Structural characterization of Nb_2CT_z MXene for energy storage application

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Introduction

MXenes, a family of two-dimensional transition metal carbides and nitrides, have attracted significant interest for energy storage due to their excellent electrical conductivity, hydrophilicity, and efficient ion transport. Among them, niobium carbide (Nb_2CT_z) has emerged as a promising material for supercapacitor electrodes. In this work, Nb_2CT_z was synthesized using the MILD etching method, enabling selective removal of aluminum and partial exfoliation into few-layer nanosheets. The synthesis process was further optimized by systematically varying the etching temperature, etching time, and etchant composition, allowing fine-tuning of the material properties. Structural and compositional characterization was performed using Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and scanning electron microscopy with energy-dispersive spectroscopy (SEM/EDX). The electrochemical performance of the prepared Nb_2CT_z electrodes was investigated by cyclic voltammetry and galvanostatic charge–discharge measurements, demonstrating their strong potential for next-generation energy storage systems.

Experimental part

The phase purity and crystallinity of synthesized samples were examined using X-ray diffraction (Raguku Ultima IV, Japan). The X-ray beam was nickel-filtered CuKα1 radiation (λ = 0.1540 nm, operating at 40 kV and 40 mA). XRD data were collected from 3 to 70° (2θ) at a scanning rate of 2°/min, with a step size of 0.02. To provide high-intensity, high-resolution measurements, parallel beam geometry, and the D/teX Ultra, a high-speed one-dimensional X-ray detector were used.

Fourier-transform infrared (FTIR) spectra of nanocomposites were obtained using attenuated total reflection (ATR) mode on a Nicolet 6700 spectrometer, while for the PEG-MXene and pure MXene the Thermo Fisher Scientific Waltham, MA, USA spectrometer in transmission mode on a KBr substrate was employed.

Electrochemical measurements were carried out on Gamry potentiostat. The capacitive performance was tested using three-electrode system composed of working electrode (MXene), counter electrode (Pt mesh) and reference electrode (saturated calomel electrode). 3 mol/dm3 KOH aequosus solution was used as electrolyte. Cyclic voltammetry and galvanostatic charge/discharge were employed for describing the electrochemical performance of electrodes. Sweep rates used in experiment were 5, 10, 50 and 100 mV/s. Current densities used for charge/discharge test were 0.25 0.5, 1 and 2 A/g.

Table 1 –Optimization conditions for synthesis of Nb_2CT_z MXene

No. Synthesis	Reagents	Reagent ratio (MAX phase : reagents)	Time of etching (h)	Temperature °C	Intercalation agent
1	LiF, HCl	1 : 1.2	96	70	/
2	LiF, HCl	1 : 1.2	96	RT	/
3	HF	1 : 3	72	RT	/
4	HF	1 : 3	24	60	/
5	LiF, HCl	1 : 1.5	120	50	/
6	LiF, HCl	1 : 1.2	120	60	DMSO

Results

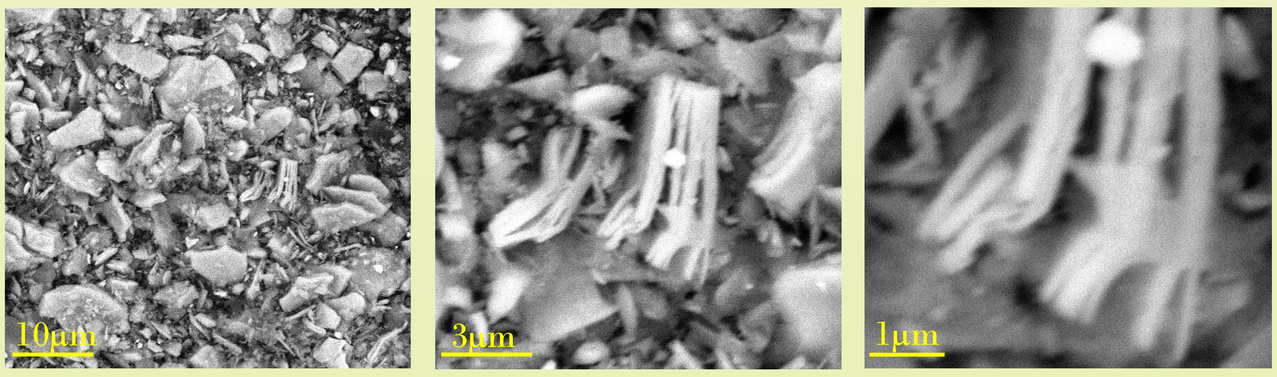

Figure 1 – SEM images of Nb_2CT_z MXene on different magnification

Table 2 –EDX analysis of Nb_2CT_z MXene

Element	Atomic Conc. %	Weight Conc. %
C	42.50	20.53
O	26.33	16.94
F	17.22	13.16
Nb	12.91	48.23
Al	1.05	1.14

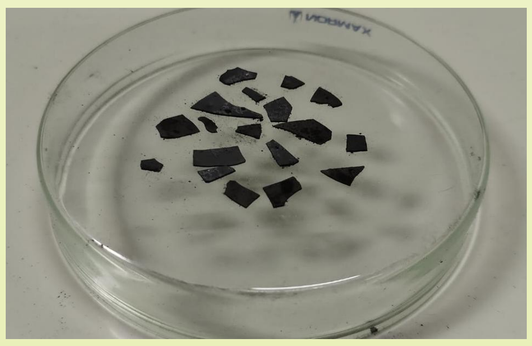

Figure 2 – Photograph of Nb_2CT_z MXene powder

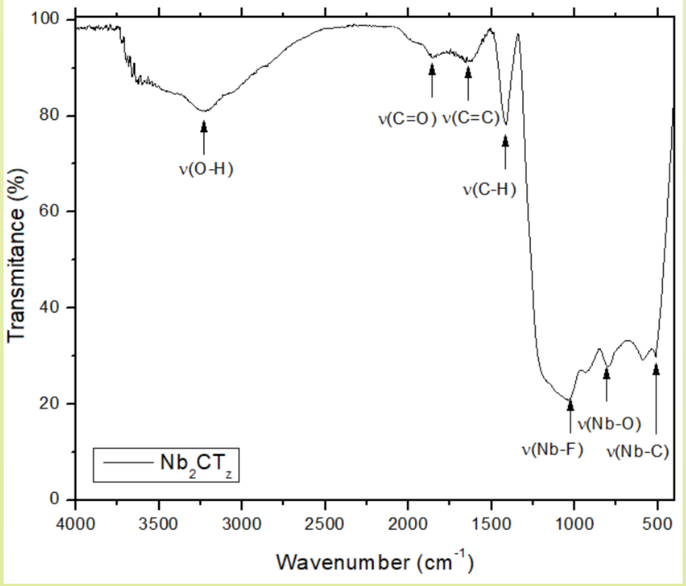

Figure 3 – FTIR spectra of Nb_2CT_z MXenes

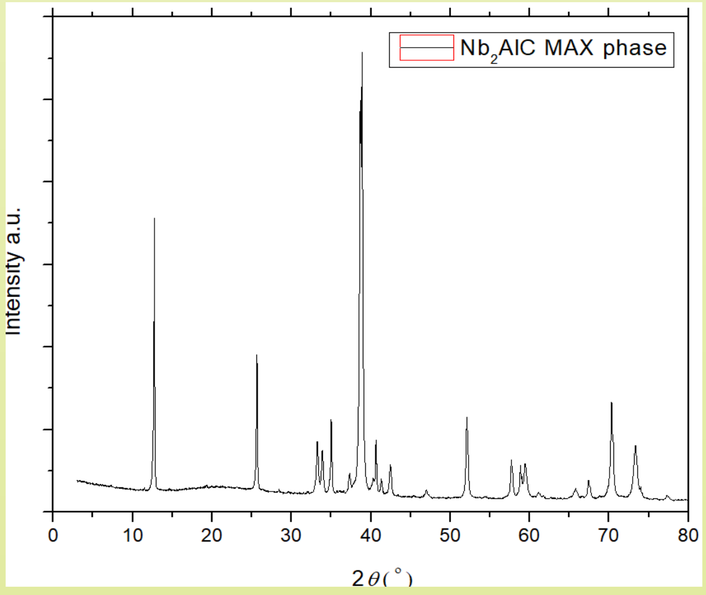

Figure 4 – XRD spectrum of Nb_2AlC MAX phase

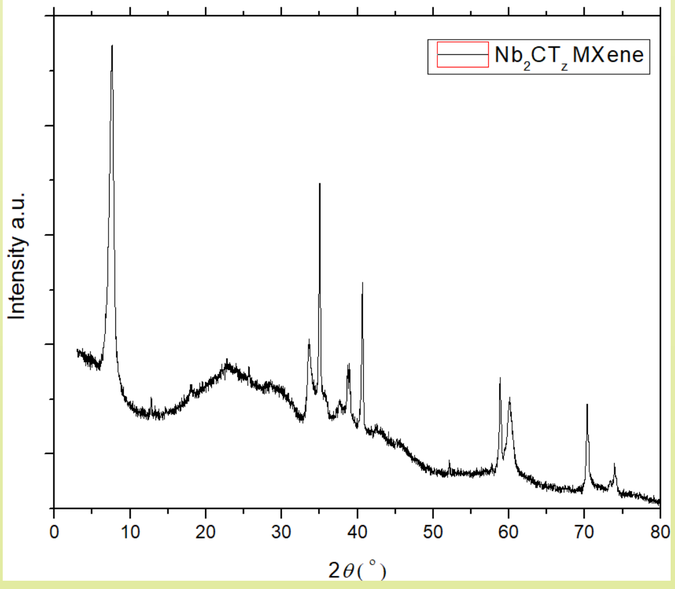

Figure 5 – XRD spectrum of Nb_2CT_z MXene

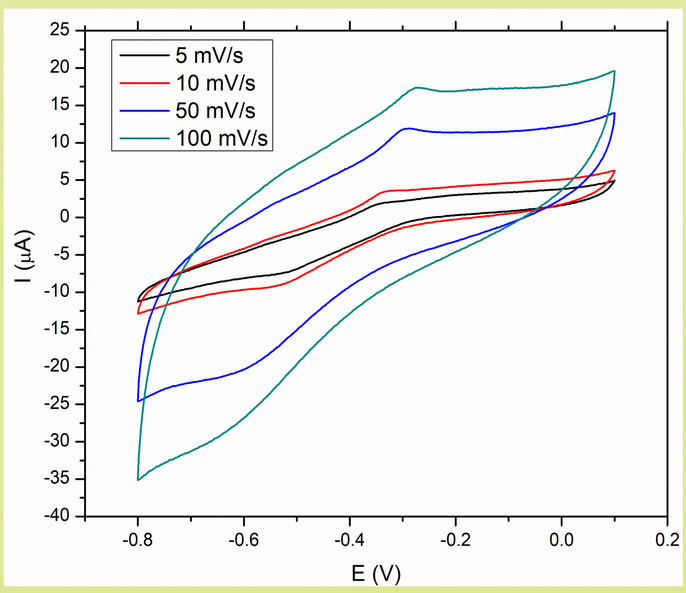

Figure 6 – Cyclic voltammetry of Nb_2CT_z MXene on different scanning speed

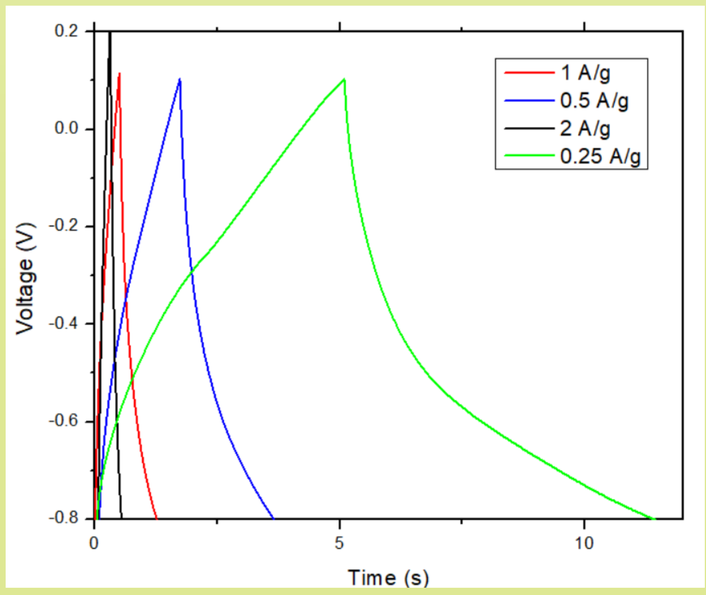

Figure 7 – Galvanostatic charge-discharge of Nb_2CT_z MXene on different current densities

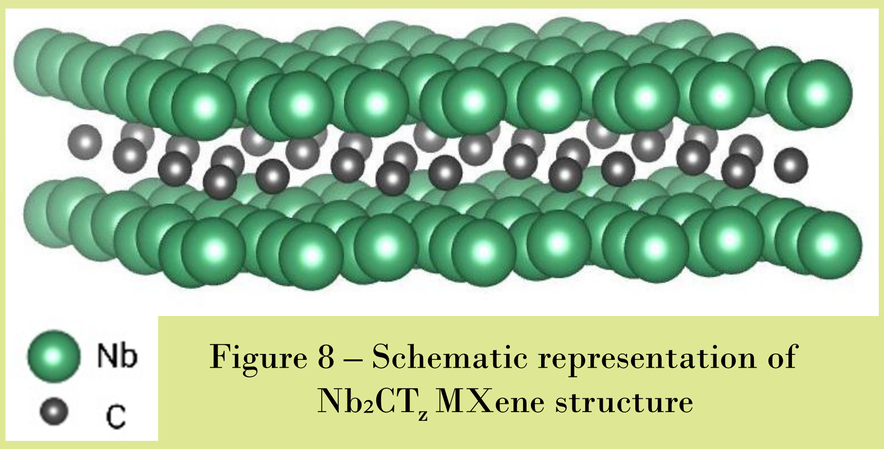

Figure 8 – Schematic representation of Nb_2CT_z MXene structure

Conclusion

In this work we successfully synthesized and characterized the Nb_2CT_z MXene as electrode for supercapacitors. Firstly, we examined the impact of the synthesis parameters such as temperature, etching duration and etchant composition. FTIR analysis confirmed the characteristic bands of the Nb_2CT_z MXene. SEM analysis showed the well-known accordion like MXene structure while the EDX results confirmed the presence of Nb, C, O and F elements with the absence of Al. XRD analysis revealed optimal etching route by showing the characteristic peak at around 6θ. Cyclic voltammetry showed faint signs of oxidation and reduction at -0.28V and -0.56V. The synthesized Nb-MXene can be further improved for supercapacitor application by more intensive delamination and functionalization with organosilanes.

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Reference:

[1] Pešić, I., Pergal, M. V., Vasiljević-Radović, D., Popović, M., Uskoković, P., Petrović, M.,Radojević, V. (2024). Capacitance breakthroughs in free-standing electrodes through MXene functionalization. Science of Sintering, (00), 28-28.