Unveiling the Supercapacitive behavior of Electrospun Cr_2CT_x/Carbon Nanofiber Membrane

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Supplementary File

Table S1: Materials utilized in the present study

Sl. No	Material	Purity (%)	Supplier
1.	Chromium metal powder, Cr	>99.5%	Kanton Laboratories
2.	Graphite powder, C	>99.5%	Sisco Research Laboratories Pvt
3.	Aluminium metal powder, Al	>99%	Sisco Research Laboratories Pvt
4.	Polyvinyl alcohol, PVA (Mw 89000–98000)	>99%	Sigma-Aldrich
5.	Hydrofluoric acid, HF	-	S D fine chem limited (SDFCL).
6.	Polyvinylidene fluoride, PVDF	-	
7.	N-methyl-2-pyrrolidone, NMP	-	
8.	Ethanol	>99%	
9.	Toluene	>99%	

Table S2: Materials Characterization

Sl. No	Characterization method	Instrument specifications
1.	X-ray diffractometer, XRD	Rigaku Miniflex, Cu-Kα radiation ($\lambda = 0.154$ nm) within the 2θ range of 10-80 ℃
2.	Raman spectrum	Renishaw inVia confocal Raman microscope in the range 500-3000 cm^{-1}
3.	BET pore volume and surface Area analyser	Belsorp Mini X pore size distribution analyzer
4.	Scanning Electron Microscopy with Energy-Dispersive X-ray spectroscopy, SEM-EDX	Apreo S LoVac
5.	Thermogravimetric analysis (TGA)	PerkinElmer, at a heating rate of 10 ℃ min^{-1}

A 0.1-0.5% w/v MXene solution was first prepared by probe sonication, followed by the addition of 10% w/v PVA. The mixture was then heated and stirred at 90 °C for 4 hours to achieve uniformity, resulting in a homogeneous spinning solution. Under optimized electrospinning conditions (25 kV voltage, 18 cm needle-to-collector distance, and 0.5 mL/h flow rate, maintained at 25 °C and 40% relative humidity), the solutions were electrospun to form a Cr_2CT_x/PVA nanofibrous mat. For the carbonization process, the obtained nanofibrous mats were first placed in a silica crucible and subjected to a stabilization step at 200 °C for 2 hours in a muffle furnace, promoting oxidative crosslinking of PVA. This was followed by calcination at different temperatures to study the effects of temperature: (i) 300 °C for 1 hour, (ii) 350 °C for 1 hour, and (iii) 400 °C for 1 hour.

We observed that temperatures above 300 °C led to fiber breakage and charring, while temperatures lower than 300 °C or lower MXene loadings (<0.5%) resulted in poor MXene distribution (not evident in EDS) and lower electrochemical performance. Therefore, 300 °C for 1 hour was optimized as the ideal carbonization condition, with 0.5% MXene loading.

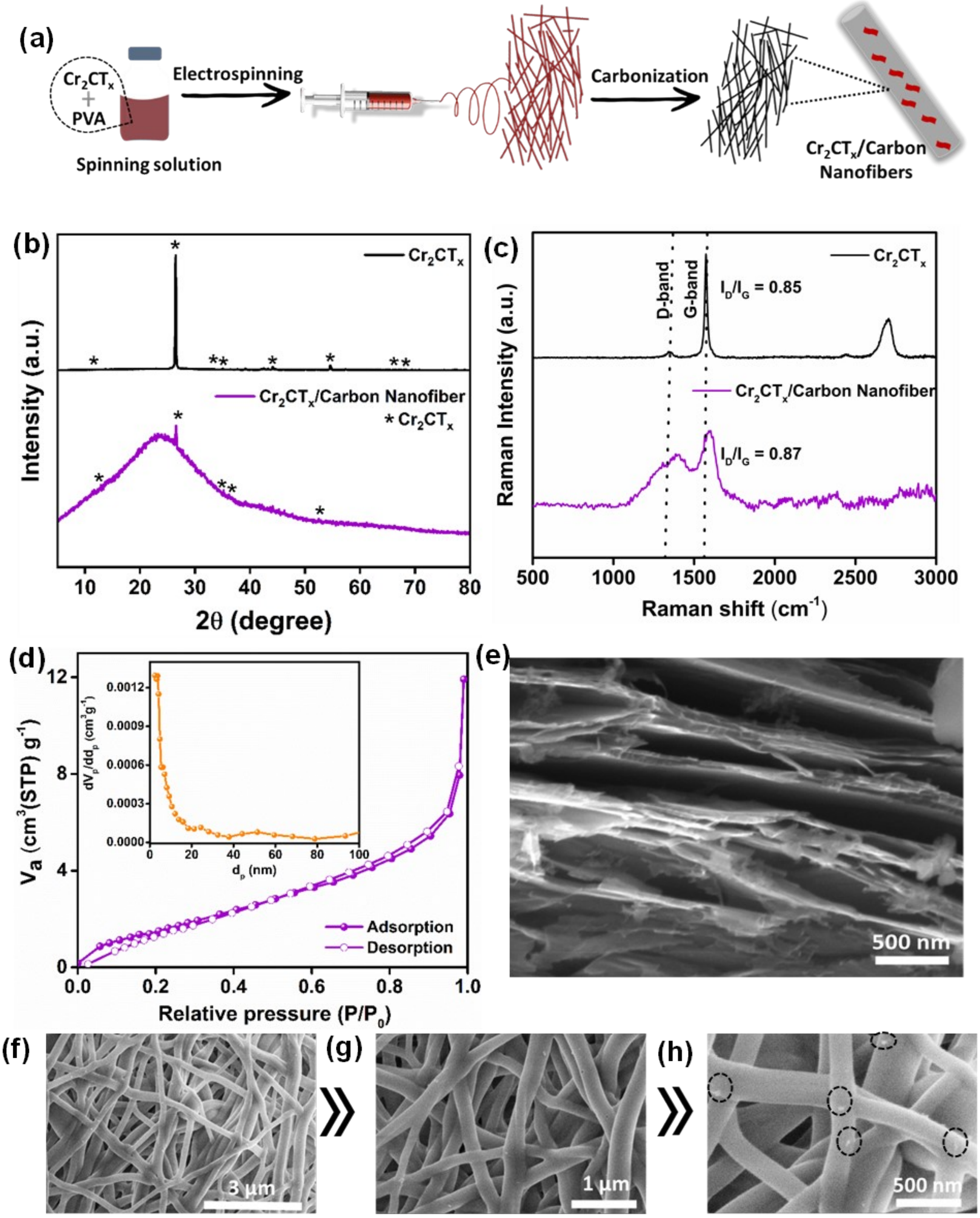


Fig. S1 (a) Schematic illustration of the preparation of Cr_2CT_x/Carbon Nanofiber composite, comparison between Cr_2CT_x/Carbon Nanofiber and pristine Cr_2CT_x; (b) XRD patterns, (c) Raman spectra, and (d) N_2 adsorption–desorption isotherms (inset: corresponding pore size distribution). SEM images of (e) Cr_2CT_x and (f-h) Cr_2CT_x/Carbon Nanofiber at various magnifications.

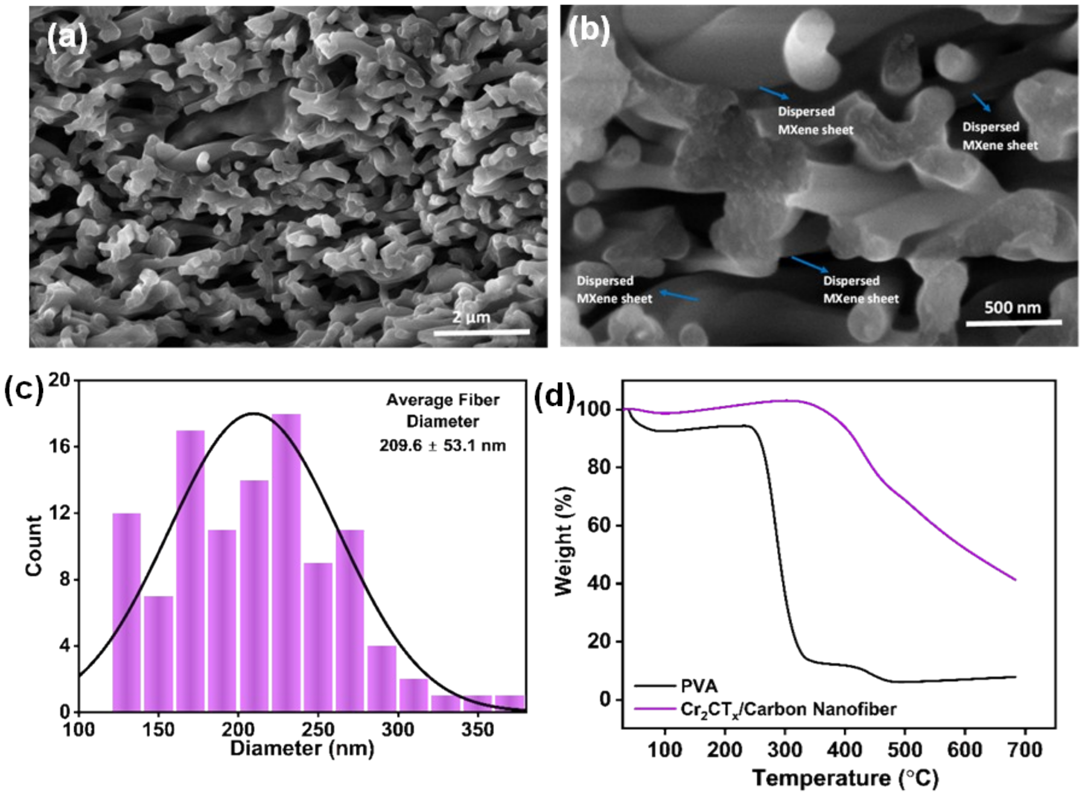
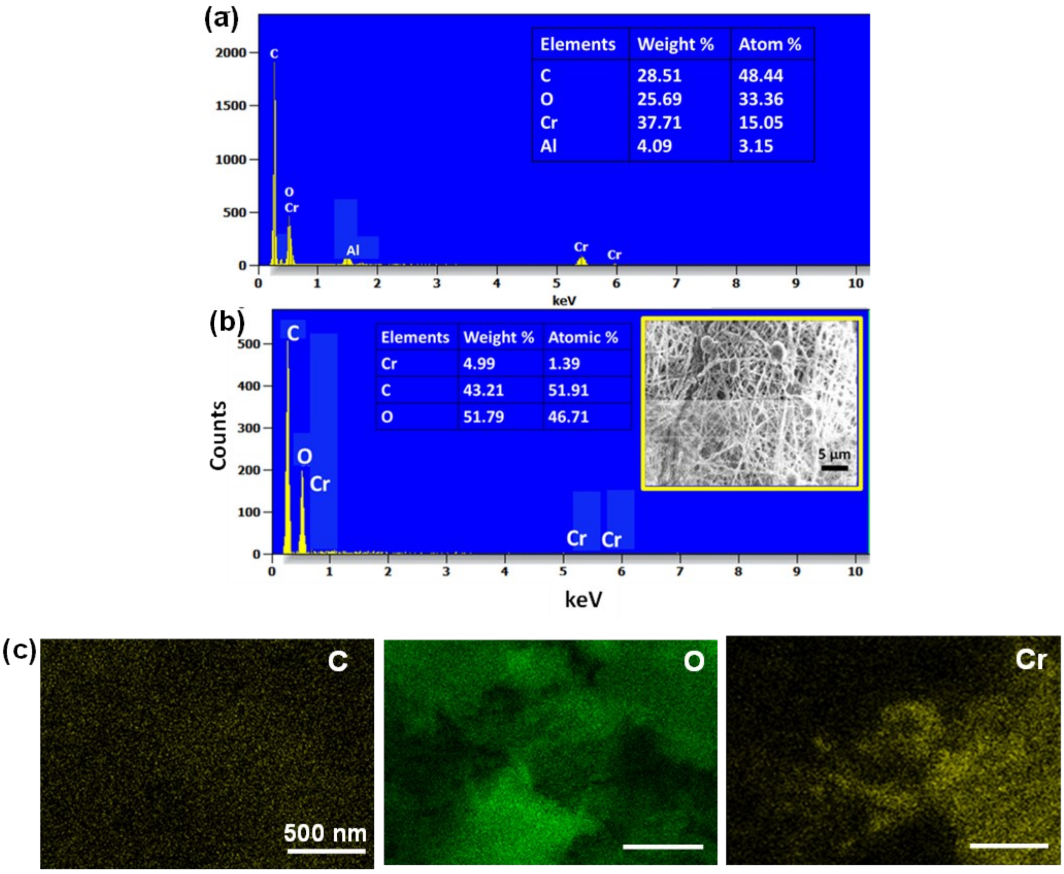


Fig. S2 (a-b) SEM images of the cross-sectional view of Cr_2CT_x/Carbon Nanofibers composite, illustrating the well-dispersed MXene sheets within the CNF network, (c) histogram representing fibre diameter and (d) TGA profile of the composite



Elements	Weight %	Atom %
C	28.51	48.44
O	25.69	33.36
Cr	37.71	15.05
Al	4.09	3.15

Elements	Weight %	Atomic %
Cr	4.99	1.39
C	43.21	51.91
O	51.79	46.71

Fig. S3 EDX analysis of (a) Cr_2CT_x, (b) Cr_2CT_x/Carbon Nanofiber and (c) EDS C, O and Cr mappings

Electrode fabrication

The Ni-foam substrate was activated by cleaning in 0.5 M H_2SO_4 solution for 15 minutes to remove surface oxides and impurities, followed by thorough rinsing with deionized water and drying at 60 °C. This treatment improves surface wettability and ensures better adhesion of the active material. Further, an aqueous slurry containing 50 mg Cr_2CT_x/carbon nanofiber, 20 mg activated carbon, 10 mg PVDF, and 60 µL of NMP was prepared using bath sonication, then drop-cast onto 1×1 cm^2 Ni-foam to form the working electrode, followed by overnight drying.

Formulae

$$i_p = a(v)^b \tag{S1}$$

$$i_p = m_1 v + m_2 (v)^{\frac{1}{2}} \tag{S2}$$

$$C_s = \frac{I\,\Delta t}{m\,\Delta V} \tag{S3}$$

$$Energy\ density = \frac{C_s \times (\Delta V)^2}{2 \times 3.6} \qquad (S4)$$

$$Power\ density = \frac{3600 \times Energy\ density}{\Delta t} \qquad (S5)$$

In equation S1, i_p represents the peak current and v is the scan rate. In equation S2, $m_1 v$ and $m_2(v)^{\frac{1}{2}}$ correspond to the capacitive and diffusive contributions, respectively. Further, in equation S1-S3 C_s denotes the specific capacitance (Fg^{-1}), I is the current (1 Ag^{-1}), Δt is the discharge time (s^{-1}), m is the electrocatalyst loading, ΔV is the potential difference.

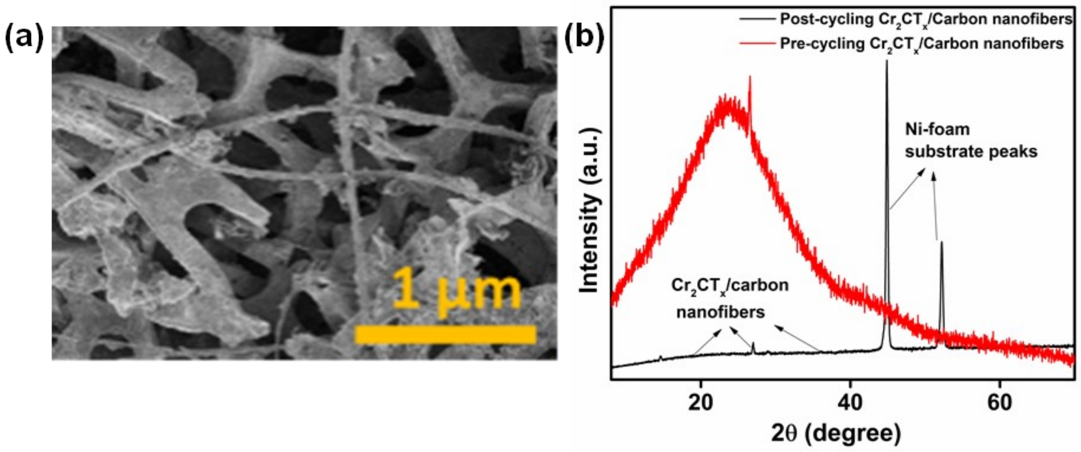


Fig. S4 Post-cycling (a) SEM image and (b) XRD of Cr_2CT_x/Carbon Nanofiber

Table S3: Comparison of synthesis strategies and capacitance performance with reported literature.

Sl. No.	Electroactive material	Synthesis strategy	Electrolyte	Capacitance	Asymmetric device-specific capacitance (C_s)	Ref.
1.	$Ti_3C_2T_x$/Carbon nanofiber	Electrospinning, PAN	1M H_2SO_4	205 mF cm^{-2} @ 50 mV s^{-1}(areal)	-	[1]
2.	$Ti_3C_2T_x$/rGO/PEDOT:PSS	Radial freezing, PEDOT:PSS	3M H_2SO_4	475 Fg^{-1} @ 5 mV s^{-1}(specific)	113 Fg^{-1} @ 0.2 Ag^{-1}	[2]
3.	MnO_2@MXene/CNT fiber	Wet spinning	0.02 M $MnSO_4$-H_2O and 0.2 M	371.1 Fg^{-1} (specific)	238 Fg^{-1} @ 1 A cm^{-3}	[3]

			Na_2SO_4			
4.	MnO_2@Co-MOF/MXene/CNF	Electrospinning, PAN	2 M KOH	-	475.4 mAhg^{-1} @ 1 Ag^{-1}	[4]
5.	$Ti_3C_2T_x$/ CNF	Electrospinning, PAN	1M H_2SO_4	120 Fg^{-1} @ 2 mVs^{-1} (specific)	-	[5]
6.	$FeCo_2S_4$/MXene /PCNF	Electrospinning, PAN-PVP	3 M KOH	446 Fg^{-1} @ 1 Ag^{-1} (specific)	249 Fg^{-1} @ 1Ag^{-1}	[6]
7.	PANI/MXene/CNF	Electrospinning, PANI	1M H_2SO_4	356 Fg^{-1} @ 0.5 Ag^{-1} (specific)	-	[7]
8.	Cr_2CT_x/Carbon Nanofiber	Electrospinning, PVA	3 M KOH	1084 @ 1Ag^{-1}	338.8 Fg^{-1}@ 1Ag^{-1}	**This work**

Several recent studies have explored various MXene-based composites for supercapacitor applications using different synthesis strategies, electrolytes, and electroactive materials. For instance, $Ti_3C_2T_x$/Carbon Nanofiber composites were synthesized via electrospinning using PAN as a precursor and tested in 1 M H_2SO_4, achieving an areal capacitance of 205 mF cm^{-2} at 50 mV s^{-1}. [1] A MnO_2@MXene/CNT fiber composite fabricated by wet spinning in a mixed electrolyte (0.02 M $MnSO_4 \cdot H_2O$ and 0.2 M Na_2SO_4) demonstrated a volumetric capacitance of 371.1 F cm^{-3} and energy and power density of 8.22 mWh cm^{-3} and 276.28 mW cm^{-3}, respectively. [3] Similarly, a MnO_2@Co-MOF/MXene/CNF composite prepared by electrospinning using PAN and tested in 2 M KOH exhibited a capacity of 475.4 mAh g^{-1}, while the device delivering an energy density of 72.5 Wh kg^{-1} and a power density of 832 W kg^{-1} power density. This highlights the advantage of incorporating metal oxide-modified MOFs over MXene for enhanced energy storage applications. [4] Another study reported a $Ti_3C_2T_x$/rGO/PEDOT:PSS composite, delivered energy density of 10.11 W h kg^{-1} with 70.5% capacitance retention. [2] Further, $Ti_3C_2T_x$/CNFs synthesized by electrospinning with PAN exhibited a specific capacitance of 120 F g^{-1} in 1 M H_2SO_4. [5] Another study developed a asymmetric device using $FeCo_2S_4$/MXene/Porous Carbon Nanofiber which exhibited specific capacitance of 249 Fg^{-1} and an energy density of 86.7 Wh kg^{-1}. [6] A PANI/MXene/Carbon Nanofiber composite, synthesized by electrospinning using PANI, achieved a specific capacitance of 356 F g^{-1} at 0.5 A g^{-1}. [7] Meanwhile, several non-CNF Cr_2CT_x MXene composites have been reported, where modifications with spinel structures such as $NiFe_2O_4$ [8], $CoFe_2O_4$[9], metal oxides, and others play a significant role. In these composites, the dopants alongside MXene strongly influence the electrochemical performance, often resulting in higher capacitance values. Further, this study emphasizes the use of the novel Cr_2CT_x MXene, where the Cr (a group 6 element) with partially filled d-orbitals helps lower the d-band center, thereby reducing the band gap and enhancing catalytic properties compared to Ti-based MXenes. Additionally, PVA was selected as the polymer precursor due to its cost-effectiveness, hydrophilicity, excellent catalyst dispersion ability, and biocompatibility compared to other polymers. Importantly, PVA itself does not contribute directly to the electrochemical performance, while the resulting carbon nanofibers (CNFs) synergistically interact with Cr_2CT_x MXene, significantly boosting the overall charge storage capability and device performance.

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