Fabrication and characterization of Polypyrrole and Polyaniline functionalized MXene electrodes

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SUSTAINABLE PRINTED ELECTRONICS

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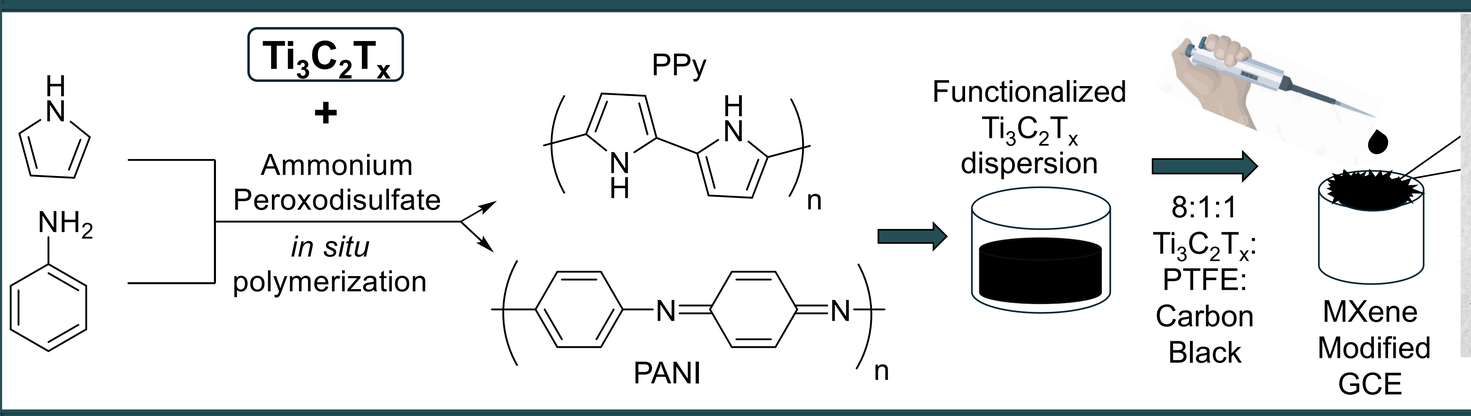
Introduction

In 2011, Dresden University discovered a new group of compounds with enhanced electronic properties, namely MXenes. Since then, many research groups around the world have endeavored in their research to advance their scope, ultimately bringing them to market applications.[1] Herein, MXenes synthesized by High Frequency Acoustic Excitation (HFAE) were drop-casted onto Glassy Carbon Electrodes (GCE) to study their electrochemical properties.[2] Additionally, MXenes were functionalized with Polypyrrole (PPy) and Polyaniline (PANI) to further enhance their electrochemical properties.. More specifically, Cyclic Voltammetry (CV), Electrochemical Impedance Spectroscopy (EIS) and Electroactive Surface Area (EASA) were studied. Structural and morphological analysis was also performed including measurements such as Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDS) and Dynamic Light Scattering (DLS).

Materials and Methods



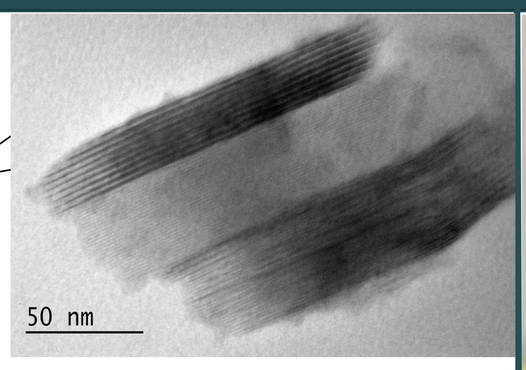
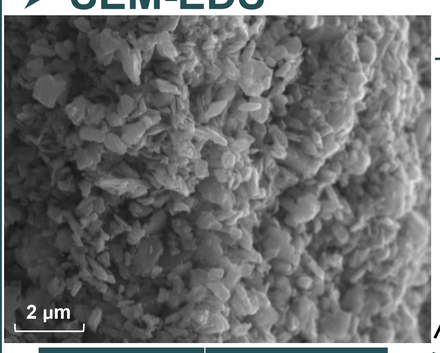


Figure 1. TEM image of $Ti_3C_2T_x$

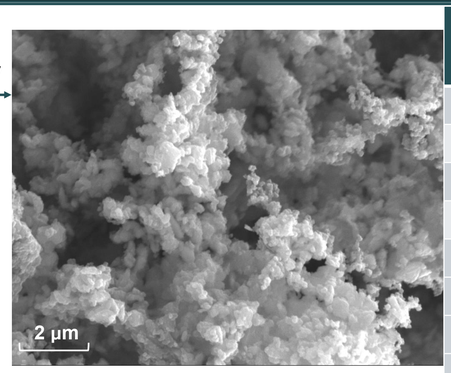
CE: Pt mesh
WE: GCE (d= 3 mm)
RE: Ag/AgCl (3.5 M KCl)
Electrolyte: 0.1 M KCl 1 mM $K_3[Fe(CN)_6]$

Results

SEM-EDS

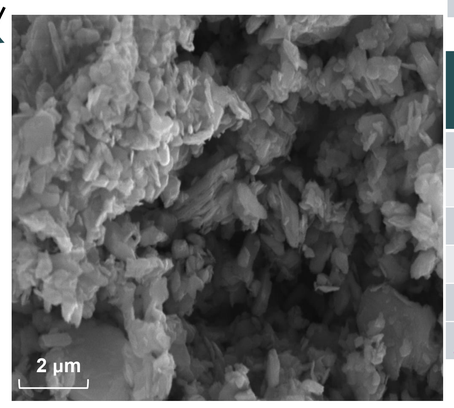


PPy



Element	Weight Ratio (%)
Titanium	38.96
Carbon	32.25
Oxygen	13.64
Nitrogen	8.82
Fluorine	3.42
Aluminum	0.89
Chlorine	0.91
Sulphur	1.10

PANI

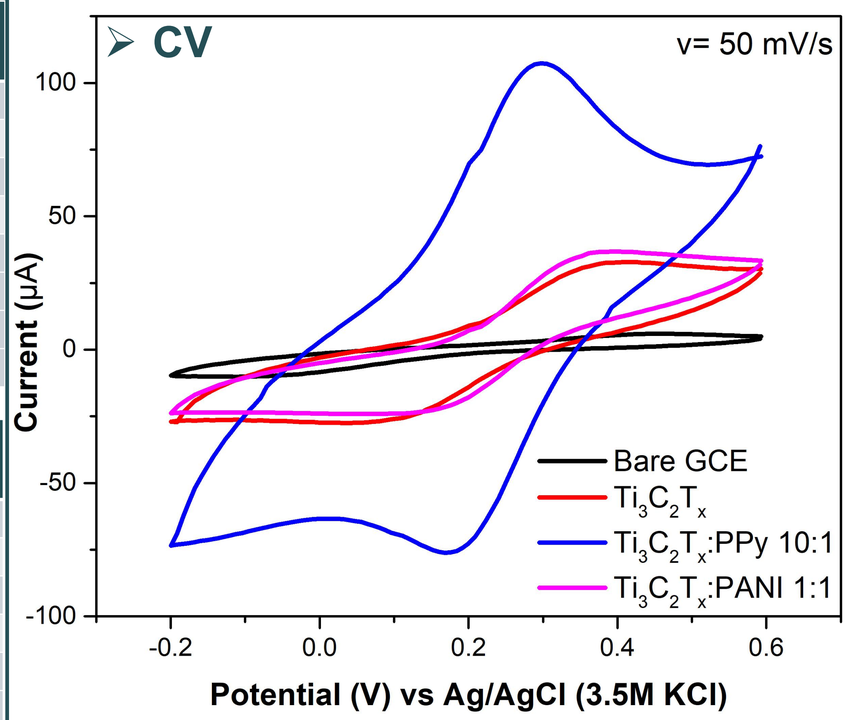


Element	Weight Ratio (%)
Titanium	61.95
Carbon	13.95
Oxygen	11.14
Nitrogen	4.16
Fluorine	7.15
Aluminum	1.64

Element	Weight Ratio (%)
Titanium	48.06
Carbon	19.80
Oxygen	16.76
Nitrogen	8.12
Fluorine	5.80
Aluminum	1.46

Figure 2. SEM-EDS analysis of $Ti_3C_2T_x$, $Ti_3C_2T_x$:PANI 1:1 and $Ti_3C_2T_x$:PPy 10:1

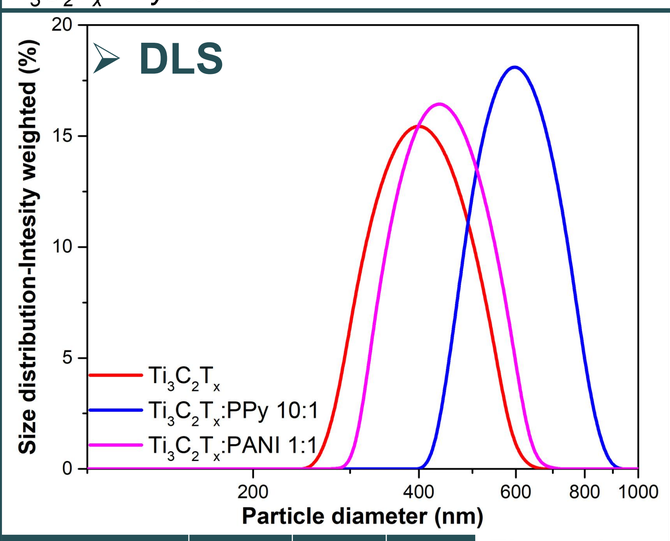
CV



Bare GCE: negligible electrochemical response
$Ti_3C_2T_x$: improved redox response
$Ti_3C_2T_x$:PANI 1:1: higher current peaks, slightly enhanced oxidation activity
$Ti_3C_2T_x$:PPy 10:1: largest loop area, most favorable electrochemical response

Figure 3. CV curves of $Ti_3C_2T_x$, $Ti_3C_2T_x$:PANI 1:1 and $Ti_3C_2T_x$:PPy 10:1 at a potential window E= -0.2 - +0.6 V vs Ag/AgCl (3.5M KCl) at a scan rate of 50 mV/s

DLS

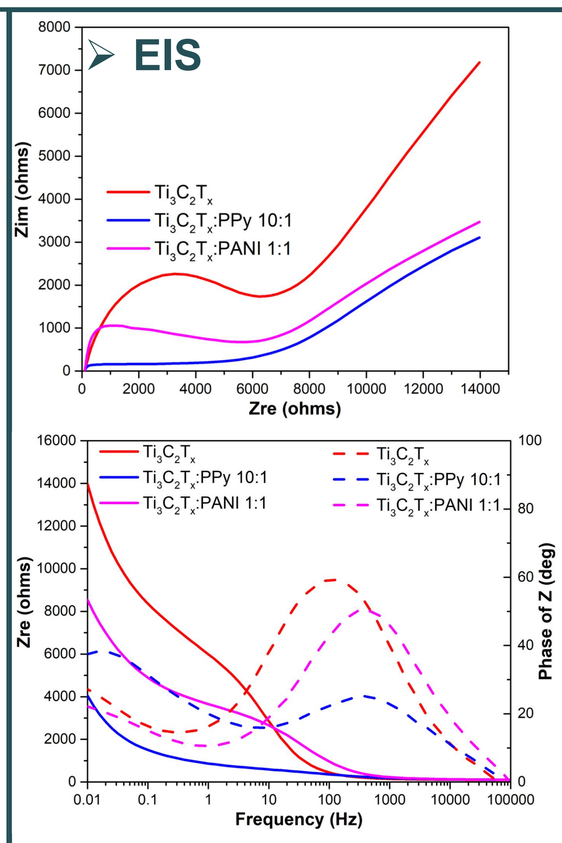


Sample	HDD (nm)	PDI (%)	ZP (mV)
$Ti_3C_2T_x$	478	0.9	-14.6
$Ti_3C_2T_x$:PPy 10:1	742	18.8	-11.4
$Ti_3C_2T_x$:PANI 1:1	537	18.7	-28.5

Figure 4. Particle size distribution of $Ti_3C_2T_x$, $Ti_3C_2T_x$:PANI 1:1 and $Ti_3C_2T_x$:PPy 10:1

$Ti_3C_2T_x$: smallest particle size
$Ti_3C_2T_x$:PANI 1:1: moderate size increase
$Ti_3C_2T_x$:PPy 10:1: largest particle size, stronger aggregation

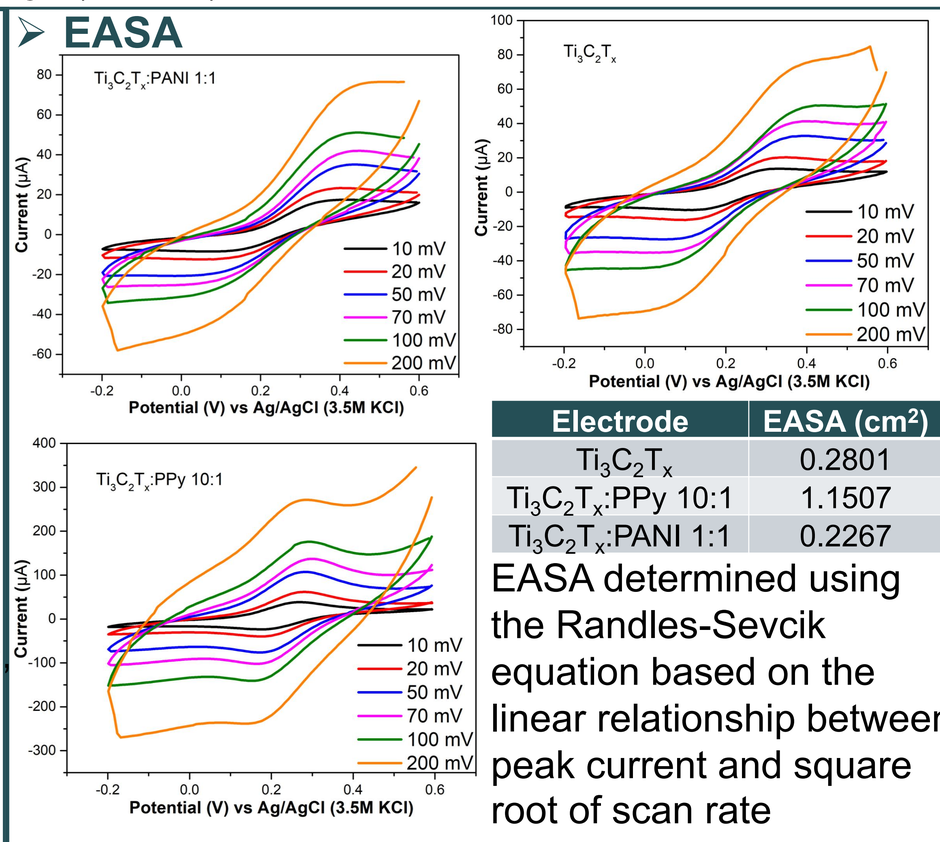
EIS



$Ti_3C_2T_x$: highest R_{ct} and impedance with largest phase angle, poor charge transfer
$Ti_3C_2T_x$:PANI 1:1: reduced R_{ct} and impedance with moderate phase response, improved charge transfer
$Ti_3C_2T_x$:PPy 10:1: lowest R_{ct} and impedance with smallest phase angle, best electrochemical activity and fastest charge transfer

Figure 5. Nyquist (top) and Bode (bottom) plots of $Ti_3C_2T_x$, $Ti_3C_2T_x$:PANI 1:1 and $Ti_3C_2T_x$:PPy 10:1 recorded in 0.1 M KCl, 1 mM $K_3[Fe(CN)_6]$ aqueous supporting electrolyte

EASA



Electrode	EASA (cm^2)
$Ti_3C_2T_x$	0.2801
$Ti_3C_2T_x$:PPy 10:1	1.1507
$Ti_3C_2T_x$:PANI 1:1	0.2267

EASA determined using the Randles-Sevcik equation based on the linear relationship between peak current and square root of scan rate

Figure 6. CV curves of $Ti_3C_2T_x$, $Ti_3C_2T_x$:PANI 1:1 and $Ti_3C_2T_x$:PPy 10:1 at a potential window E= -0.2 - +0.6 V vs Ag/AgCl (3.5 M KCl) at scan rates of 10, 20, 50, 70, 100 and 200 mV/s

Conclusions

Electrochemical and morphological analyses reveal distinct electrode performances. **$Ti_3C_2T_x$**, while featuring the smallest particle size, displayed the highest charge transfer resistance and weakest redox activity. **$Ti_3C_2T_x$:PANI** increased in size but showed reduced impedance, leading to enhanced redox peaks and improved charge transfer. **$Ti_3C_2T_x$:PPy**, despite showing larger particle size, achieved the most favorable electrochemical performance, with the largest CV loop area, lowest resistance, and fastest charge transfer.

References

[1] A. Vahidmohammadi, J Rosen, Y. Gogotsi, The world of two-dimensional carbides and nitrides (MXenes), *Science*, **372**, 6547, (2021)

[2] T. S. Gopal, S. K. Jeong, T. A. Alrebdi, S. Pandiaraj, A. Alodhayb, M. Muthuramamoorthy, A.N. Grace, MXene-based composite electrodes for efficient electrochemical sensing of glucose by non-enzymatic method, *Mater. Today Chem.*, **24**, 100891, (2022)

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