Supplementary data

Ultrafast microwave synthesis of Ti-based MXenes with high yields

Jovana Paskaš[a,b], Miloš Dujović[c], Milica Govedarica[b], Vasa Radonić[b], Vladimir V. Srdić[a,d], Nikola Kanas[b,e#]

[a]*University of Novi Sad, Faculty of Technology, Department of Materials Engineering, 21000 Novi Sad, Serbia*

[b]*University of Novi Sad, BioSense Institute, 21000 Novi Sad, Serbia*

[c]*Texas A&M University, College Station, USA*

[d]*Serbian Academy of Sciences and Arts, 11000 Belgrade, Serbia*

[e]*University of Novi Sad, Faculty of Technical Sciences, 21000 Novi Sad, Serbia*

The starting point of this research was based on the lowest power setting available in a conventional microwave oven, which was 220 W. Since this power exceeded the microwave power used by Zhu *et al.* (200W)[1], it was assumed that the overall synthesis time could be further reduced. However, as no clear quantitative relationship between microwave power and etching time has been established, preliminary experiments were conducted to evaluate the system response.

In these preliminary experiments, the MAX phase was exposed to microwave irradiation for 90 s without intermediate mixing or cooling steps (Table 2). The analysis indicated that MXene formation had already initiated, however, the etching process was incomplete, and residual MAX phase was still present. These findings suggest that a longer effective reaction time is required to achieve complete etching. Furthermore, the introduction of mixing steps enhances the interaction between the MAX phase and the etching agent, facilitating deeper penetration of the etchant and promoting more efficient exfoliation.

Table 2. Experimental details related to Ti_3C_2 and Ti_3CN syntheses

MAX phase	MXene	Synthesis		Mixing and cooling	
		Number of cycles	Duration of cycles (s)	Number of cycles	Duration of cycles (s)
Ti_3AlC_2	Ti_3C_2	1	90	0	0
Ti_3AlCN	Ti_3CN	4	3	3	60

Figure S1a and b show the X-ray diffractograms of Ti_3C_2 and Ti_3CN etched for 90 s. Hence, it represents the reason why particular etching times were selected in the experiments.

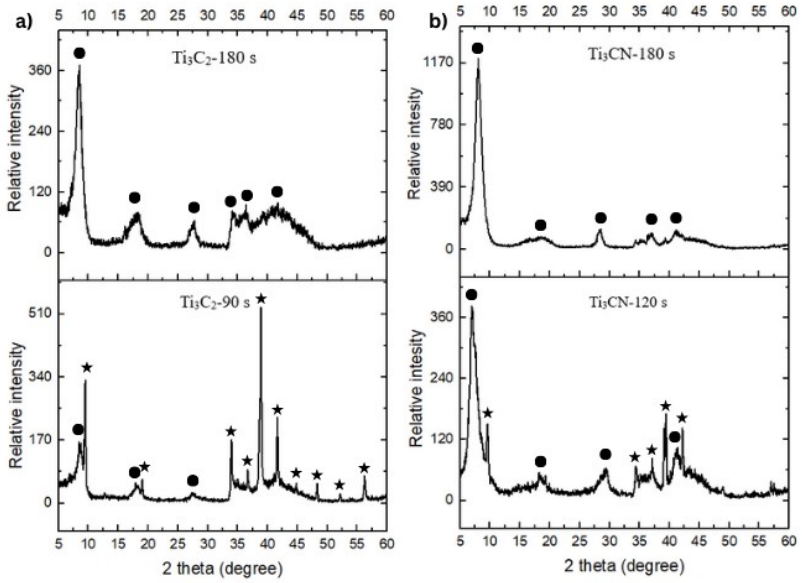

Figure S1. X-ray diffractograms of a) Ti_3C_2 samples (90 s and 180 s of microwave synthesis) and b) Ti_3CN samples (120 s and 180 s of microwave synthesis). Peaks of the MXene phase and MAX phases are marked with • and *, respectively

The equivalent circuits presented in Figure S2 was used to model the interfacial electrochemical behavior of the planar sensor modified with a MXene-based composite. The circuit of the bare electrode include several key components: the solution resistance (R_{sol}), representing ionic resistance through the electrolyte; the charge transfer resistance (R_{ct}) which reflects the kinetics of electron transfer across the electrode/electrolyte interface and elements accounting for capacitive and diffusive phenomena. Capacitive behavior is modeled using a constant phase element (*CP*), often replacing an ideal capacitor to better represent the

frequency dispersion caused by surface heterogeneity, roughness, or porosity of the electrode, particularly relevant for MXene-modified surfaces. The Warburg impedance (*W*), typically observed as a linear tail at low frequencies in the Nyquist plot, represents diffusion-controlled ion transport within the electrolyte and through the porous electrode structure.

The electrochemical behavior of the MXene/PEDOT:PSS composite electrode was investigated using electrochemical impedance spectroscopy across a frequency range of 1 Hz to 100 kHz, with the experimental data analyzed through equivalent circuit modeling to extract quantitative parameters governing the electrochemical processes at the composite interface. The impedance response was most accurately described by a R(WC) equivalent circuit, comprising a solution resistance (R_{sol}) in series with a parallel combination of a Warburg diffusion element (*W*) and a double-layer capacitance (*C*), yielding fitted parameters of R_{sol} = 263.4 ± 1.4 Ω with 0.53% error, *W* = 963.0 ± 18.1 σ with 1.88% error, and *C* = 22.60 ± 2.66 μF with 11.79% error. The pronounced Warburg impedance indicates that the electrochemical process is predominantly governed by mass transport diffusion within the porous MXene/PEDOT:PSS composite architecture rather than interfacial charge transfer kinetics, where the solution resistance reflects ionic transport through the composite matrix and the capacitance arises from the extensive surface area of the MXene/PEDOT:PSS interface, suggesting that optimizing ion diffusion pathways represents a critical factor for enhancing the electrochemical performance of these composite systems.

Additionally, the effective area can be assessed using the known diffusional coefficient, which is represented by the slope of the current versus scan rate from the Randles-Shevchick equation:

$$I_p = 2.69 \cdot 10^5 \cdot A \cdot D^{1/2} \cdot n^{3/2} \cdot v^{1/2} \cdot c$$

where A is the electroactive area in cm^2, I_p is anodic current peak in A, D is the diffusion coefficient ($6.5 \cdot 10^{-6}$ cm2/s in case used redox probe), n is the number of electrons transferred in half-reaction (in our case 1), v is scan rate of 0.5 V/s. The calculate effective area is A=0.596 cm2.

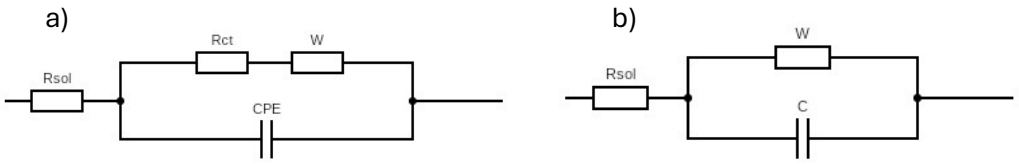

Figure S2: Picture shows different cases of equivalent circuits for a) bare electrode and PEDOT: PSS, b) MXene/PEDOT: PSS composite