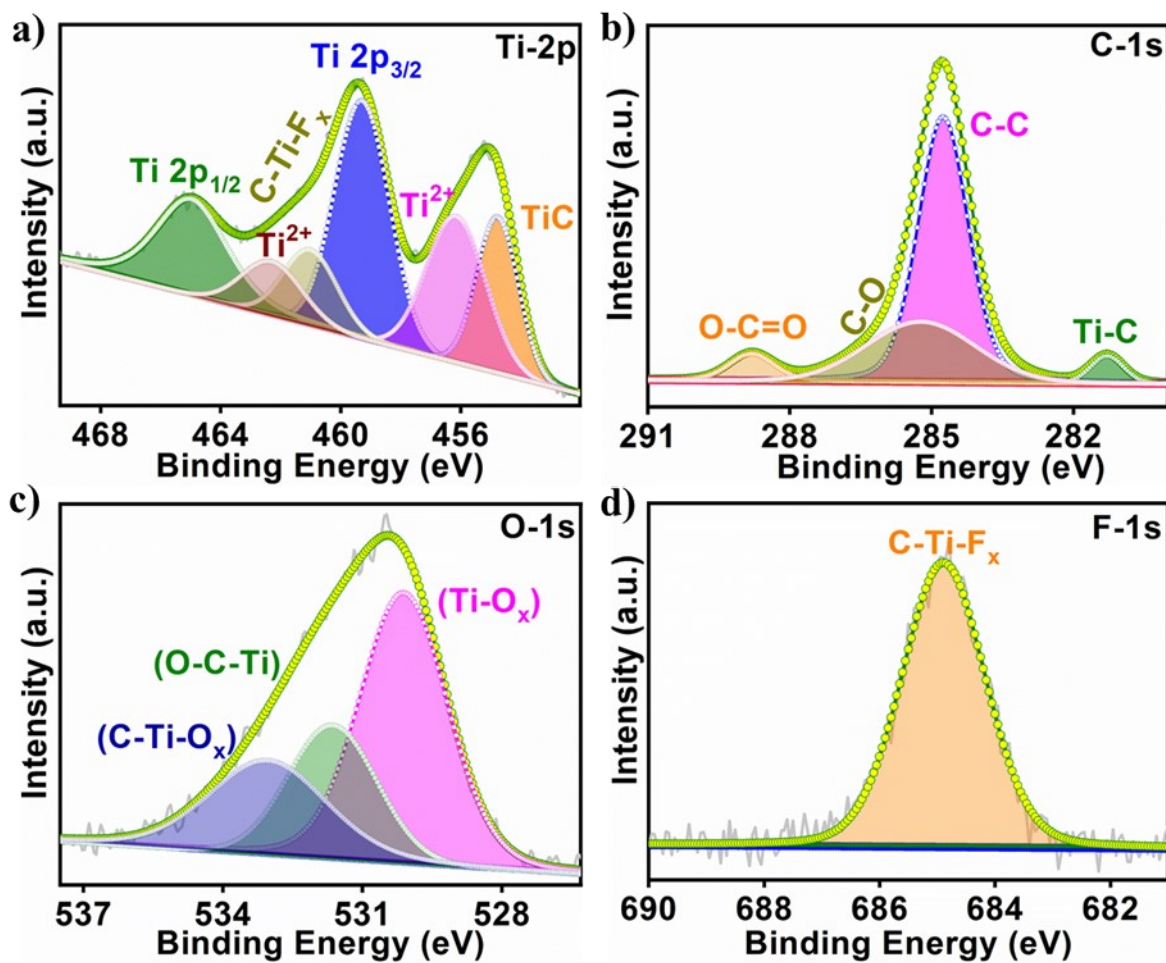


Figure S4. XP spectra (a) Ti 2p, (b) C 1s, (c) O 1s and (d) F 1s core levels of MXene ($\text{Ti}_3\text{C}_2\text{T}_x$).

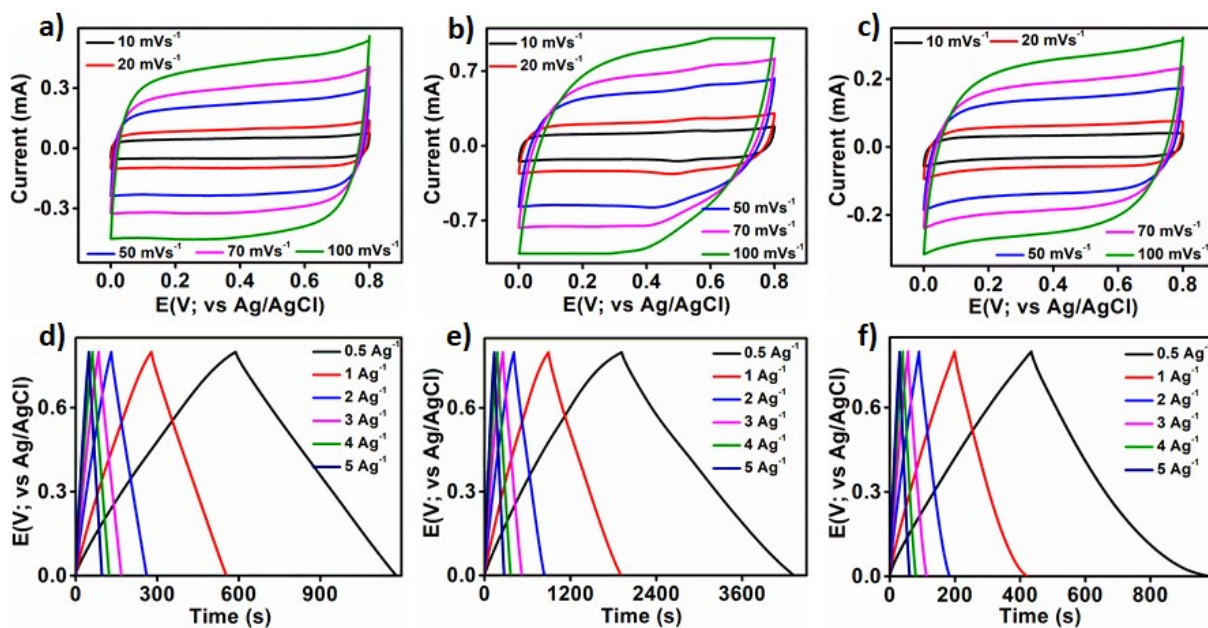


Figure S5. (a-c) CV and (d-f) GCD curves at various scan rates and current densities recorded for MC-900, MC-1000 and MC-1100 in 0.5 M H₂SO₄ acidic electrolyte.

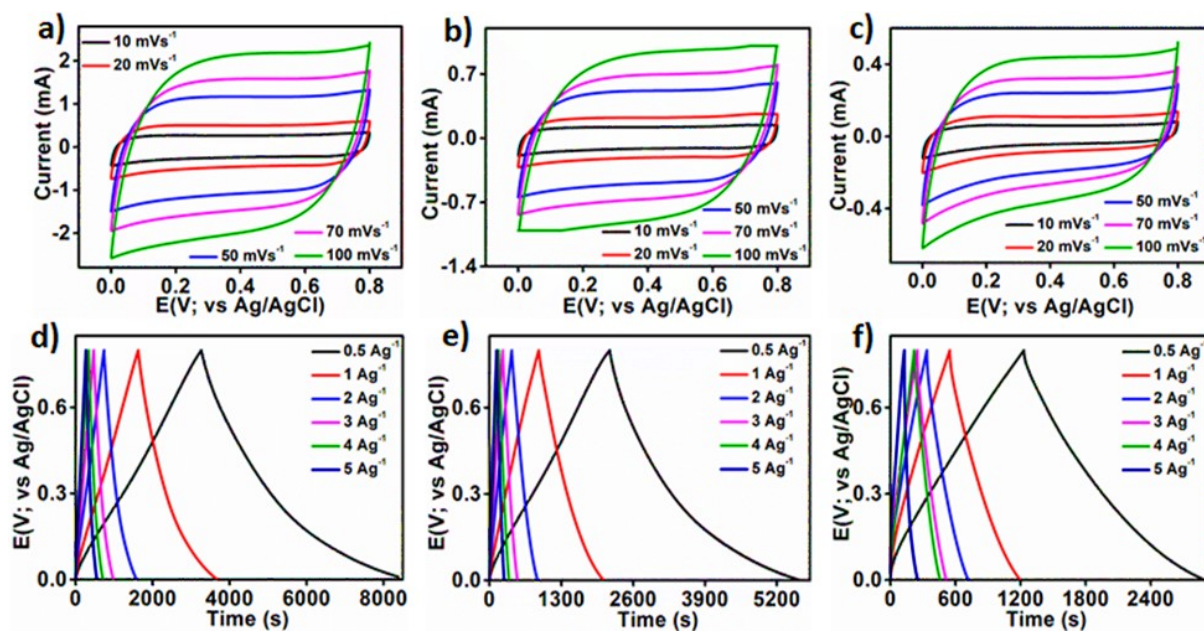


Figure S6. (a-c) CV and (d-e) GCD curves at various scan rates and current densities recorded for Ti₃C₂T_x/MC-9, Ti₃C₂T_x/MC-5 and Ti₃C₂T_x/MC-1 in 0.5 M H₂SO₄ acidic electrolyte.

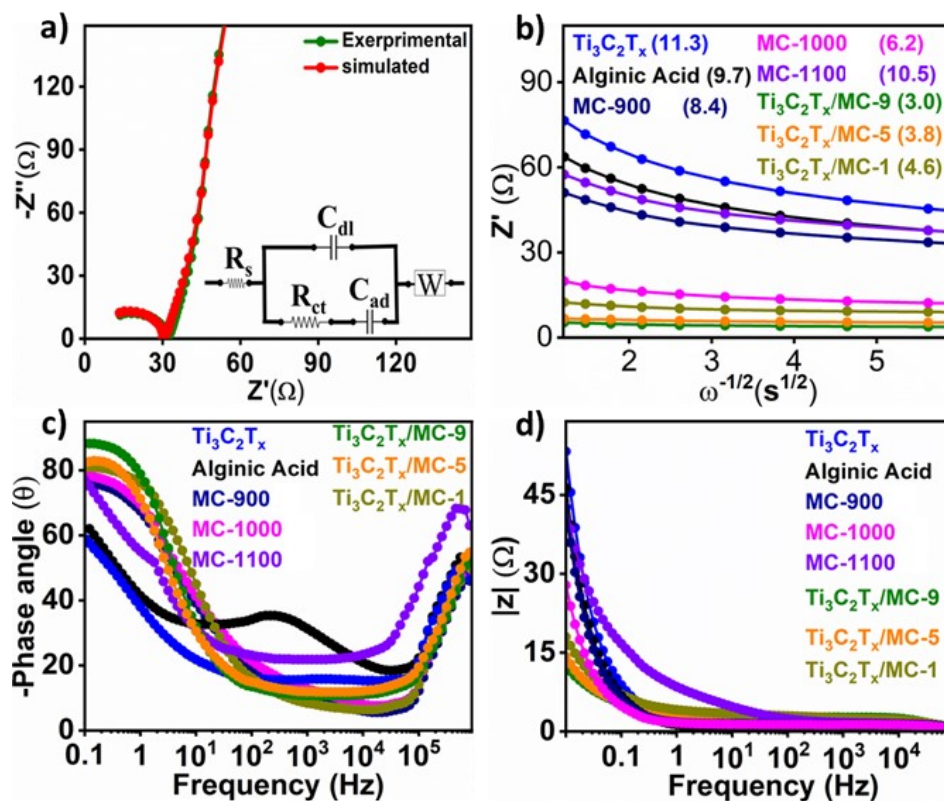


Figure S7. (a) Experimental and fitted EIS curves for $\text{Ti}_3\text{C}_2\text{T}_x/\text{MC-9}$ electrode and the inset shows corresponding equivalent circuit diagram; (b) linear fit of Z' data against the square root of the period (ω) in the high-frequency region; Bode plot (c) -phase angle vs. frequency, (d) $|z|$ vs. frequency.

Table S2. The transport properties of various electrodes obtained from simulated impedance spectra in Fig. (6e and 6f).

| Materials | $R_s(\Omega)$ | $R_{ct}(\Omega)$ | $Z_w(m\ \Omega\ s^{-1/2})$ | $C_{ad}(mF)$ |
|-------------------|---------------------------------|------------------------------------|--|--------------------------------|
| Alginic Acid | 13.5 | 52.0 | 9.7 | 0.04 |
| MC-900 | 13.5 | 45.5 | 8.4 | 0.03 |
| MC-1000 | 12.8 | 39.1 | 6.2 | 0.6 |
| MC-1100 | 13.4 | 50.1 | 10.5 | 0.9 |
| $Ti_3C_2T_x$ | 15.4 | 56.1 | 11.3 | 0.06 |
| $Ti_3C_2T_x/MC-9$ | 12.8 | 31.6 | 3.0 | 0.4 |
| $Ti_3C_2T_x/MC-5$ | 12.8 | 34.1 | 3.8 | 0.6 |
| $Ti_3C_2T_x/MC-1$ | 13.5 | 35.6 | 4.6 | 0.7 |

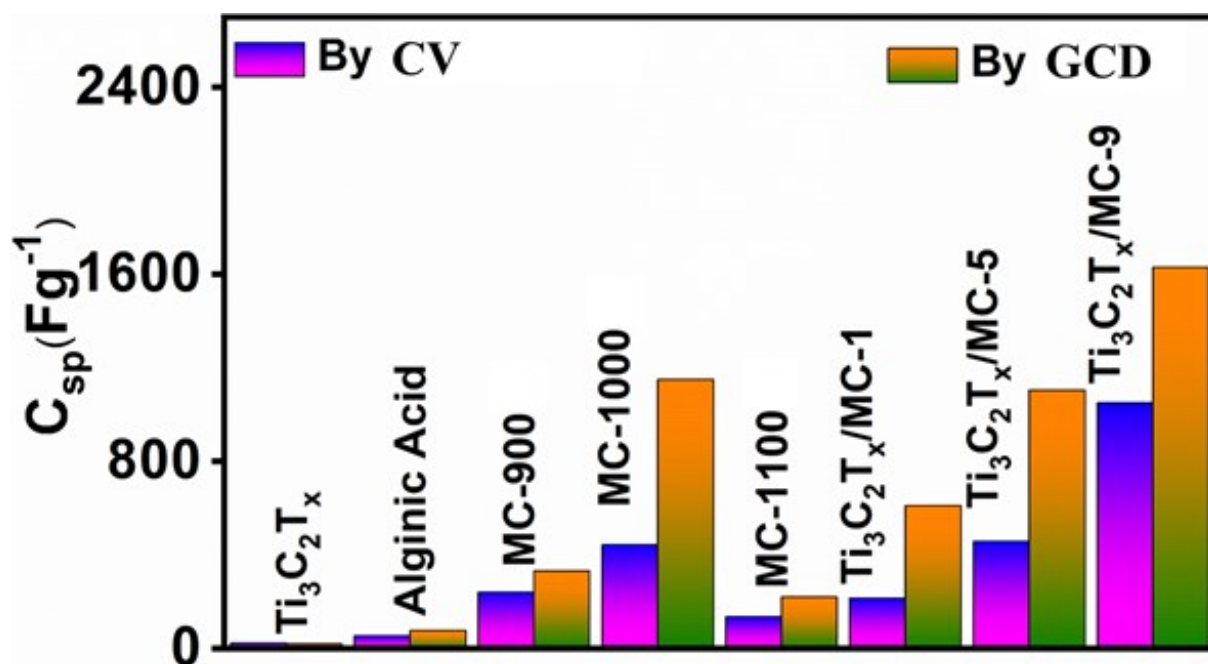


Figure S8. The specific capacitance for various synthesized nanocomposites from cyclic voltammetry (blue-pink bar) and galvanostatic charge-discharge (green-orange bar) at sweep rate and current density of 100 mV s⁻¹ and 1 A g⁻¹ respectively.

Table S3 The calculated specific capacitance in half-cell configuration for various active electrode materials.

| Materials | Specific Capacitance (F g ⁻¹) | |
|---|--|--|
| | by CV @ scan rate of 100 mV s ⁻¹ | by GCD @ current density of 1 A g ⁻¹ |
| Alginic Acid | 54 | 76 |
| MC-900 | 240 | 331 |
| MC-1000 | 442 | 1149 |
| MC-1100 | 134 | 219 |
| Ti ₃ C ₂ T _x | 22 | 18 |
| Ti ₃ C ₂ T _x /MC-9 | 1049 | 1629 |
| Ti ₃ C ₂ T _x /MC-5 | 457 | 1102 |
| Ti ₃ C ₂ T _x /MC-1 | 214 | 610 |

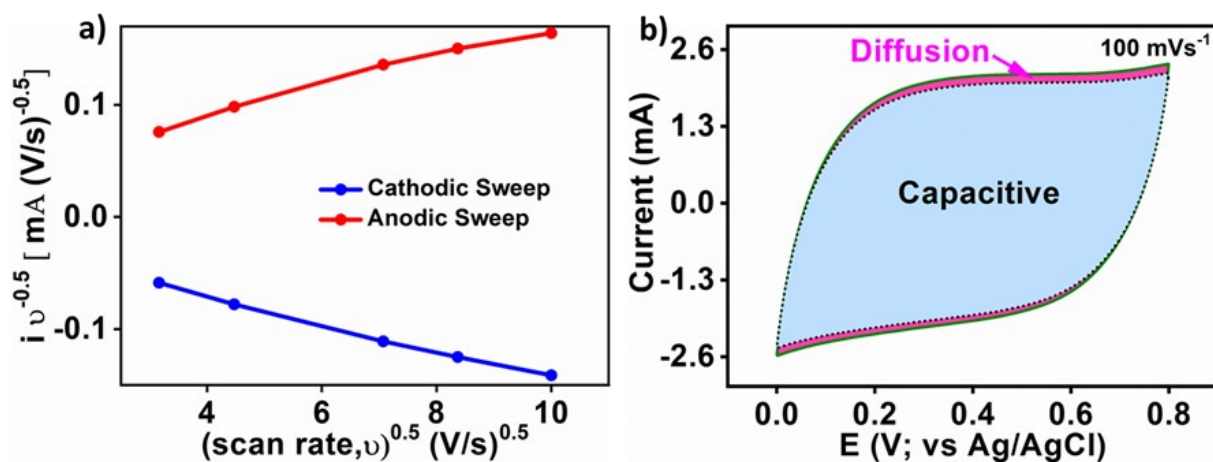


Figure S9. (a) The plot of $i/(\text{scan rate}, v)^{0.5}$ against $v^{0.5}$ for both anodic and cathodic voltammetric sweeps under an acidic medium to evaluate the value of k_1 and k_2 in equation 10 (from the main text); (b) CV curves at 100 mV s $^{-1}$, showing the surface and diffusion-controlled charge storage contributions.

Table S4. The calculated specific capacitance of $\text{Ti}_3\text{C}_2\text{T}_x/\text{MC-9}$ electrode material under the half-cell configuration.

| Cycles No. (1K=1000) | Specific Capacitance (F g^{-1}) | |
|-------------------------|---|---|
| | by CV @ scan rate of 100 mV s^{-1} | by GCD @ current density of 1 A g^{-1} |
| 0k | 1049 | 1629 |
| 5k | 985 | 1484 |
| 10k | 1017 | 1633 |
| 15k | 1036 | 1627 |
| 20k | 953 | 1629 |
| 25k | 1051 | 1632 |
| 30k | 1036 | 1640 |
| 35k | 933 | 1627 |

Table S5. The calculated Specific Capacitance from CV and GCD curves of fabricated MC-1000//Ti₃C₂T_x/MC-9 asymmetric supercapacitor device.

| Cycles No. (1K=1000) | Specific capacitance (F g⁻¹) | |
|---------------------------------|---|---|
| | by CV @ scan rate of 100 mV s ⁻¹ | by GCD @ current density of 1 A g ⁻¹ |
| 0k | 51.7 | 80.3 |
| 10k | 47.8 | 71.4 |
| 20k | 47.5 | 71.5 |
| 30k | 48.6 | 76.1 |
| 40k | 48.5 | 76.3 |
| 50k | 50.1 | 80.3 |
| 60k | 50.5 | 78.3 |

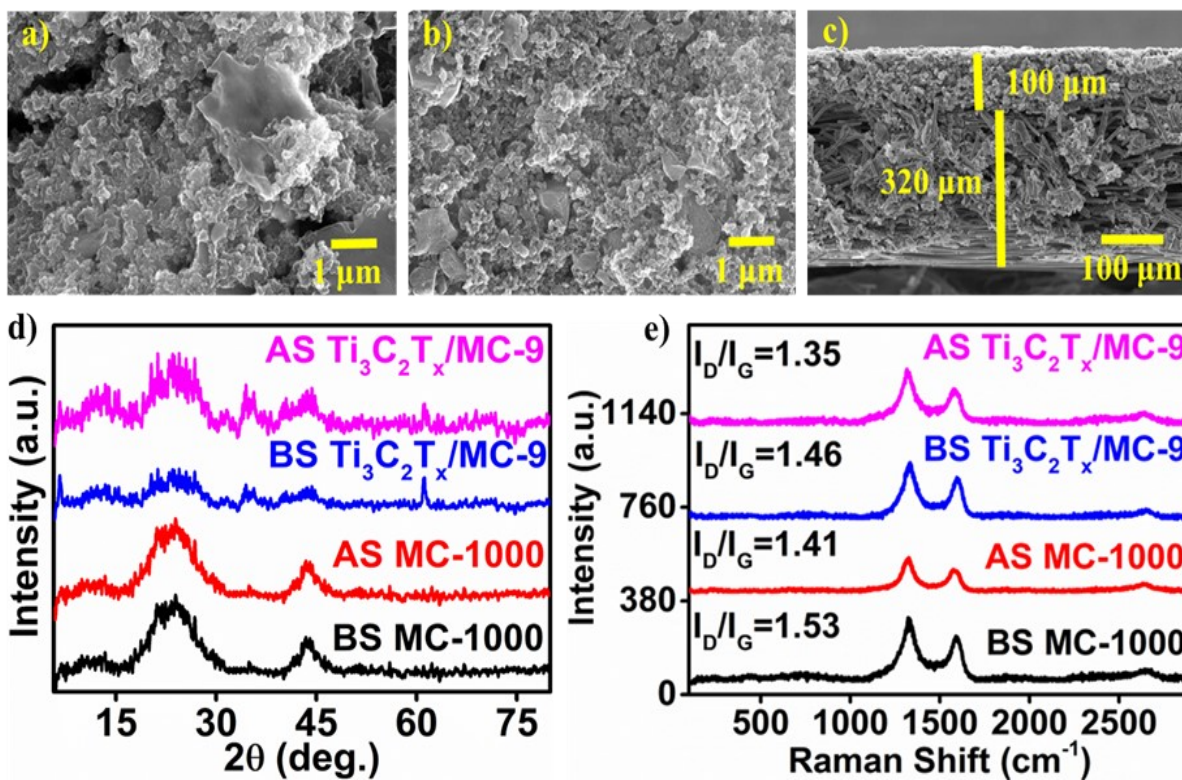


Figure S10. Post stability study of the Coin-Cell ASC; SEM images of (a-b) MC-1000, before and after stability, respectively; (c) SEM image displaying cross section of the Ti₃C₂T_x/MC-9 electrode material deposited on carbon paper showing its thickness; (d) XRD patterns and (e) Raman spectra of Ti₃C₂T_x/MC-9 and MC-1000, before and after stability study.

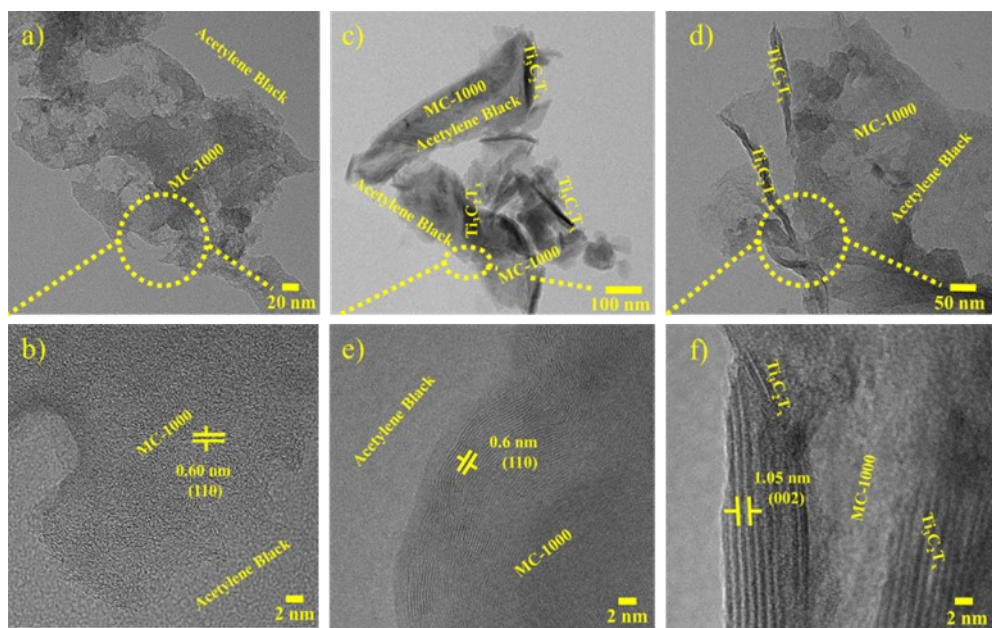


Figure S11. Post stability study: TEM and HRTEM images of (a-b) MC-1000 and (c-f) $\text{Ti}_3\text{C}_2\text{T}_x/\text{MC-9}$ nanocomposite.

Table S6. The comparative electrochemical performance for the MXene ($\text{Ti}_3\text{C}_2\text{T}_x$) and carbon based electrode materials.

| Materials | Preparation Method | Electrolyte | Specific Capacitance | | Cycling Stability ^(a) | Cycling Stability ^(b) | Ref. |
|--|---|--------------------------------|--|---------------------------------------|---|---|------|
| | | | Electrode (a) | Device (b) | | | |
| CPCM/MXene | annealing treatment at 800°C | 1mol/L H_2SO_4 | 362 F g ⁻¹ , 0.5 A g ⁻¹ | -- | 93.87 % @10,000 | -- | 1 |
| CP@rGO//MXene | oxidative polymerization | 3 M H_2SO_4 | 280 F g ⁻¹ , 0.5 A g ⁻¹ | 57, 59, and 47 F g ⁻¹ , | -- | 88 % @20000 75 % @20000 80 % @10000 | 2 |
| MXene/N-doped carbon foam | annealing treatment at 800°C | 1M KOH | 332 F g ⁻¹ | 63 F g ⁻¹ | 99.2 % @10,000 | 96 % @2500 | 3 |
| $\text{Ti}_3\text{C}_2\text{T}_x$ -MCNT | polymerization | 0.5M Na_2SO_4 | 1.93 F cm ⁻² | 0.94 F cm ⁻² | 94 % @1000 | 72 % @1000 | 4 |
| MXene-RuO ₂ | hydrothermal | 1M H_2SO_4 | 388 F g ⁻¹ | 93 F g ⁻¹ | 88 % @20000 | 86 % @20000 | 5 |
| $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheet / $\text{Ti}_3\text{C}_2\text{T}_x$ QD/RGO | wet-spinning technique, | 1m H_2SO_4 | 542 F cm ⁻³ | 53.1 F cm ⁻³ | 95.6 % @5,000 | 96.6 % @5000 | 6 |
| $\text{Ti}_3\text{C}_2\text{T}_x$ /RGO | hydrothermal | 2M KOH | 154.3 F g ⁻¹ | -- | 85 % @ 6,000 | -- | 7 |
| $\text{Ti}_3\text{C}_2\text{T}_x$ /RGO | vacuum-assisted filtration | 1M H_2SO_4 | 140 F g ⁻¹ | 29 F g ⁻¹ | 85 % @10,000 | 85 % @10,000 | 8 |
| rGO: $\text{Ti}_3\text{C}_2\text{T}_x$ | self-assembly 20000 rpm | 1M H_2SO_4 | 254 F g ⁻¹ 2 mV s ⁻¹ | -- | 193 F g ⁻¹ @100 mVs ⁻¹ | --- | 9 |
| MXene-rGO hydrogel | electrostatic self-assembly | 3M H_2SO_4 | 320 F g ⁻¹ | -- | 46 % @8000 | -- | 10 |
| $\text{Ti}_3\text{C}_2\text{T}_x$ -RGO | film through vacuum filtration | 3M H_2SO_4 | 505 F g ⁻¹ | -- | 50 % @10,000 | -- | 11 |
| $\text{Ti}_3\text{C}_2\text{T}_x$ /SCNT | Self-assembled composite film. | 1M KOH | 314 F cm ⁻³ | -- | 95 % @10000 | -- | 12 |
| rGO/ $\text{Ti}_3\text{C}_2\text{T}_x$ | ultrasonic treatment calcined at 300°C | 6M KOH | 405 F g ⁻¹ | 148.5 F g ⁻¹ | 99.9 % @10,000 | 100 % @10,000 | 13 |

| | | | | | | | |
|--|--|--|--|--|---------------------------|---------------------------|----------------------|
| MXene/RGO | Ultrasonication After about 3 days, | 1M H ₂ SO ₄ | 233 F g ⁻¹ | -- | 91 % @ 10,000 | -- | 14 |
| Ti ₃ C ₂ /CNTs | EPD | 6M KOH | 134 F g ⁻¹ | 55.3 F g ⁻¹ | @10000 | -- | 15 |
| 3D porous MXene/rGO | 300°C electric heating plates in a glove box | 3M H ₂ SO ₄ | 340.8 F g ⁻¹ | -- | 90.7 % @ 40,000 | -- | 16 |
| 3D macroscopic graphene/MXene hydrogel | hydrothermal | 6M KOH | 267.7 F g ⁻¹ | -- | 100 % @10000 | -- | 17 |
| Ti₃C₂T_x/MC-9 | Solvothermal | 0.5 M H₂SO₄ | 1629 F g⁻¹ at 1 A g⁻¹ | 80.3 F g⁻¹ at 1 A g⁻¹ | 99.9 % @35,000 | 97.5 % @60,000 | This Work |

- ❖ CP = conducting Polymer
- ❖ CPCM = chitosan porous carbon Spheres
- ❖ Pin = Polyindole

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