



Article

Electrochemical Impedance Analysis of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene for Pseudocapacitive Charge Storage

Nafiza Anjum ¹, Abdullah Al Noman ¹, Md Mostafizur Rahman ² , Debashis Sen ³, Robert A. Lazenby ³ and Okenwa I. Okoli ^{1,2,*}

¹ High-Performance Materials Institute, FAMU-FSU College of Engineering, Tallahassee, FL 32310, USA; na20@fsu.edu (N.A.)

² Herff College of Engineering, University of Memphis, Memphis, TN 38152, USA

³ Department of Chemistry and Biochemistry, Florida State University, Tallahassee, FL 32306, USA; rlazenby@fsu.edu (R.A.L.)

* Correspondence: o.okoli@memphis.edu

Abstract: This study investigates the electrochemical behavior of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene for supercapacitor applications, focusing on its charge storage mechanisms using Electrochemical Impedance Spectroscopy (EIS). A novel equivalent circuit (EC) model, incorporating a diffusion layer resistance and a constant phase element, was developed to represent the impedance spectra, achieving a low error margin of 4.6%. The cycling stability of MXenes and charge storage parameters were evaluated using the developed EC model. This study demonstrated that the irreversible anodic oxidation of MXene begins around 0.3 V due to water molecule attack from the aqueous electrolyte, resulting in the formation of a titanium oxide layer that increases charge transfer resistance and impairs charge storage. It was further revealed that the cycling stability of MXene is also related to the oxidation of MXene, and the initial capacitance of 493 F/g at 100 mV/s is reduced by 27.5% after 1000 cycles. The contribution of charge storage factors was analyzed, with 85% of MXene's capacitance found to be surface controlled. This research offers a deeper understanding of MXene's charge storage mechanisms, providing critical insights into optimizing its electrochemical performance and stability. By establishing advanced modeling approaches and addressing challenges related to oxidation and resistance, this work enhances MXene's potential for high-power supercapacitors in electromechanical actuator applications.



Academic Editor: Nikolay Mukhin

Received: 26 February 2025

Revised: 11 March 2025

Accepted: 13 March 2025

Published: 17 March 2025

Citation: Anjum, N.; Noman, A.A.; Rahman, M.M.; Sen, D.; Lazenby, R.A.; Okoli, O.I. Electrochemical Impedance Analysis of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene for Pseudocapacitive Charge Storage. *J. Compos. Sci.* **2025**, *9*, 139. <https://doi.org/10.3390/jcs9030139>

Copyright: © 2025 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

Keywords: electrochemical impedance spectroscopy; equivalent circuit model; MXene; charge storage mechanism

1. Introduction

Power-dense applications, such as flight control surfaces in aircraft and launch vehicles, have traditionally relied on hydraulic actuation systems. However, the shift toward more electric solutions is gaining momentum due to their advantages in integration and life cycle cost reduction. The development of electromechanical actuators has intensified the need for efficient electrical power sources. These actuators must deliver high-power maneuvers while ensuring sufficient mission energy density to support demanding duty cycles [1].

To meet these high-power requirements, a conventional battery-based approach involves either placing multiple battery strings in parallel or investing in costly high-power batteries. However, supercapacitors provide a superior alternative by offering rapid energy discharge, high power density, and exceptional cycle life. There is a growing interest in using supercapacitors (also called ultracapacitors or electrochemical capacitors) as power

sources for electromechanical applications, particularly in space environments. This interest is driven by their ability to deliver high power pulses with minimal heat generation, operate across a broad temperature range ($-40\text{ }^{\circ}\text{C}$ to $+65\text{ }^{\circ}\text{C}$), and sustain over one million charge–discharge cycles [2,3].

Among emerging supercapacitor materials, MXene-based supercapacitors show significant promise. MXenes, a class of two-dimensional (2D) transition metal carbides, nitrides, and carbonitrides, have revolutionized energy storage due to their metallic conductivity, hydrophilicity, tunable surface chemistries, and layered structures [4–6]. These properties enable high capacitance, rapid charge–discharge rates, and excellent stability, making them ideal candidates for supercapacitor applications. Of all MXene materials, $\text{Ti}_3\text{C}_2\text{T}_x$ MXene has been extensively studied for energy storage applications due to its superior electrical conductivity and better stability [7,8]. The breakthrough study by Lukatskaya et al. (2013) revealed the pseudocapacitive effect of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene, leading to volumetric capacitance values as high as 442 F/cm^3 [9]. Ghidui et al. demonstrated that $\text{Ti}_3\text{C}_2\text{T}_x$ free-standing electrodes in $1\text{ M H}_2\text{SO}_4$ achieved an impressive specific gravimetric capacitance of 246 F/g at 2 mV/s scan speed, along with a volumetric capacitance of 910 F/cm^3 [10].

Despite these advances, fully optimizing MXene-based supercapacitors for high-power electromechanical actuator applications requires a deeper understanding of their charge storage mechanisms. Addressing these knowledge gaps is critical for harnessing MXene's full potential, enabling their effective integration into high-performance energy storage systems for next-generation electromechanical applications.

Charge storage mechanisms in supercapacitors are primarily governed by two factors: electrical double-layer capacitance (EDLC) and pseudocapacitance. In EDLCs, charges are stored in the porous electrodes in a purely capacitive manner within an electrical double-layer (EDL) [11–13]. In several cases, additional pseudocapacitive energy storage is possible through redox reactions and/or ion intercalation [14]. This is the case for 2D materials such as graphene oxide (GO) and MXenes [14,15], where energy density is thus improved. MXenes exhibit a dominant pseudocapacitive charge storage behavior, facilitated by redox reactions at the surface functional groups and intercalation of ions within the interlayer spacing [16].

Advanced electrochemical techniques, including cyclic voltammetry (CV), galvanostatic charge–discharge (GCD), and Electrochemical Impedance Spectroscopy (EIS), collectively provide a comprehensive understanding of the electrochemical processes involved in the charge storage mechanisms of MXene-based systems, guiding the optimization of their design. EIS is a very powerful technique to characterize electrochemical phenomena, such as ion transport kinetics, interfacial resistance, and charge transfer processes, occurring at different frequencies in supercapacitors and provides information about their resistive and capacitive behavior and the identification of diffusion-limited processes. In EIS, the impedance is represented as a complex quantity as shown in Equation (1).

$$Z(\omega) = Z' - jZ'' \quad (1)$$

where Z' (the real part) corresponds to resistance (R), and Z'' (the imaginary part) corresponds to $1/\omega C$, with C as capacitance and ω as angular frequency. A Nyquist plot is often used to visualize these components, plotting Z' on the x-axis and Z'' on the y-axis [17]. For supercapacitor electrodes, a typical Nyquist plot is shown in Figure 1. It exhibits three distinct features: a semicircle at high frequency, a tilted linear section at middle frequencies, and a vertical tail at low frequencies. At very high frequencies, ions cannot penetrate pores or layers due to insufficient time, leaving only the external surface accessible, which makes resistive components dominant. Conversely, at very low frequencies, ions fully penetrate the layers, charge saturation dominates, and the electrode behaves as an ideally polariz-

able porous interface, resulting in a vertical line on the Nyquist plot. In the intermediate frequency range, resistance and capacitance become frequency-dependent, and diffusion processes play a significant role.

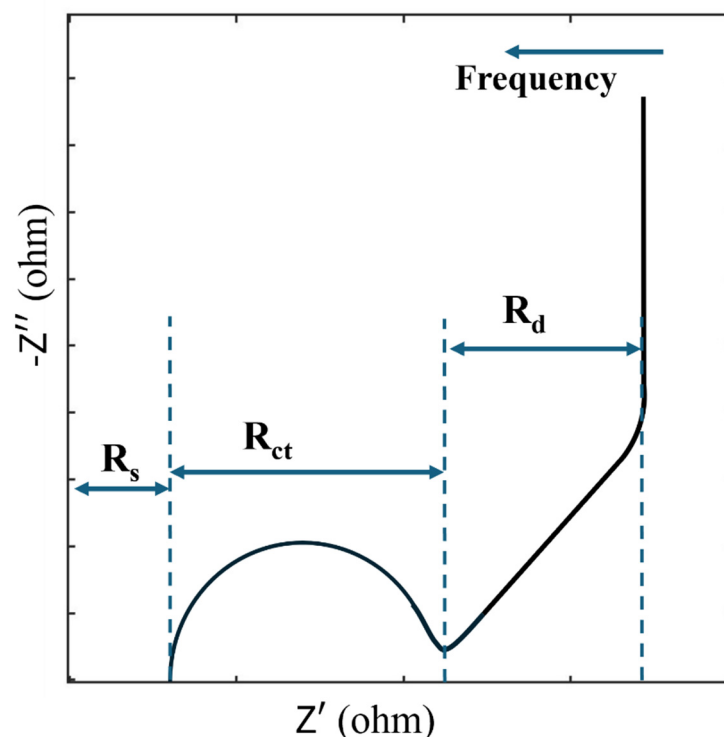


Figure 1. Typical Nyquist plot for a supercapacitor.

Nyquist plots can be used to model electrochemical processes in MXene interfaces through the equivalent electrical circuit (EC) [3,18,19]. The EC assists in quantifying charge storage parameters like different resistances and capacitances involved in the system.

To date, very few studies have been performed on exploring MXene's charge storage mechanism using an EIS study. Aguedo et al. studied MXene-modified electrodes using EIS to prepare effective electrochemical biosensors [20]. Mainka et al. studied EIS data and a general EC model to characterize fiber-shaped MXene/graphene oxide supercapacitors [21]. Hui et al. used EIS and a basic EC to model hydrothermally treated MXene and study its electrochemical activities [22]. In other studies, EIS was used to observe the qualitative electrochemical behavior of MXenes, without using any EC for quantitative studies [22,23]. Hence, the knowledge on MXene's electrochemical processes for film-type supercapacitor application is still scarce. A low-error general EC to represent the electrochemical charge storage mechanisms in MXene is yet to be established.

The development of an EC from the EIS data requires in-depth understanding of the electrochemical behavior of MXene at different conditions. Chen et al. investigated how transitions in surface functionalization affect MXenes' pseudocapacitive behavior in acidic environments. They concluded that the pseudocapacitance is inherently linked to Faradaic processes, wherein specific surface functional groups act as active centers that facilitate charge transfer through interactions with the ionic electrolyte, thereby influencing electron storage and mobility [23]. Boota et al. studied the functionalization of titanium carbide MXenes with quinones and identified that specific surface functional groups can stabilize redox reactions, improving energy storage efficiency [24]. Huang et al. found that $\text{Ti}_3\text{C}_2\text{T}_x$ MXene undergoes reversible H^+ ion intercalation in acidic electrolyte. This reversible process is characterized by a transformation between $\text{Ti}_3\text{C}_2\text{O}_2$ and $\text{Ti}_3\text{C}_2(\text{OH})_2$.

on the MXene surface, highlighting the role of oxygen-rich functional groups in enhancing pseudocapacitive behavior [25]. In addition to this, short ion diffusion and intercalation pathways of MXene are unique properties that lead to improvements in pseudocapacitive performance. It was demonstrated in several studies that modification of surface functional group can simultaneously enhance surface redox reaction and ion diffusion and hence improve overall pseudocapacitance [26,27].

Representing such complexities in charge storage behavior in the developed EC necessitates a detailed understanding of the physical processes involved in the charge storage mechanism and interplays between structure, synthesis, and performance. To optimize the performance of supercapacitor, it is also important to understand the electrochemical behavior of MXene in an anodic and a cathodic potential window and to examine a suitable potential window for electrochemical stability. Furthermore, understanding the electrochemical processes involved during charging and discharging is crucial to ensure enhanced cycle life. Interpreting EIS data can be challenging without a clear understanding of interfacial processes, making it difficult to select an appropriate EC. Proper circuit elements identified from Nyquist plot fitting provide qualitative and quantitative parameters such as time constants or activation energies for specific processes [28]. Modification of the basic ECs is often necessary for specific systems, as they deviate from the ideal behavior. For example, in cases where rough surfaces are involved, redox properties cannot be adequately described using capacitive elements alone. Instead, a constant phase element (CPE) is utilized to account for these complexities [29].

The contribution of this research lies in its detailed investigation of the electrochemical behavior of MXenes for energy storage application. Electrochemical processes at different voltage ranges were studied to understand the physical processes involved during charging and discharging and to determine a suitable voltage range for the desired application. EIS technique was employed, and a modified EC with low error percentage was developed that resembles the investigated charging processes of MXenes. Furthermore, the oxidative degradation of MXene during CV cycling is observed and studied to quantify the different involved resistive and capacitive elements using the developed EC. Finally, the contribution of different types of pseudocapacitance like surface-controlled and diffusion-controlled capacitance is studied and quantified. This research adds to the growing body of knowledge on MXene-based materials, paving the way for their broader application in energy storage technologies.

2. Materials and Methods

2.1. Materials

Lithium fluoride (LiF, 98.5%) was obtained from Alfa Aesar (Haverhill, MA, USA), while concentrated hydrochloric acid (37% HCl) was sourced from Thermo Fisher Scientific (Waltham, MA, USA). Ti_3AlC_2 MAX phase ($\geq 90\%$, $\leq 100\ \mu\text{m}$ mesh size) was purchased from Sigma-Aldrich (St. Louis, MO, USA). Nafion dispersion (5 wt% in water and 1-propanol, density 0.93 g/mL) was acquired from Beantown Chemical (Hudson, NH, USA). A PVDF membrane with a 0.45 μm pore size was sourced from Millipore Sigma (St. Louis, MO, USA).

2.2. Synthesis of MXene

MXene was synthesized using a modified MILD etching method, with an HCl and LiF etchant solution. First, 10 mL of 9M HCl and 1 g of LiF were stirred for 10 min. Then, 0.5 g of Ti_3AlC_2 MAX was slowly added to control the exothermic reaction. The mixture was stirred at 200 rpm for 24 h at 35 °C. After etching, it was washed with deionized (DI) water until the pH reached around 5, resulting in a dark-green supernatant containing

delaminated MXene flakes. At this stage, the supernatant appeared dark green, while a black slurry of etched $\text{Ti}_3\text{C}_2\text{T}_x$ settled above a gray layer of unetched or partially etched $\text{Ti}_3\text{AlC}_2\text{-Ti}_3\text{C}_2\text{T}_x$. The supernatant was transferred to a beaker, and 50 mL of DI water was added to the centrifuge tube to disperse the remaining sediment by manual shaking. The mixture was ultrasonicated for 2 min, centrifuged at 3500 rpm for 5 min, and the supernatant was collected. This process was repeated until the supernatant became clear, ensuring the complete delamination of MXene. Finally, the collected supernatant was vacuum-filtered through a PVDF membrane and dried at room temperature to form thin films. Proper PPE and safety measures were followed throughout.

2.3. Preparation of Drop-Cast MXene Electrode for Electrochemical Testing

To prepare the working electrode, a 3 mm diameter glassy carbon electrode was first polished with a $0.05\ \mu\text{m}$ alumina suspension, rinsed with deionized water, and air-dried. An optical micrograph of the bare electrode is shown in Figure 2a. A solution was prepared by mixing 100 μL of either DI water or dimethyl sulfoxide (DMSO) solvent with 20 μL of 5% Nafion binder, followed by dispersing 5 mg of ground $\text{Ti}_3\text{C}_2\text{T}_x$ MXene in the mixture. Subsequently, 1.0 μL of this solution was drop-cast onto the electrode. The electrode is referred to as a drop-cast DI-MXene electrode when DI is used as the solvent (Figure 2b) and a drop-cast DMSO-MXene electrode when DMSO is used (Figure 2c). The electrode was then allowed to dry at room temperature for 1 h in case of drop-cast DI-MXene, and for 6 h in case of drop-cast DMSO-MXene. The mass loading for the 3 mm electrode was calculated to be $5.9\ \text{g}/\text{m}^2$.

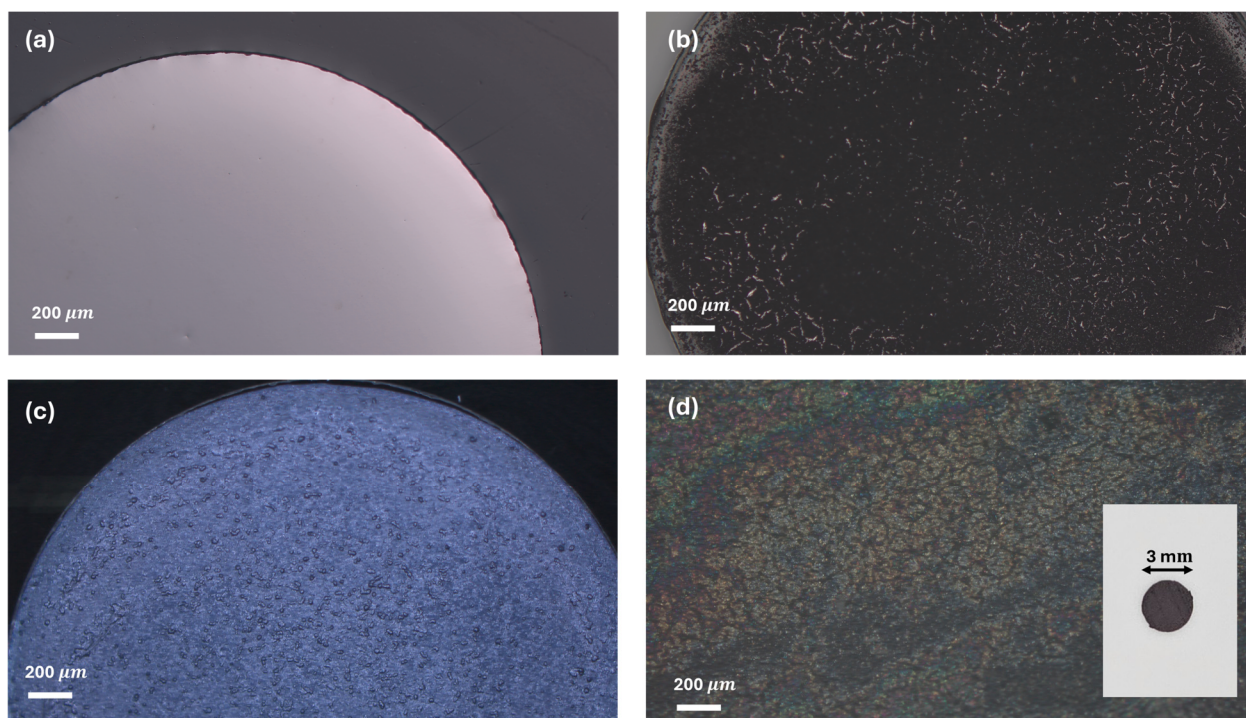


Figure 2. Microscopic images of (a) bare electrode, (b) drop-cast DI-MXene electrodes, (c) drop-cast DMSO-MXene electrodes, and (d) punch-paste MXene electrode.

2.4. Preparation of Punch-Paste MXene Electrode for Electrochemical Testing

A 3 mm glassy carbon electrode was polished, rinsed, and dried as described earlier. A 3 mm disk was punched from the corresponding $\text{Ti}_3\text{C}_2\text{T}_x$ MXene film. The binder solution was prepared by mixing 20 μL of 5% Nafion with 100 μL of deionized water. A 1 μL drop of the binder was applied to the glassy carbon electrode, and the punched 3 mm film was

carefully placed on top. The electrode was then left at room temperature for an hour to dry. This electrode is referred to as punch-paste MXene electrode (Figure 2d). The weight of the 3 mm punched disk was 0.04 mg, hence the mass loading is 6 g/m².

2.5. Preparation of Three Electrode System

The electrochemical performance of the prepared electrodes was evaluated using a standard three-electrode system. The working electrode consisted of either the drop-cast or punch-paste MXene-coated glassy carbon electrode, as described in previous sections. A platinum wire was used as the counter electrode, providing a stable and inert surface for electron exchange. The reference electrode was an Ag/AgCl (saturated KCl) electrode. All electrochemical measurements were conducted in a 1 M Na₂SO₄ aqueous solution, which served as the electrolyte. The system was assembled under ambient conditions, and all experiments were performed at room temperature.

2.6. Characterization

Electrochemical properties of the electrodes were studied using cyclic voltammetry (CV), galvanostatic charge–discharge (GCD), and Electrochemical Impedance Spectroscopy (EIS). These studies were conducted using CHI660E instrument electrochemical analyzer (CH Instrument, Inc., Austin, TX, USA) in a three-electrode system described in Section 2.5.

Scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS) were conducted using a Phenom XL G2 SEM (Thermo Scientific). X-ray diffraction (XRD) was performed using a SmartLab SE Automated Multipurpose X-ray diffractometer (Rigaku) with Cu K α radiation at 40 kV and 45 mA. The EIS data were analyzed by fitting it to an equivalent circuit model using the built-in fitting tool of the CH Instruments potentiostat software (CHI version 19.05, CH Instrument, Inc., Austin, TX, USA). Initial parameter values for circuit elements were assigned to 0, and the software iteratively optimized them to achieve the best fit. The simulated data were then generated, and the goodness of fit was evaluated by examining residuals and chi-square values.

The specific capacitance of the electrode was calculated from the CV curve using Equation (2).

$$C = \frac{A}{mk\Delta V} \quad (2)$$

where C = specific capacitance, A = area of the CV plot, m = mass loading in electrode, k = scan rate, and ΔV = voltage range of CV.

3. Results and Discussion

3.1. Optimizing Electrode Preparation for Better Performance

The electrochemical performance of MXene-based electrodes is highly dependent on the electrode preparation method. In this study, three different electrode fabrication techniques—drop-cast DI-MXene, drop-cast DMSO-MXene, and punch-paste MXene—were evaluated to determine their effectiveness in achieving uniform surface coverage and optimal electrochemical behavior.

The drop-cast DI-MXene electrode, upon drying, showed cracks and failed to cover the GC surface completely. The microscopic image of this drop-cast electrode is shown in Figure 2b. This is attributed to the loss of interlayer water molecules during drying and grinding, which reduces MXene's hydrophilicity, making dispersion in DI water challenging [30].

On the other hand, as DMSO provides better dispersion media of MXene than DI water, drop-cast DMSO-MXene electrode resulted in a crack-free film. This film fully covered the

GC surface (Figure 2c). However, drying this electrode at room temperature required over 10 h, which could be impractical for large-scale applications.

An alternative approach involved preparing a freestanding MXene film, which was then punched into a 3 mm disk and adhered to the GC surface using a DI water/Nafion binder solution. This method offered a uniform and smooth surface of the MXene film with better adhesion with the GC surface (Figure 2d). Additionally, the drying time was significantly reduced to less than one hour, making this method more time-efficient. In the inset of Figure 2d the punched film is shown.

Figure 3 presents a comparison of CV curves obtained from the electrodes prepared using three different methods. The results indicate that the drop-cast DI water-MXene electrode showed poor capacitance, masking the true electrochemical potential of MXene. This limitation may be attributed to the cracked and incomplete surface coverage, which exposed the GC surface and hindered charge storage. In contrast, the punch-paste MXene electrode demonstrated the highest capacitance, attributed to its smooth, uniform surface and strong adhesion to the GC electrode. The reduced drying time and improved electrode integrity resulted in more efficient charge transfer and better electrochemical stability. Consequently, the punch-paste electrode preparation method was selected for subsequent electrochemical analyses.

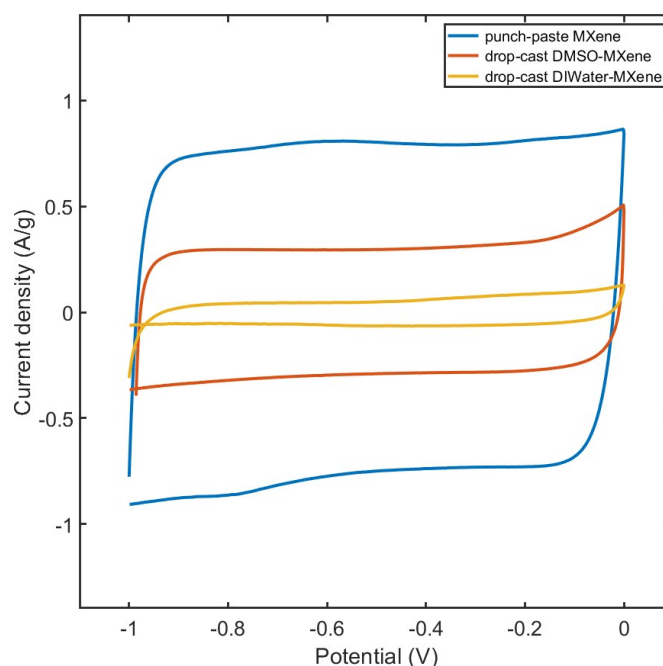


Figure 3. CV curves for MXene electrodes prepared by drop-casting DI water-MXene solution, drop-casting DMSO-MXene solution, and pasting MXene film.

3.2. Suitable Potential Window for Charge Storage

MXene electrode was characterized by CV in the negative potential window ranging from -1 V to 0 V, where it exhibited high stability with negligible changes during the initial cycles. The CV curves for the 1st and the 10th scan are shown in Figure 4a. The CV curves are substantially similar, indicating electrochemical stability of MXene in this potential range. Although MXenes are primarily pseudocapacitive in nature, the CV curves in Figure 4a display a nearly rectangular shape, resembling electric double-layer capacitor (EDLC) behavior. Unlike conventional faradaic materials, MXenes exhibit EDLC-like symmetric charge–discharge behavior with no pronounced redox peaks due to several key factors. Their redox reactions occur at or near the surface where the surface termi-

nations participate in the redox reaction. Their hydrophilic surfaces, rich in functional groups ($-\text{OH}$, $-\text{O}$, and $-\text{F}$), provide high ion accessibility, facilitating fast redox kinetics and preventing sluggish battery-type behavior. This enables ultra-fast and reversible charge transfer that mimics electrical double-layer capacitance (EDLC). Additionally, their high metallic conductivity allows rapid electron transport, minimizing resistive voltage drops and contributing to an ideal capacitive response. The rounded edges observed in the CV curves are attributed to internal resistances inherent to electrochemical processes.

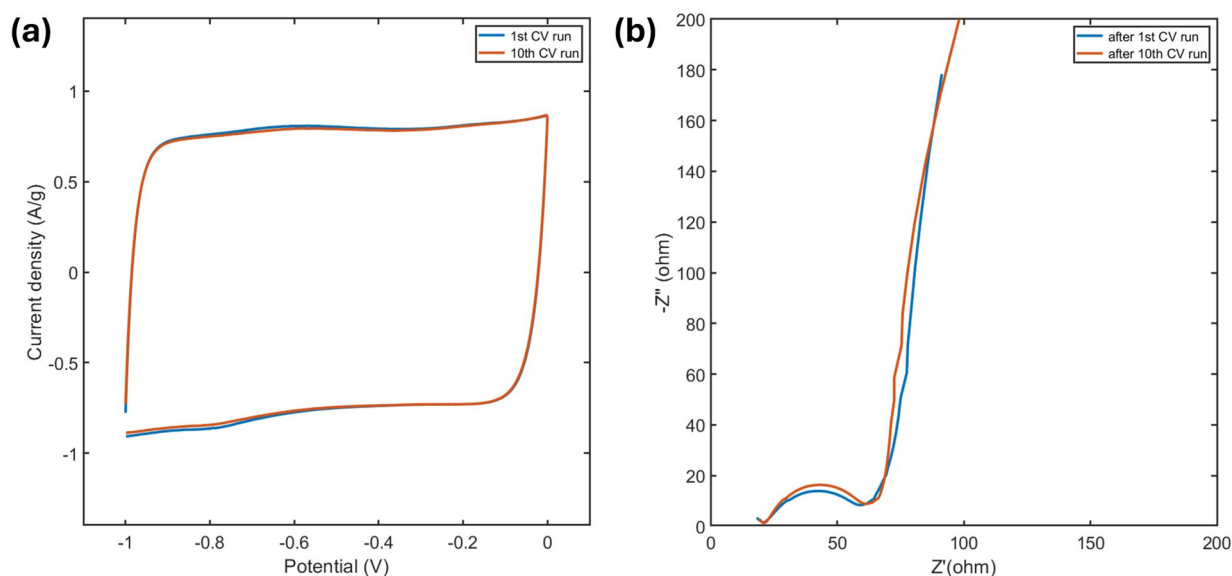


Figure 4. (a) 1st and 10th CV scan of MXene in cathodic potential window at a scan rate of 5 mV/s, (b) Nyquist plots obtained after the 1st and 10th cathodic CV run at a bias voltage of 0 V.

A slight peak appears at approximately -0.6 V during charging and another at -0.8 V during discharging. These peaks suggest the occurrence of a relatively slower Faradaic redox reactions, likely facilitated by intercalation or diffusion of electrolyte ions at the electrode surface, which contribute to the pseudocapacitive behavior of MXenes in this potential window [31]. The nature of this slower redox reaction has been studied in more detail in Section 3.6.

The Nyquist plots obtained from the EIS study of MXene after 1st and 10th CV run in the negative potential window are shown in Figure 4b. The applied bias voltage is 0 V. At very high frequencies, ions cannot penetrate pores or layers due to insufficient time, leaving only the external surface accessible, which makes resistive components dominant [32,33]. Conversely, at very low frequencies, ions fully penetrate the layers, charge saturation dominates, and the electrode behaves as an ideally polarizable porous interface, resulting in a vertical line on the Nyquist plot. In the intermediate frequency range, resistance and capacitance become frequency-dependent, and diffusion processes play a significant role [34]. In Figure 4b, the plots exhibit depressed semicircles at high frequencies which is a characteristic of Faradaic charge transfer processes [35]. In each plot, a deviated vertical low-frequency line is observed followed by the semicircle, indicating electrostatic charge storage mechanisms such as ion intercalation. In addition to that, this vertical low frequency line also indicates absence of leakage resistance, signifying negligible self-discharge [36]. However, as the low-frequency line deviates slightly from an ideal capacitive behavior, this indicates electrolyte ion diffusion and resulting adsorption of oxidized MXene [37].

No notable alteration in the charge transfer resistance or the capacitive behavior was observed from Figure 4b after the first 10 cathodic runs, again emphasizing the stabil-

ity of MXene in this potential window and its effectiveness as an electrode material for electrochemical applications.

Figure 5a presents the first two anodic CV scans from -1 V to 1 V at a scan rate of 5 mV/s. During the first scan, a prominent peak appears around 0.8 V, but it disappears in subsequent scans and does not reappear even after sweeping the electrode in negative potentials. This behavior indicates an irreversible oxidation reaction occurring at this voltage. Tian et al.'s study on the potential-dependent oxidation of $\text{Ti}_3\text{C}_2\text{O}_2$ MXene, using grand canonical free energy curves, supports this observation. They reported an oxidation potential of 1.3 V for MXene attacked by water [38]. Experimentally, this potential is found to be significantly lower, around 1.094 V vs. the standard hydrogen electrode (SHE, $\text{pH} = 0$) [39]. Tian et al. found that presence of defects or oxygen-containing species in the carbon layer can lower the oxidation potential by nearly 1 V [38].

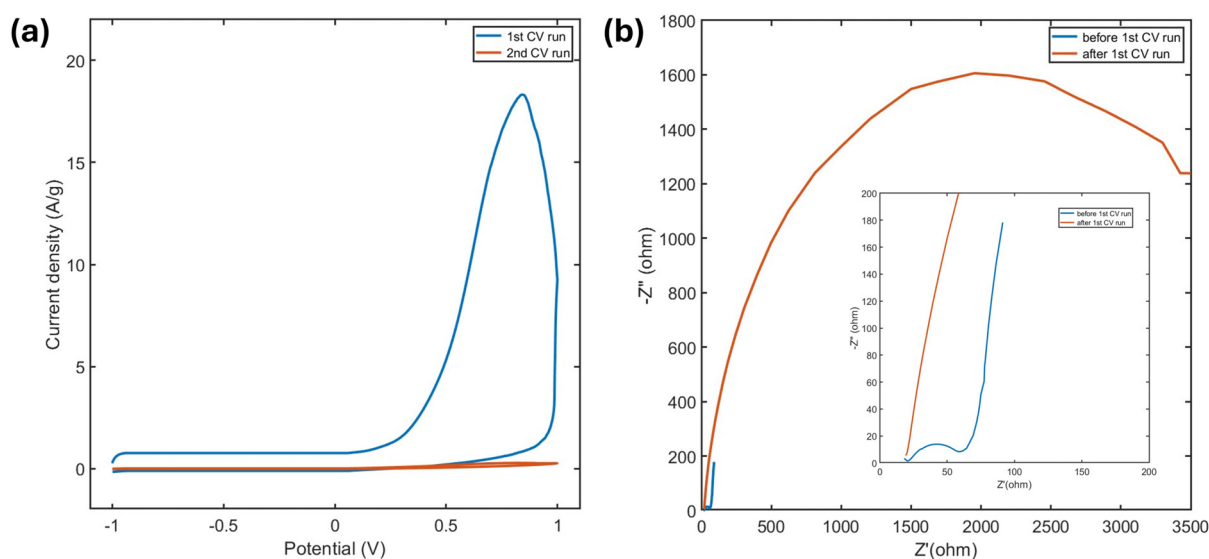


Figure 5. (a) First two CV scans of MXene in anodic potential window at a scan rate of 5 mV/s, (b) Nyquist plot obtained before and after the 1st anodic CV run.

Further insights into this process were gained from EIS conducted before and after the anodic CV sweep. The Nyquist plot (Figure 5b) shows a substantial enlargement of the semicircle after the anodic run, indicating a significant increase in charge transfer resistance. This behavior confirms the deposition of an insulating oxide layer on the MXene surface.

From these findings and the CV data, it can be inferred that MXene is oxidized in the presence of aqueous electrolyte in the anodic potential window. Oxidation begins at approximately 0.2 V and shows a peak at around 0.8 V, forming an irreducible titanium oxide layer. This oxide layer acts as a solid barrier, significantly hindering charge and ion transfer and drastically reducing the capacitance of the MXene electrode.

To further support this claim, XRD spectra and SEM images were analyzed. The SEM image of $\text{Ti}_3\text{C}_2\text{T}_x$ MXenes (Figure 6a) shows that the $\text{Ti}_3\text{C}_2\text{T}_x$ MXenes surface is non-porous with a smoother surface. The etched MXene flakes are aligned to make a layered film structure during vacuum filtration. However, after the anodic oxidation in CV scan, the $\text{Ti}_3\text{C}_2\text{T}_x$ MXene transforms into a porous structure (Figure 6b), where the layered structure is not retained anymore. Additionally, the elemental analysis (atomic number %) obtained from EDS, displayed in the inset of Figure 6a,b, demonstrated a significant increase in oxygen content after the anodic oxidation. It provides support on the claim that $\text{Ti}_3\text{C}_2\text{T}_x$ MXenes converts into oxides when run on the positive potential range. To further demonstrate the nature of oxidation, XRD spectra were obtained for $\text{Ti}_3\text{C}_2\text{T}_x$ MXenes before

and after the anodic CV run. The XRD spectra of MXenes after the anodic run revealed the presence of rutile TiO_2 phases as it showed peaks at $2\theta = 27.4^\circ$, 36.07° , 54.28° and 56.57° , as shown in Figure 6c. Before the anodic run, XRD spectra show characteristic $\text{Ti}_3\text{C}_2\text{T}_x$ MXene (002) peak at $2\theta = 5.9^\circ$, which corresponds to an interlayer space of 3 nm. This interlayer space provides space for ion intercalation during the charging process, which facilitates high capacitance. However, after anodic oxidation, the disappearance of the (002) peak suggests that the layered structure of MXene is disrupted due to oxidation, contributing to the loss of capacitance observed after anodic oxidation. These findings confirm that MXenes are highly stable in the negative potential window but are electrochemically unstable in the positive potential window due to oxidation reactions caused by water attack from the aqueous electrolyte, which results in reduced capacitance.

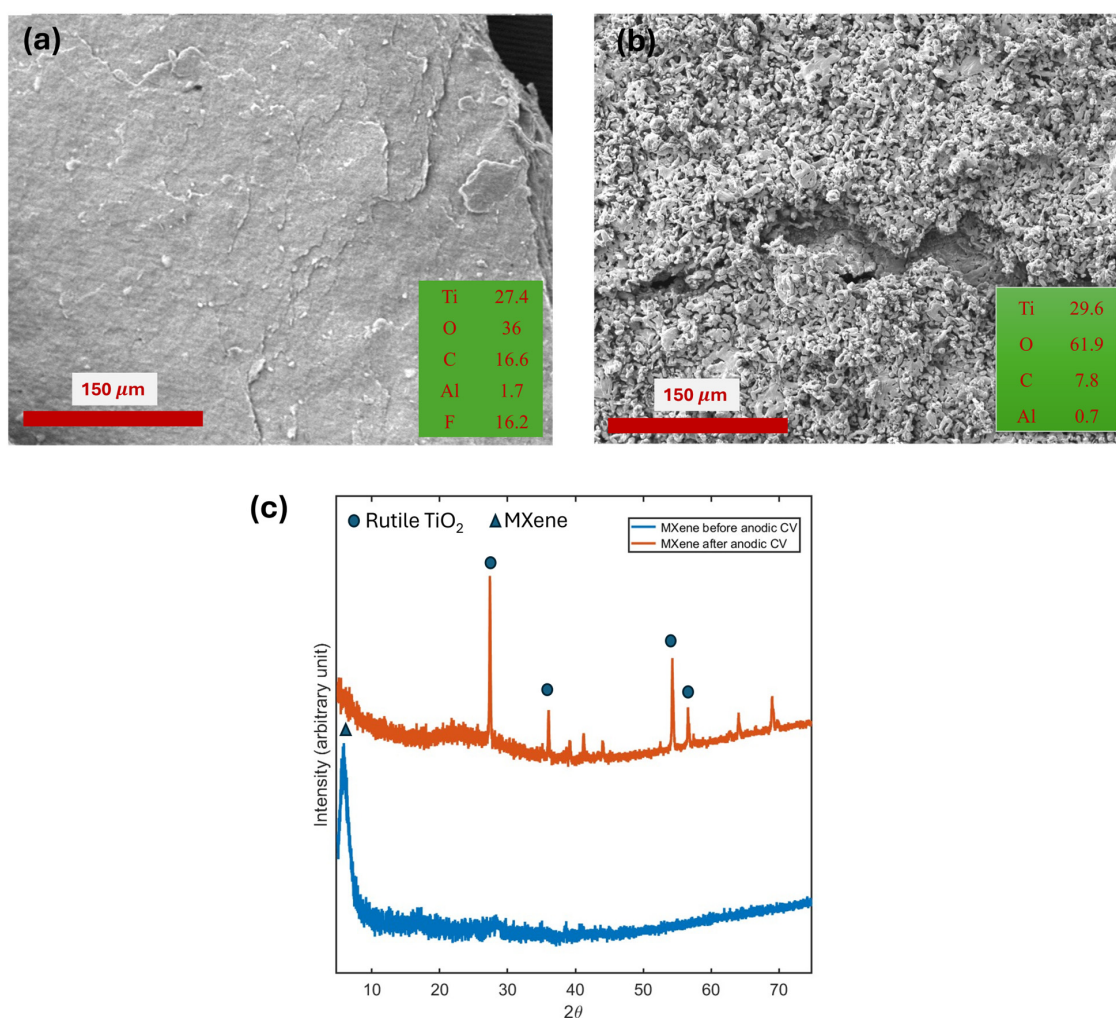


Figure 6. SEM image of MXene (a) before and (b) after anodic run. EDS results in atomic % is shown in inset. (c) XRD data before and after anodic run.

3.3. Developing EC Model

The optimization of supercapacitors requires advanced modeling techniques to accurately capture the physical phenomena occurring at the material scale and to characterize their electrical performance effectively. In EIS, these phenomena can be modeled using ECs. A commonly used model for supercapacitors is the Randles circuit. It consists of a parallel connection of charge-transfer resistance (R_{ct}) and capacitance (C), which represent faradaic processes. Ion diffusion is modeled through a finite Warburg impedance (W) connected in series with R_{ct} . This Warburg impedance assumes that diffusion is inherently coupled to

charge transfer in faradaic reactions [40]. Additionally, a series resistance (R_s) accounts for electrical and connective losses at high frequencies.

The Randles circuit simplifies the complex interactions in MXene-based systems, often overlooking critical phenomena such as non-uniform charge distribution and dynamic interactions at the electrode–electrolyte interface [41]. Furthermore, the circuit model does not adequately account for the mixed ionic–electronic conduction behavior of MXene, leading to less accurate predictions of performance in hybrid systems [21]. Hence, in this study a modified EC is proposed for MXene electrode in supercapacitor application.

The model was designed to meet two key criteria: it needed to be accurate enough to represent the main physical processes occurring at the material level while being simple enough for practical use in in situ characterization.

Circuit-a, as shown in Figure 7a, represents the basic Randles circuit. However, the simulated Nyquist plot generated using this circuit failed to accurately depict the MXene electrode system due to its simplicity, resulting in an error percentage of 19.3% compared to actual data. To modify this circuit, Circuit-b (Figure 7b) was introduced, incorporating an additional capacitive element (C_2) in series with Circuit-a. This element represents the double-layer capacitance of blocked electrodes, such as those in supercapacitors [42]. The simulated plot using Circuit-b showed improved accuracy, reducing the error percentage to 14.1%.

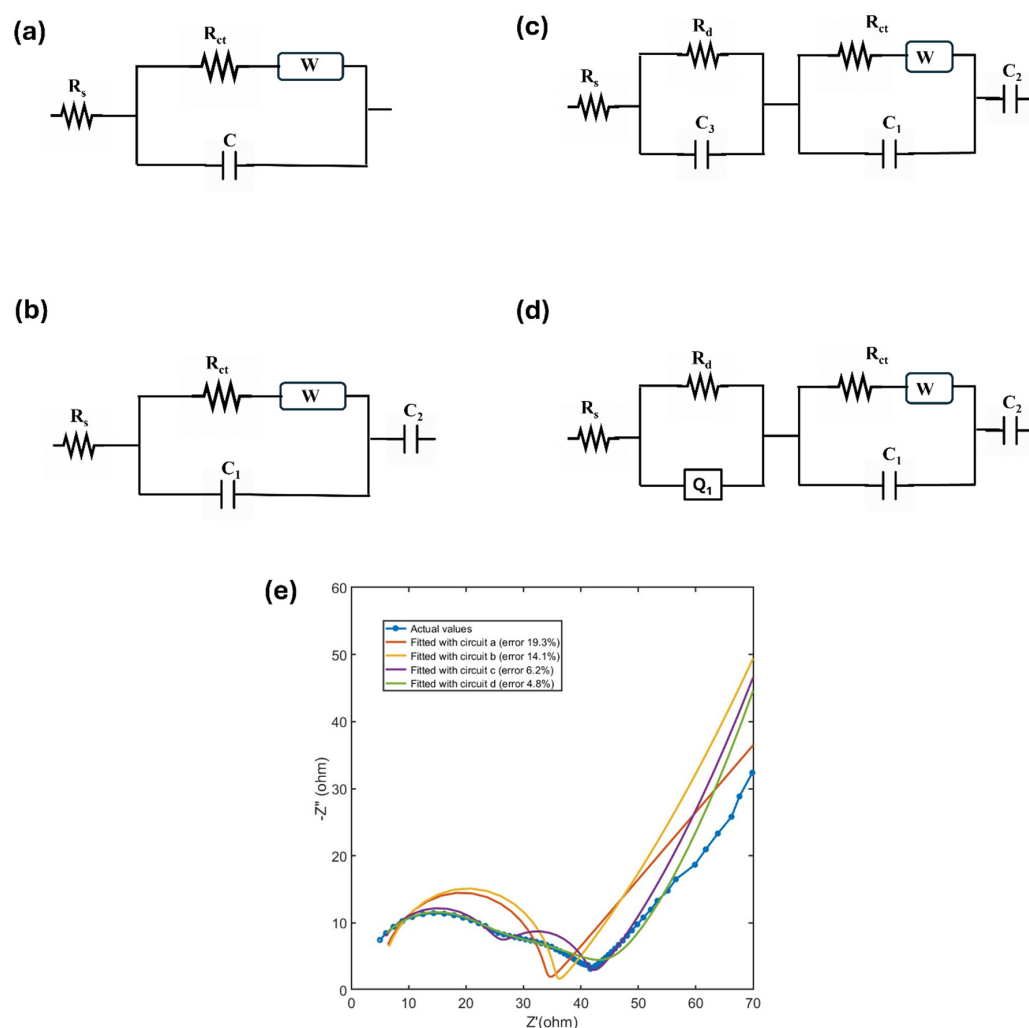


Figure 7. (a–d) Different ECs studied. (e) Fitted data derived from the ECs vs. the actual recorded data in the Nyquist plot.

$\text{Ti}_3\text{C}_2\text{T}_x$ MXene undergoes aqueous oxidation in the anodic potential range, and over repetitive cycling. This results in a second semicircle in the Nyquist plot due to oxide layer formation as will be discussed in the next two sections. To account for this phenomenon, Circuit-c (Figure 7c) was developed by adding a capacitive element (C_3) in parallel with a resistance (R_d) and placing it in series with the original circuit. This addition accounts for the diffusion resistance arising from the formation of the solid oxide layer. Similar strategies have been employed in the literature to model the Solid Electrolyte Interphase (SEI) in lithium-ion batteries [43]. This circuit further reduced the error percentage to 6.2%.

The second semicircle in the Nyquist plot deviates from the perfect round shape. This deviation might be due to practical resistive factors, like surface roughness, chemical inhomogeneity, and heterogeneous electrode–electrolyte interfaces caused by ion adsorption [44]. One notable modification to account for these phenomena is the use of constant phase elements (CPEs), denoted as Q_1 , instead of conventional capacitance (C_3). CPEs account for real life application factors mentioned above and results in a depressed semicircle on the Nyquist plot, with its center falling below the horizontal axis [31]. This modified circuit (circuit-d) is shown in Figure 7d, which further reduced the error percentage to 4.6%.

Apart from the model assumptions, the error margin resulted from fitting EIS data using different ECs may also include other random errors, such as background noise, inconsistencies in electrode preparation, and environmental factors such as temperature fluctuations. To mitigate these potential errors and enhance the reliability of our analysis, three independent sets of fittings were conducted using each proposed circuit model and averaged the results to ensure consistency and reduce statistical bias.

The fitted Nyquist plots simulated from these four circuits are shown in Figure 7e, and circuit d best resembles the recorded impedance behavior of MXene electrode while maintaining a practical level of complexity.

3.4. Voltage Dependent Electrochemical Behavior Using EC

EIS data have been recorded applying different bias voltages in both anodic and cathodic regions. This provides detailed insights into the electrochemical behavior of the MXene electrode in these ranges.

In the cathodic range, the Nyquist plots retain a similar shape (Figure 8a), but the Warburg region shifts to the right as the bias voltage becomes more negative. This shift reflects an increase in diffusion resistance with increasing negative potential.

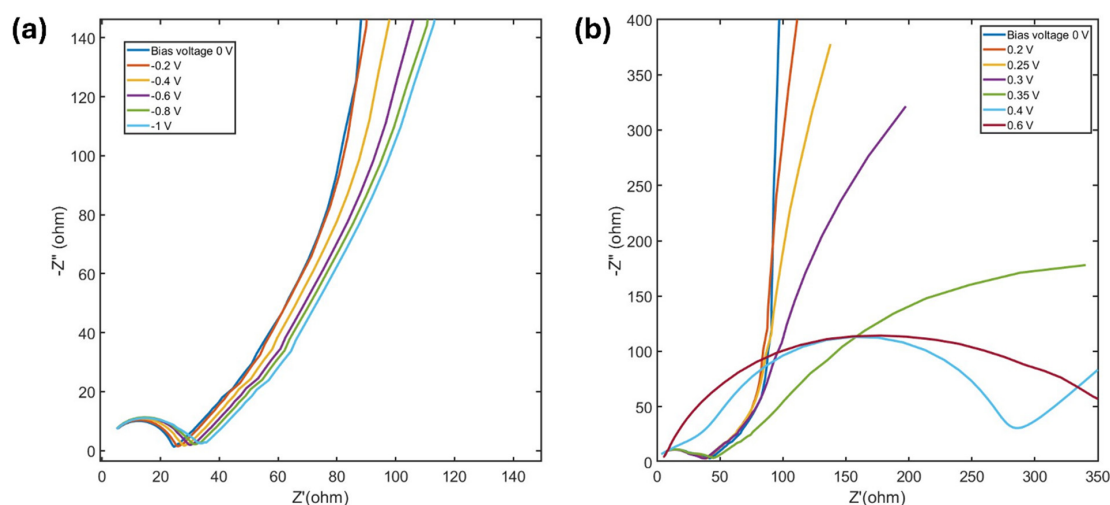


Figure 8. Nyquist plot of MXene electrode obtained from recorded EIS data at different bias voltage in (a) cathodic potential range and (b) anodic potential range.

Figure 8b shows Nyquist plots for various anodic bias. The curves at 0 V, 0.2 V and 0.25 V show a semicircle and a sloping line. Interestingly, at 0.3 V, a second semicircle appears in the Nyquist plot, which further enlarges as the potential is increased. This second semicircle, typically observed in the lower frequency region, often arises from adsorption and desorption processes at the electrode–electrolyte interface [45,46]. It is often linked to the formation of a solid interphase film [47]. In the MXene electrode, as previously discussed, water molecules are adsorbed at anodic potentials, and a solid oxidized MXene layer begins to form at around 0.3 V. Thus, the second semicircle at 0.3 V represents both the adsorption of water molecules and the onset of the oxide layer’s formation. From 0.4 V, the first semicircle becomes significantly larger, indicating increased polarization resistance. This resistance results from the oxide layer formation, which inhibits Faradaic currents and limits subsequent electrochemical processes detectable within the test frequency range.

In the low-frequency region, the sloping line reflects the Warburg impedance, associated with ion diffusion. The ion diffusion coefficient (D) can be determined using Equation (3) [48].

$$D = 0.5R^2T^2/S^2n^4F^4C^2\sigma^2 \quad (3)$$

where R is the gas constant, n is the number of electrons, F is the Faraday constant, and σ is the Warburg factor. Thus, D is inversely proportional to σ^2 .

In Figure 8b, the slope of the Warburg region is infinite at lower voltage, which depicts capacitive behavior and tends to bend down towards finite space with increasing voltage. This change in slope indicates a decrease in D and suggests reduced ion diffusion at higher voltages prior to oxide layer formation. This also indicates a reduction in capacitive behavior in higher positive potential. Hence, it can be concluded that, due to a resistive oxide layer formation, the capacitance of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene reduces dramatically at voltages higher than 0.3 V.

Table 1 summarizes the values of resistances (R_s , R_{ct} , and R_d) obtained from the developed EC model. The series resistance (R_s) remains consistently low and shows minimal variation across all bias voltages. The charge transfer resistance (R_{ct}) initially decreases slightly up to 0.3 V, as the higher potential facilitates charge transfer. However, beyond this point, R_{ct} increases sharply, corresponding to the formation of an insulating oxide layer. Similarly, the diffusion resistance (R_d) increases slightly as the voltage increases to 0.3 V but then rises significantly due to the greater diffusion hindrance caused by the oxidized layer.

Table 1. Values of the resistances (R_s , R_{ct} , and R_d) and CPE values obtained from the EC model.

Voltage (V)	R_s (Ω)	R_{ct} (Ω)	R_d (Ω)
−1	0.001	32.41	14.93
−0.8	0.001	31.65	14.94
−0.6	0.001	30.3	14.95
−0.4	0.001	29.8	14.99
−0.2	0.001	28.13	15.01
0.0	0.001	27.2	15.0
0.2	0.001	26.26	15.03
0.25	0.001	24.44	15.23
0.3	0.001	23.96	15.96
0.35	0.001	31.38	19.51
0.4	0.001	72.86	233.3
0.6	0.001	558	1570

3.5. Analysis of Cyclic Stability Using Developed EC

As $\text{Ti}_3\text{C}_2\text{T}_x$ MXene undergoes charging and discharging cycles, it faces a degradation in capacitance. To understand cyclic stability, $\text{Ti}_3\text{C}_2\text{T}_x$ MXene was characterized using CV at a scan rate of 100 mV/s over 1000 cycles, and the changes in capacitance and resistances were analyzed. Initially, the MXene exhibited a capacitance of 493 F/g at this scan rate. After 1000 cycles, the capacitance decreased to 357 F/g, reflecting a retention of 72.5%. Figure 9a illustrates the corresponding CV curves at various cycle intervals. This reduction in capacitance can be attributed to the adsorption of water molecules and/or oxidation of MXene and the resulting increase in resistance, as analyzed through Nyquist plots and the developed EC model.

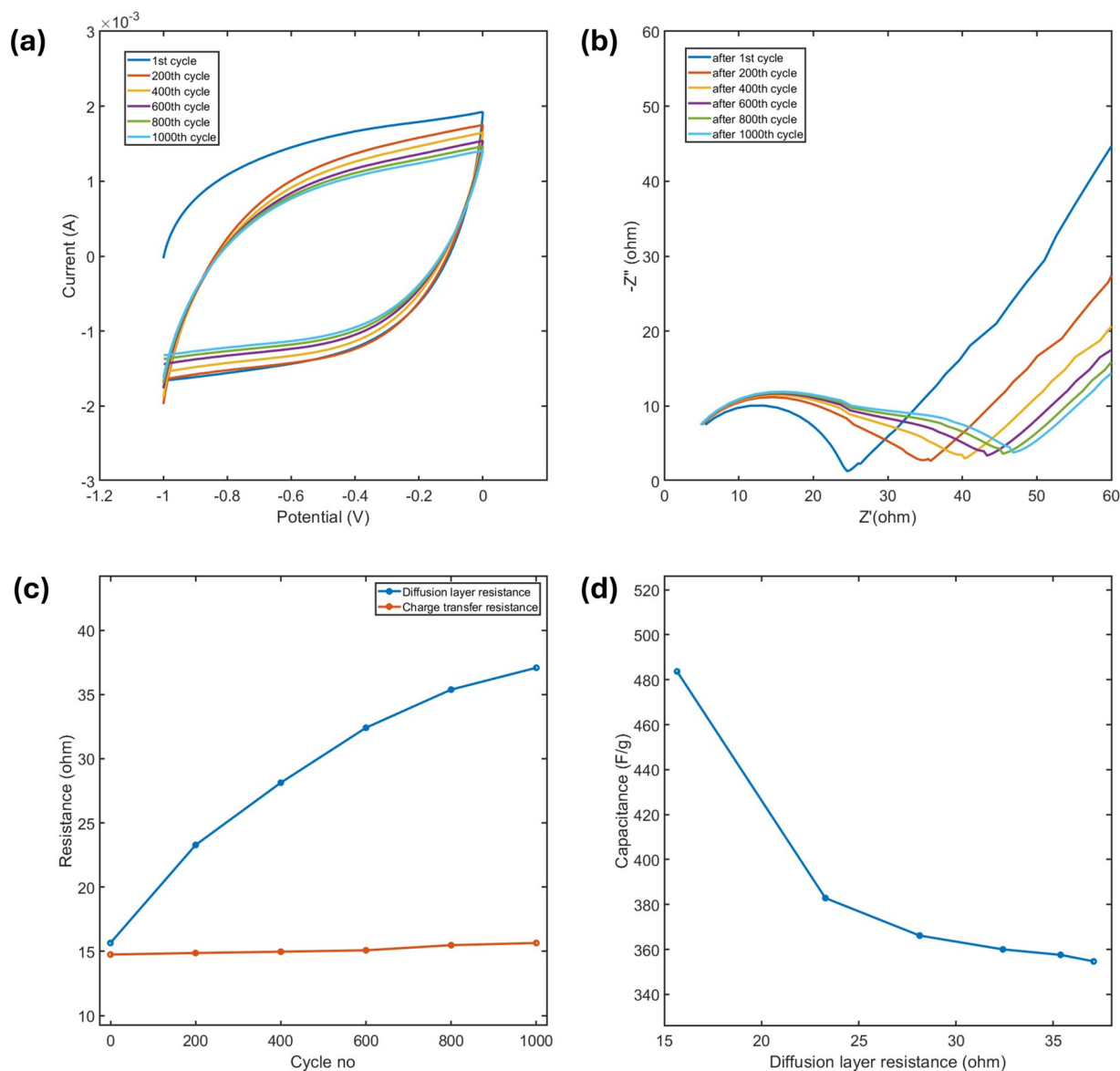


Figure 9. (a) CV curves of MXene at 100 mV/s over different cycle intervals. (b) Nyquist plots at various cycles. (c) Variation in charge transfer resistance (R_{ct}) and diffusion resistance (R_d) with cycling. (d) Correlation between the reduction in capacitance and the increase in diffusion layer resistance (R_d) during cycling.

The Nyquist plots at different cycle numbers, shown in Figure 9b. Initially, only a single semicircle, corresponding to charge transfer resistance (R_{ct}), is observed. However, after 200 cycles, a second semicircle begins to appear, becoming more prominent with

continued cycling. This second semicircle can be attributed to the adsorption of water molecules and gradual formation of a resistive layer likely due to oxidation, as discussed previously. Using the EC model, the charge transfer resistance (R_{ct}) and the diffusion resistance (R_d) were quantified. Figure 9c highlights the progressive increase in R_{ct} and R_d with the number of cycles. The rate of R_d increase is initially higher, likely because a larger MXene surface is exposed to water attack. As oxidation proceeds, and a resistive layer forms, it inhibits further water diffusion, causing the oxidation rate to slow. In contrast, R_{ct} increases at a comparatively slower rate, with its underlying cause requiring further investigation in future studies.

Figure 9d shows a reduction in capacitance as the R_d increases with cycling. These findings underscore that the cycling stability of $Ti_3C_2T_x$ MXene in aqueous electrolytes is primarily governed by its susceptibility to oxidation and the associated rise in resistances. This highlights the need for strategies to mitigate oxidation and enhance the long-term performance of MXene-based energy storage systems.

3.6. Analysis of Surface-Controlled and Diffusion-Controlled Capacitance

Figure 10a shows CV curves of the MXene electrode at various scan rates. The variation in capacitive current (i) with scan rate (v) provides insight into whether surface-controlled reactions or diffusion-controlled processes dominate the charge storage mechanism. To better understand the charge storage behavior during the charge–discharge process, a series of kinetic analyses were performed on the electrode at specific charging and discharging states.

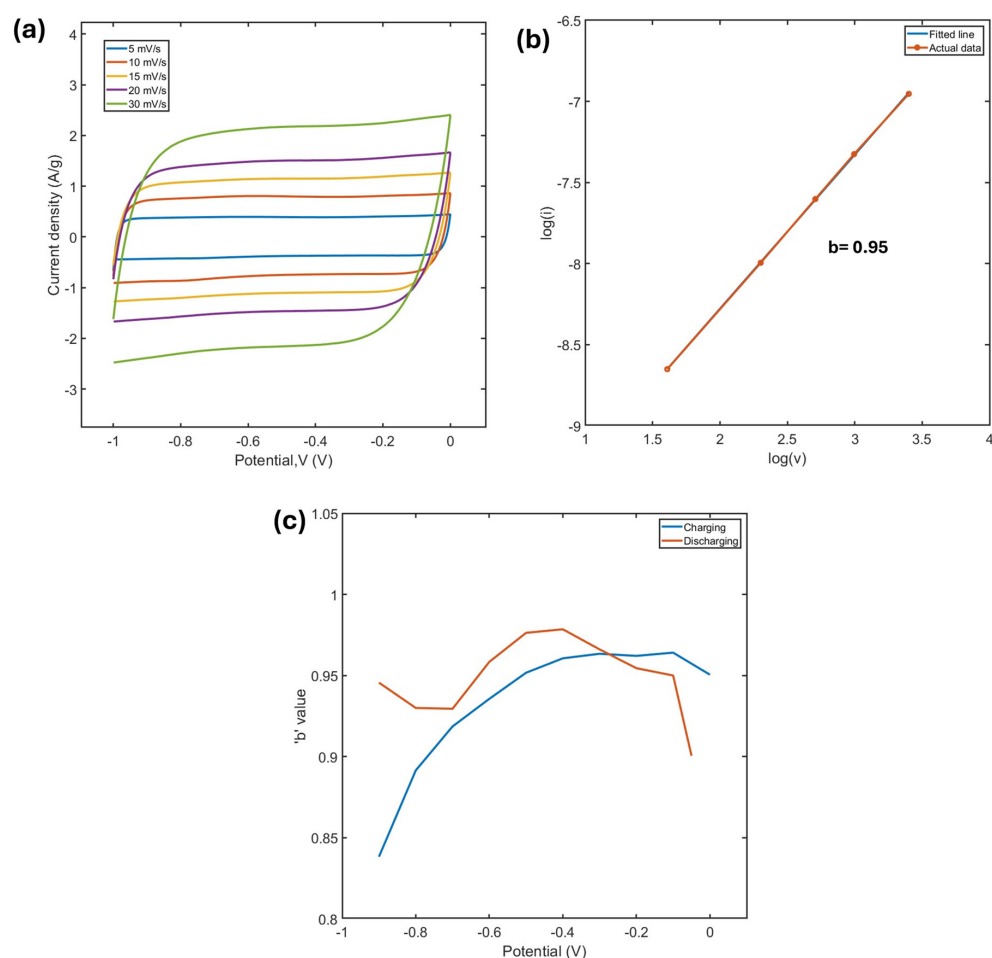


Figure 10. (a) CV plots of MXene at different scan rates (b) linear fitting of $\log(i)$ vs. $\log(v)$ to calculate b value (c) variation in b values at different cathodic potential in both charging and discharging condition.

The b value calculated from Equation (4) serves as a critical indicator of the dominant charge storage mechanism of the pseudocapacitive electrode [16]:

$$\log i = \log a + b \log v \quad (4)$$

b can be derived from plotting the logarithm of the current ($\log i$) at a given potential as a function of the logarithm of the scan rate ($\log v$), if the plot generates a linear relationship. Then, b is obtained by calculating the slope of the resulting curve using Equation (4). When $b \approx 1$, the charge storage process is dominated by surface-controlled behavior, whereas $b \approx 0.5$ indicates a diffusion-controlled mechanism. For the MXene electrode, Figure 9b demonstrates the linear relationship of $\log i$ vs. $\log v$ at a specific potential with b being close to 1.

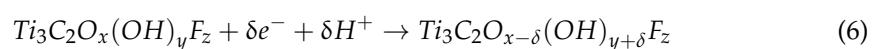
Figure 9c highlights the b value obtained from the kinetic analysis at various potentials during the charging and discharging branches of the CV curves. The b values remain close to 1 across most of the voltage range during both charging and discharging. This suggests that surface-controlled reactions primarily dominate the electrochemical process. However, at the beginning of the charging and discharging cycles, the b values decrease. This reduction is attributed to the rounding edges observed in the CV curves (instead of a pure rectangular form) during these stages, indicating a deviation from the ideal capacitive behavior caused by internal resistances. The relationship between $\log i$ and $\log v$ in this range is not perfectly linear, leading to a compromised slope. Additionally, a slight decrease in b values is observed in the potential range of -0.5 V to -0.8 V. This reduction indicates that the charge accumulation in this potential range is associated with a more diffusion dominant, slower process, which can be supported by the redox peaks observed in this potential in Figure 5a. Furthermore, the redox peak at this potential becomes negligible as the scan rate is increased (Figure 10a), which is likely because at higher scan rates, this slower, diffusion-related process becomes less dominant due to time constraints.

A more quantitative approach is adopted to separate the capacitive effects into surface (pseudocapacitive) and diffusion-controlled (Faradaic) processes. Surface capacitance is primarily related to charge storage on the electrode surface and can be calculated by isolating the k_1 term in Equation (5).

$$i = k_1 v + k_2 v^{0.5} \quad (5)$$

where i is the current, v is the scan rate, k_1 accounts for the surface-controlled process, and k_2 represents the diffusion-controlled contribution. By plotting $i/v^{0.5}$ against $v^{0.5}$, the intercept gives k_2 , and the slope provides k_1 . The respective capacitances are then derived based on these constants.

The calculation of capacitive and diffusion-limited contributions to the total capacitance reveals that the overall capacitance in MXene is predominantly surface-controlled (Figure 11a). However, there is a noticeable contribution from diffusion-limited processes, particularly near the peaks observed in the CV curves. In MXene, two distinct pseudocapacitive charge storage mechanisms coexist: first, electrostatic charge accumulation due to ion intercalation within the interlayer space, second, Faradaic protonation at the O-terminated surface, which can be expressed by Equation (6) [16,49].



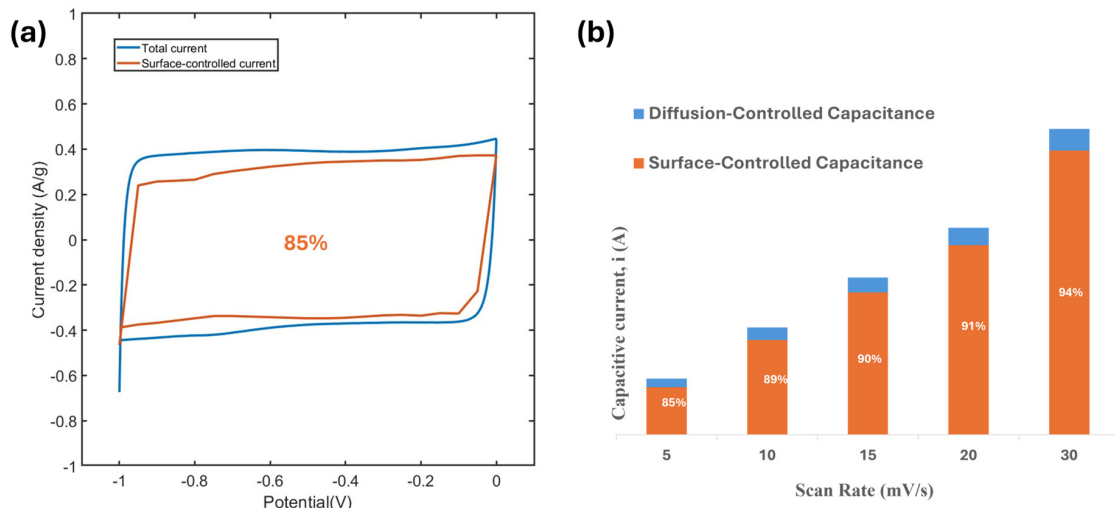


Figure 11. (a) Surface controlled capacitance in comparison with the total capacitance. (b) Variation in surface-controlled capacitance at different scan rates.

These mechanisms divide the overall electrochemical behavior into two parts. The rectangular-shaped regions of the CV curve correspond to surface-controlled pseudocapacitance, characterized by a b value close to 1, indicating rapid, non-diffusion-dependent charge storage. In contrast, the peaks in the CV curve are associated with diffusion-controlled Faradaic charge accumulation processes.

The surface-controlled contribution is further increased with increasing scan rate as depicted in Figure 11b. This indicates that at higher scan rate, diffusion becomes even less dominant, likely because of the intrinsic time limitations.

These findings highlight the complex interplay between surface and diffusion phenomena in MXene, contributing to its high electrochemical performance and versatility as an electrode material.

Table 2 summarizes key performance metrics of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene in comparison to other conventional materials, highlighting both its advantages and limitations in the context of supercapacitor applications.

Table 2. Comparison of basic properties of MXene family and other materials.

Type	Electrical Conductivity($\text{S}\cdot\text{m}^{-1}$)	Specific Capacitance	Capacitance Retention	Challenges	Refs.
Carbon materials (EDLC)	$\sim 10^6$ (Carbon nanotube) $\sim 10^8$ (Graphene)	200–300 F/g (Carbon nanotube) 100–200 F/g (Graphene)	>90% after 10,000 cycles	Low energy density due to absence of redox reactions	[50–52]
Conductive polymers (Pseudocapacitance)	$\sim 10^5$	200–500 F/g	50–60% after 1000 cycles	Poor mechanical stability, swelling, and contraction during cycling leads to degradation Oxidation in ambient air,	[53,54]
MXene (Pseudocapacitance)	$\sim 10^5 - 10^6$	493 F/g [this work]	72.5% after 1000 cycles [this work]	restacking of layers, potential degradation in aqueous media	[16]

Despite its oxidative stability challenges, $\text{Ti}_3\text{C}_2\text{T}_x$ MXene stands out as a promising supercapacitor material due to its exceptionally high capacitance, fast charge transfer kinetics, and predominantly surface-controlled charge storage, making it a strong candidate for high-performance energy storage applications when suitable stabilization strategies are implemented.

4. Conclusions

This study provides a comprehensive analysis of the electrochemical behavior of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene. An equivalent circuit was developed to accommodate the diffusion resistance arising from the gradual formation of the solid oxide layer as well as other practical factors that inhibit ideal capacitance development. The developed equivalent circuit model captures the impedance behavior of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene with a low error margin of 4.6%. Analysis of the EIS data using the developed model suggests oxidation-related limitations in applying $\text{Ti}_3\text{C}_2\text{T}_x$ MXene as supercapacitor electrode. In negative potential window (−1 to 0 V), $\text{Ti}_3\text{C}_2\text{T}_x$ MXene shows better electrochemical stability. However, the capacitance degrades from 493 F/g to 357 F/g with a 72.5% retention after 1000 cycles due to aqueous oxidation. In the positive potential range (0 to 1 V), this MXene shows an irreversible oxidation reaction that makes it unsuitable for anodic use. The contributions of surface-controlled and diffusion-controlled processes were also quantified, highlighting the dominance of surface functional groups in developing pseudocapacitive behavior.

This research addresses the challenges associated with determining suitable potential windows and obtaining cyclic stability of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene. While MXene exhibits a significantly high specific capacitance, its stability is hindered by oxidative degradation over repeated cycles in aqueous electrolytes. To enhance its long-term stability in supercapacitor applications, future research should focus on strategies to suppress oxidative degradation, such as surface functionalization, electrolyte modification, or protective coatings. Further advancements in equivalent circuit modeling may also improve the predictive accuracy of impedance analysis, enabling the better optimization of MXene-based energy storage systems.

Author Contributions: N.A.: Designing and planning the experiments, synthesis and characterization of materials, data analysis, and writing the primary draft of the manuscript. A.A.N.: Data analysis and writing draft of the manuscript. D.S.: Electrochemical characterization. M.M.R.: Reviewing and editing. R.A.L. and O.I.O.: Overall supervision and reviewing and editing. All authors have read and agreed to the published version of the manuscript.

Funding: The experiments were funded in part by National Science Foundation (NSF) #1950500.

Data Availability Statement: The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding author(s).

Acknowledgments: The authors would like to acknowledge the financial support from National Science Foundation (NSF).

Conflicts of Interest: The authors declare no conflict of interest.

Abbreviations

EC	Equivalent Circuit
EIS	Electrochemical Impedance Spectroscopy
CV	Cyclic Voltammetry
GCD	Galvanostatic Charge–Discharge
CPE	Constant Phase Element
EDLC	Electric Double-Layer Capacitance
MILD	Minimally Intensive Layer Delamination
GC	Glassy Carbon

References

- Steffan, S.; Semrau, G.L. High Power Density Modular Electric Power System for Aerospace Applications. In Proceedings of the 12th International Energy Conversion Engineering Conference, Cleveland, OH, USA, 28–30 July 2014; AIAA Propulsion and Energy Forum; American Institute of Aeronautics and Astronautics: Reston, VA, USA, 2014.
- Shchegolkov, A.V.; Lipkin, M.S.; Shchegolkov, A.V. Preparation of WO_3 Films on Titanium and Graphite Foil for Fuel Cell and Supercapacitor Applications by Electrochemical (Cathodic) Deposition Method. *Russ. J. Gen. Chem.* **2022**, *92*, 1161–1167. [\[CrossRef\]](#)
- Syamsai, R.; Grace, A.N. Ta_4C_3 MXene as Supercapacitor Electrodes. *J. Alloys Compd.* **2019**, *792*, 1230–1238. [\[CrossRef\]](#)
- Akhter, R.; Maktedar, S.S. MXenes: A Comprehensive Review of Synthesis, Properties, and Progress in Supercapacitor Applications. *J. Mater.* **2023**, *9*, 1196–1241. [\[CrossRef\]](#)
- Borah, A.J.; Natu, V.; Biswas, A.; Srivastava, A. A Review on Recent Progress in Synthesis, Properties, and Applications of MXenes. *Oxf. Open Mater. Sci.* **2025**, *5*, itae017. [\[CrossRef\]](#)
- Ronchi, R.M.; Arantes, J.T.; Santos, S.F. Synthesis, Structure, Properties and Applications of MXenes: Current Status and Perspectives. *Ceram. Int.* **2019**, *45*, 18167–18188. [\[CrossRef\]](#)
- Song, B.; Shi, C.; Qu, Y.; Liu, H.; Yang, H.; Wu, X.; Liu, X. The Electrical Properties and Charge Transport Mechanism of MXenes. *Chin. Chem. Lett.* **2025**, 110823. [\[CrossRef\]](#)
- Jia, L.; Zhou, S.; Ahmed, A.; Yang, Z.; Liu, S.; Wang, H.; Li, F.; Zhang, M.; Zhang, Y.; Sun, L. Tuning MXene Electrical Conductivity towards Multifunctionality. *Chem. Eng. J.* **2023**, *475*, 146361. [\[CrossRef\]](#)
- Lukatskaya, M.R.; Mashtalir, O.; Ren, C.E.; Dall’Agnese, Y.; Rozier, P.; Taberna, P.L.; Naguib, M.; Simon, P.; Barsoum, M.W.; Gogotsi, Y. Cation Intercalation and High Volumetric Capacitance of Two-Dimensional Titanium Carbide. *Science* **2013**, *341*, 1502–1505. [\[CrossRef\]](#)
- Ghidiu, M.; Lukatskaya, M.R.; Zhao, M.-Q.; Gogotsi, Y.; Barsoum, M.W. Conductive Two-Dimensional Titanium Carbide ‘Clay’ with High Volumetric Capacitance. *Nature* **2014**, *516*, 78–81. [\[CrossRef\]](#)
- Zubietta, L.; Bonert, R. Characterization of Double-Layer Capacitors (DLCs) for Power Electronics Applications. In Proceedings of the Conference Record of 1998 IEEE Industry Applications Conference. Thirty-Third IAS Annual Meeting (Cat. No.98CH36242), St. Louis, MO, USA, 12 October 1998; Volume 2, pp. 1149–1154.
- German, R.; Hammar, A.; Lallemand, R.; Sari, A.; Venet, P. Novel Experimental Identification Method for a Supercapacitor Multipore Model in Order to Monitor the State of Health. *IEEE Trans. Power Electron.* **2016**, *31*, 548–559. [\[CrossRef\]](#)
- Wu, C.-C.; Young, W.-B. On the Fabrication Processes of Structural Supercapacitors by Resin Transfer Molding and Vacuum-Assisted Resin Transfer Molding. *J. Compos. Sci.* **2024**, *8*, 418. [\[CrossRef\]](#)
- Boota, M.; Anasori, B.; Voigt, C.; Zhao, M.-Q.; Barsoum, M.W.; Gogotsi, Y. Pseudocapacitive Electrodes Produced by Oxidant-Free Polymerization of Pyrrole between the Layers of 2D Titanium Carbide (MXene). *Adv. Mater.* **2016**, *28*, 1517–1522. [\[CrossRef\]](#) [\[PubMed\]](#)
- Zhang, J.; Seyedin, S.; Qin, S.; Wang, Z.; Moradi, S.; Yang, F.; Lynch, P.A.; Yang, W.; Liu, J.; Wang, X.; et al. Highly Conductive $\text{Ti}_3\text{C}_2\text{T}_x$ MXene Hybrid Fibers for Flexible and Elastic Fiber-Shaped Supercapacitors. *Small* **2019**, *15*, 1804732. [\[CrossRef\]](#) [\[PubMed\]](#)
- Hu, M.; Chen, L.; Jing, Y.; Zhu, Y.; Dai, J.; Meng, A.; Sun, C.; Jia, J.; Li, Z. Intensifying Electrochemical Activity of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene via Customized Interlayer Structure and Surface Chemistry. *Molecules* **2023**, *28*, 5776. [\[CrossRef\]](#)
- Rozain, C.; Millet, P. Electrochemical Characterization of Polymer Electrolyte Membrane Water Electrolysis Cells. *Electrochim. Acta* **2014**, *131*, 160–167. [\[CrossRef\]](#)
- Melchior, S.A.; Raju, K.; Ike, I.S.; Erasmus, R.M.; Kabongo, G.; Sigalas, I.; Iyuke, S.E.; Ozoemena, K.I. High-Voltage Symmetric Supercapacitor Based on 2D Titanium Carbide (MXene, Ti_2CT_x)/Carbon Nanosphere Composites in a Neutral Aqueous Electrolyte. *J. Electrochem. Soc.* **2018**, *165*, A501. [\[CrossRef\]](#)
- Zhang, X.; Liu, Y.; Dong, S.; Ye, Z.; Wei, Y. Low-Temperature Synthesized Nanocomposites with Amorphous FeOOH on $\text{Ti}_3\text{C}_2\text{T}_x$ for Supercapacitors. *J. Alloys Compd.* **2018**, *744*, 507–515. [\[CrossRef\]](#)
- Gajdosova, V.; Lorencova, L.; Prochazka, M.; Omastova, M.; Micusik, M.; Prochazkova, S.; Kveton, F.; Jerigova, M.; Velic, D.; Kasak, P.; et al. Remarkable Differences in the Voltammetric Response towards Hydrogen Peroxide, Oxygen and $\text{Ru}(\text{NH}_3)_6^{3+}$ of Electrode Interfaces Modified with HF or LiF-HCl Etched $\text{Ti}_3\text{C}_2\text{T}_x$ MXene. *Microchim. Acta* **2019**, *187*, 52. [\[CrossRef\]](#)
- Mainka, J.; Gao, W.; He, N.; Dillet, J.; Lottin, O. A General Equivalent Electrical Circuit Model for the Characterization of MXene/Graphene Oxide Hybrid-Fiber Supercapacitors by Electrochemical Impedance Spectroscopy—Impact of Fiber Length. *Electrochim. Acta* **2022**, *404*, 139740. [\[CrossRef\]](#)
- Shao, H.; Xu, K.; Wu, Y.-C.; Iadecola, A.; Liu, L.; Ma, H.; Qu, L.; Raymundo-Piñero, E.; Zhu, J.; Lin, Z.; et al. Unraveling the Charge Storage Mechanism of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene Electrode in Acidic Electrolyte. *ACS Energy Lett.* **2020**, *5*, 2873–2880. [\[CrossRef\]](#)
- Bo, Z.; Chen, Y.; Yu, Q.; Yan, J.; Cen, K.; Liu, Z. Unveiling the Energy Storage Mechanism of MXenes under Acidic Conditions through Transitions of Surface Functionalizations. *J. Phys. Chem. C* **2024**, *128*, 2352–2361. [\[CrossRef\]](#)

24. Boota, M.; Urbankowski, P.; Porzio, W.; Barba, L.; Osti, N.C.; Bleuel, M.; Keum, J.K.; Mamontov, E. Understanding Functionalization of Titanium Carbide (MXene) with Quinones and Their Pseudocapacitance. *ACS Appl. Energy Mater.* **2020**, *3*, 4127–4133. [\[CrossRef\]](#)
25. Hao Ran Huang, J.; Tseng, S.-W.; Peter Chen, I.-W. In-Situ Electrochemical XRD and Raman Probing of Ion Transport Dynamics in Ionic Liquid-Etched $\text{Ti}_3\text{C}_2\text{T}_x$ MXene for Energy Storage Applications. *Chem. Eng. J.* **2025**, *503*, 158232. [\[CrossRef\]](#)
26. Liu, Z.; Tian, Y.; Yang, J.; Xu, S.; Tian, Q.; Yan, P.; Han, B.; Xu, Q. Ultrafast Ion Transport in 2D Confined MXene for Improved Electrochemical Performance: Boron-Atom-Substituted –OH Termination. *ACS Nano* **2024**, *18*, 32950–32958. [\[CrossRef\]](#)
27. Wang, J.; Chen, J.; Liu, N.; Lei, J.; Gao, H.-W.; Yu, F.; Pan, F.; Ma, J. Wedge-Like Microstructure of $\text{Al}_2\text{O}_3/\text{i-Ti}_3\text{C}_2\text{T}_x$ Electrode with “Nano-Pumping” Effect for Boosting Ion Diffusion and Electrochemical Defluoridation. *Adv. Sci.* **2024**, *12*, 2411659. [\[CrossRef\]](#)
28. Bag, M.; Renna, L.A.; Adhikari, R.Y.; Karak, S.; Liu, F.; Lahti, P.M.; Russell, T.P.; Tuominen, M.T.; Venkataraman, D. Kinetics of Ion Transport in Perovskite Active Layers and Its Implications for Active Layer Stability. *J. Am. Chem. Soc.* **2015**, *137*, 13130–13137. [\[CrossRef\]](#)
29. Aguedo, J.; Lorencova, L.; Barath, M.; Farkas, P.; Tkac, J. Electrochemical Impedance Spectroscopy on 2D Nanomaterial MXene Modified Interfaces: Application as a Characterization and Transducing Tool. *Chemosensors* **2020**, *8*, 127. [\[CrossRef\]](#)
30. Wu, T.; Kent, P.R.C.; Gogotsi, Y.; Jiang, D. How Water Attacks MXene. *Chem. Mater.* **2022**, *34*, 4975–4982. [\[CrossRef\]](#)
31. Laschuk, N.O.; Easton, E.B.; Zenkina, O.V. Reducing the Resistance for the Use of Electrochemical Impedance Spectroscopy Analysis in Materials Chemistry. *RSC Adv.* **2021**, *11*, 27925–27936. [\[CrossRef\]](#)
32. Li, X.; Liu, F.; Huang, D.; Xue, N.; Dang, Y.; Zhang, M.; Zhang, L.; Li, B.; Liu, D.; Wang, L.; et al. Nonoxidized MXene Quantum Dots Prepared by Microexplosion Method for Cancer Catalytic Therapy. *Adv. Funct. Mater.* **2020**, *30*, 2000308. [\[CrossRef\]](#)
33. Lin, H.; Gao, S.; Dai, C.; Chen, Y.; Shi, J. A Two-Dimensional Biodegradable Niobium Carbide (MXene) for Photothermal Tumor Eradication in NIR-I and NIR-II Biowindows. *J. Am. Chem. Soc.* **2017**, *139*, 16235–16247. [\[CrossRef\]](#) [\[PubMed\]](#)
34. Navalpotro, P.; Anderson, M.; Marcilla, R.; Palma, J. Insights into the Energy Storage Mechanism of Hybrid Supercapacitors with Redox Electrolytes by Electrochemical Impedance Spectroscopy. *Electrochim. Acta* **2018**, *263*, 110–117. [\[CrossRef\]](#)
35. Touhami, S.; Mainka, J.; Dillet, J.; Taleb, S.A.H.; Lottin, O. Transmission Line Impedance Models Considering Oxygen Transport Limitations in Polymer Electrolyte Membrane Fuel Cells. *J. Electrochem. Soc.* **2019**, *166*, F1209. [\[CrossRef\]](#)
36. Da Silva, L.M.; Cesar, R.; Moreira, C.M.R.; Santos, J.H.M.; De Souza, L.G.; Pires, B.M.; Vicentini, R.; Nunes, W.; Zanin, H. Reviewing the Fundamentals of Supercapacitors and the Difficulties Involving the Analysis of the Electrochemical Findings Obtained for Porous Electrode Materials. *Energy Storage Mater.* **2020**, *27*, 555–590. [\[CrossRef\]](#)
37. Song, H.-K.; Sung, J.-H.; Jung, Y.-H.; Lee, K.-H.; Dao, L.H.; Kim, M.-H.; Kim, H.-N. Electrochemical Porosimetry. *J. Electrochem. Soc.* **2004**, *151*, E102. [\[CrossRef\]](#)
38. Tian, Y.; Hou, P.; Zhang, H.; Xie, Y.; Chen, G.; Li, Q.; Du, F.; Vojvodic, A.; Wu, J.; Meng, X. Theoretical Insights on Potential-Dependent Oxidation Behaviors and Antioxidant Strategies of MXenes. *Nat. Commun.* **2024**, *15*, 10099. [\[CrossRef\]](#)
39. Tang, J.; Mathis, T.S.; Kurra, N.; Sarycheva, A.; Xiao, X.; Hedhili, M.N.; Jiang, Q.; Alshareef, H.N.; Xu, B.; Pan, F.; et al. Tuning the Electrochemical Performance of Titanium Carbide MXene by Controllable In Situ Anodic Oxidation. *Angew. Chem. Int. Ed.* **2019**, *58*, 17849–17855. [\[CrossRef\]](#)
40. Wang, D.; Wang, L.; Lou, Z.; Zheng, Y.; Wang, K.; Zhao, L.; Han, W.; Jiang, K.; Shen, G. Biomimetic, Biocompatible and Robust Silk Fibroin-MXene Film with Stable 3D Cross-Link Structure for Flexible Pressure Sensors. *Nano Energy* **2020**, *78*, 105252. [\[CrossRef\]](#)
41. Chen, J.; Tong, P.; Huang, L.; Yu, Z.; Tang, D. Ti_3C_2 MXene Nanosheet-Based Capacitance Immunoassay with Tyramine-Enzyme Repeats to Detect Prostate-Specific Antigen on Interdigitated Micro-Comb Electrode. *Electrochim. Acta* **2019**, *319*, 375–381. [\[CrossRef\]](#)
42. Masarapu, C.; Zeng, H.F.; Hung, K.H.; Wei, B. Effect of Temperature on the Capacitance of Carbon Nanotube Supercapacitors. *ACS Nano* **2009**, *3*, 2199–2206. [\[CrossRef\]](#)
43. Wu, Y.; Sundaresan, S.; Balasingam, B. Battery Parameter Analysis through Electrochemical Impedance Spectroscopy at Different State of Charge Levels. *J. Low Power Electron. Appl.* **2023**, *13*. [\[CrossRef\]](#)
44. Bhattarai, J.K.; Tan, Y.H.; Pandey, B.; Fujikawa, K.; Demchenko, A.V.; Stine, K.J. Electrochemical Impedance Spectroscopy Study of Concanavalin A Binding to Self-Assembled Monolayers of Mannosides on Gold Wire Electrodes. *J. Electroanal. Chem.* **2016**, *780*, 311–320. [\[CrossRef\]](#) [\[PubMed\]](#)
45. Wang, J.; Li, X.; Wang, Z.; Guo, H.; Huang, B.; Wang, Z.; Yan, G. Systematic Investigation on Determining Chemical Diffusion Coefficients of Lithium Ion in $\text{Li1} + x\text{VPO4F}$ ($0 \leq x \leq 2$). *J. Solid State Electrochem.* **2015**, *19*, 153–160. [\[CrossRef\]](#)
46. Manjakkal, L.; Djurdjic, E.; Cvejic, K.; Kulawik, J.; Zaraska, K.; Szwagierczak, D. Electrochemical Impedance Spectroscopic Analysis of RuO₂ Based Thick Film pH Sensors. *Electrochim. Acta* **2015**, *168*, 246–255. [\[CrossRef\]](#)
47. Chen, N.; Yao, Y.; Wang, D.; Wei, Y.; Bie, X.; Wang, C.; Chen, G.; Du, F. $\text{LiFe}(\text{MoO}_4)_2$ as a Novel Anode Material for Lithium-Ion Batteries. *ACS Appl. Mater. Interfaces* **2014**, *6*, 10661–10666. [\[CrossRef\]](#)
48. Zhao, S.; Meng, X.; Zhu, K.; Du, F.; Chen, G.; Wei, Y.; Gogotsi, Y.; Gao, Y. Li-Ion Uptake and Increase in Interlayer Spacing of Nb_4C_3 MXene. *Energy Storage Mater.* **2017**, *8*, 42–48. [\[CrossRef\]](#)

49. Lukatskaya, M.R.; Kota, S.; Lin, Z.; Zhao, M.-Q.; Shpigel, N.; Levi, M.D.; Halim, J.; Taberna, P.-L.; Barsoum, M.W.; Simon, P.; et al. Ultra-High-Rate Pseudocapacitive Energy Storage in Two-Dimensional Transition Metal Carbides. *Nat. Energy* **2017**, *2*, 17105. [\[CrossRef\]](#)
50. Ma, C.; Ma, M.-G.; Si, C.; Ji, X.-X.; Wan, P. Flexible MXene-Based Composites for Wearable Devices. *Adv. Funct. Mater.* **2021**, *31*, 2009524. [\[CrossRef\]](#)
51. Dhandapani, E.; Thangarasu, S.; Ramesh, S.; Ramesh, K.; Vasudevan, R.; Duraisamy, N. Recent Development and Prospective of Carbonaceous Material, Conducting Polymer and Their Composite Electrode Materials for Supercapacitor—A Review. *J. Energy Storage* **2022**, *52*, 104937. [\[CrossRef\]](#)
52. Kausar, A.; Ahmad, I.; Lam, T.D. Graphene Footprints in Energy Storage Systems—An Overview. *e-Prime—Adv. Electr. Eng. Electron. Energy* **2023**, *6*, 100361. [\[CrossRef\]](#)
53. Nezakati, T.; Seifalian, A.; Tan, A.; Seifalian, A.M. Conductive Polymers: Opportunities and Challenges in Biomedical Applications. *Chem. Rev.* **2018**, *118*, 6766–6843. [\[CrossRef\]](#)
54. Wei, H.; Dong, J.; Fang, X.; Zheng, W.; Sun, Y.; Qian, Y.; Jiang, Z.; Huang, Y. $\text{Ti}_3\text{C}_2\text{T}_x$ MXene/Polyaniline (PANI) Sandwich Intercalation Structure Composites Constructed for Microwave Absorption. *Compos. Sci. Technol.* **2019**, *169*, 52–59. [\[CrossRef\]](#)

Disclaimer/Publisher’s Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.