

RESEARCH ARTICLE

Synergistic PANI–Functionalized $\text{Ti}_3\text{C}_2\text{T}_x$ MXene Hybrid Nanocomposite for Enhanced Supercapacitor Application

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ABSTRACT: Supercapacitors have arisen as indispensable energy-storage classifications to address the performance disparity between high-energy dense batteries and high-power capacitors, enabling fast charge–discharge competence, long operational lifetimes, and applications such as regenerative braking and hybrid energy systems. Among the diverse range of advanced electrode materials, MXene-derived composites have gained significant consideration owing to their outstanding conductivity, compatibility with redox-active polymers and tunable surface functionalization. In this work, $\text{Ti}_3\text{C}_2\text{T}_x(\text{TMX})$ and $\text{Ti}_3\text{C}_2\text{T}_x$ –polyaniline (TMXP) nanocomposites were obtained through a controlled etching and in-situ oxidative polymerization approach. Structural phase identification and bonding analysis were carried out using XRD and FTIR techniques, and XPS was used to find chemical combination and functional group analysis. Further, the morphology was identified using field FESEM and TEM to elucidate the surface architecture. The electrochemical characteristics were assessed via cyclic voltammetry (CV) analysis. Electrochemical impedance spectroscopy (EIS) by employing 1M KOH electrolyte. As fabricated modified TMX and TMXP electrodes exhibited an improved specific capacitance of 628.5 F/g and 862.8 F/g at 1 A/g. Also, the pristine MXene and TMXP modified electrodes showed a retention of 83% and 92 % over 5000 cycles. Hence, $\text{Ti}_3\text{C}_2\text{T}_x$ integrated PANI nanocomposite can be a suitable candidate for advanced supercapacitors.

Keywords: 2D materials, MXenes, Polyaniline (PANI), Supercapacitors, *In-situ* polymerization

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1. INTRODUCTION

In the recent era, there has been a rapidly escalating demand for efficient and reliable energy storage devices, driven by the widespread deployment of renewable energy systems, the proliferation of portable and wearable electronics, electric vehicles (EVs), and the emergence of smart sensing technologies [1]. These applications require energy storage devices that can deliver rapid charge–discharge competence, enhanced cyclic stability, high power density, and working safety. Consequently, the growth of fast-response and durable

energy storage technologies has become a critical research focus [2]. Among several electrochemical energy storage technologies, namely rechargeable batteries and supercapacitors, supercapacitors have gained significant interest because of their superior power density, rapid charge–discharge potential, long-term cycling stability, and minimal maintenance needs.

These positive attributes make supercapacitors highly suitable for applications that demand rapid energy delivery and long-term operational reliability [3-4]. As a result, supercapacitors play a vital role in the advancement of next-generation energy systems. The electrochemical behavior of supercapacitors primarily depends on the characteristics of their active electrode materials. Two-dimensional MXenes have emerged as promising electrochemical electrode applications owing to their metallic electrical conductivity, layered structure, tuneable surface functionalization and

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mechanical flexibility [5]. These MXenes are classified within the family of transition metal carbides and nitrides and are commonly represented by the formula $\text{M}_{n+1}\text{X}_n\text{T}_x$, where M denotes an early transition metal, X denotes nitrogen and/or carbon, and T_x corresponds to surface functional groups such as $-\text{OH}$, $-\text{F}$, and $-\text{O}$ introduced during the etching process. Generally, MXenes are derived from their MAX phases through the selective removal of the Al layer via a selective etching method [6].

Among the MXene family, $\text{Ti}_3\text{C}_2\text{T}_x$ is the most extensively investigated material for capacitor applications, because of its high intrinsic conductivity, $-\text{OH}$, $-\text{O}$ and $-\text{F}$ surface terminations, and excellent electrochemical activity. Though, despite these advantages, TMX suffers from several inherent limitations, including self-restacking, layer aggregation, and oxidative degradation, which restrict electrolyte ion diffusion, reduce accessible active sites, and ultimately deteriorate electrochemical performance during prolonged cycling [7]. Various modification strategies have been explored to address these drawbacks, such as engineering surface functional groups, regulating interlayer spacing and constructing hybrid architectures with metal oxides, conductive polymers and carbonaceous materials to enhance electrochemical performance, like structural stability, suppress restacking, and improve charge transport kinetics. Among these approaches, the incorporation of conductive polymers, particularly polyaniline (PANI), has established to be highly effective [8].

The incorporation of PANI into pristine $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets not only improves the interlayer spacing of MXene layers but also introduces additional pseudocapacitive redox-active sites, thereby enhancing electrical conductivity and facilitating rapid electron and ion transport. Moreover, PANI offers advantages such as low cost, facile synthesis, and reversible redox behavior, making it an attractive candidate for high-performance supercapacitor electrodes [9]. Earlier investigations have highlighted the improved charge storage capability and conductivity of MXene/ PANI hybrid systems for energy storage applications. Liao *et al.* described a $\text{Ti}_3\text{C}_2\text{T}_x\text{@PANI}$ electrode exhibiting a specific capacitance of 301.0 F/g at a current density of 1 A/g with 90.74% of its initial capacitance after 10,000 charge-discharge cycles [10]. Wang *et al.* fabricated graphene-decorated $\text{Ti}_3\text{C}_2\text{T}_x\text{@PANI}$ modified electrodes, achieving 452 F/g at 1 A/g, and 80.4% stability maintained across 5,000 cycling tests [11]. Similarly, Mohammadi *et al.* demonstrated MXene/PANI hybrid modified electrodes with a specific capacitance of 503 F/g and outstanding cycling stability of 98.3% over 10,000 cycles [12]. Tang *et al.* further reported $\text{Ti}_3\text{C}_2\text{T}_x\text{-PANI}$ fibre-based asymmetric supercapacitors attaining an aerial capacitance of 510 mF/cm^2 [13]. In our previous work, MXene/PANI composites were successfully developed for electrochemical biosensor applications, demonstrating the versatility of this hybrid strategy.

Motivated by these findings, the present study reports the assembly of pristine TMX and PANI incorporated TMX nanocomposite electrodes through a selective in situ etching route, followed by an in situ oxidative polymerization

technique. The electrochemical energy storage efficiency of the TMXP electrode was systematically estimated in 1 M KOH electrolyte. Notably, the synergistic TMXP hybrid electrode resulted in a charge storage capability of 862.8 F/g at 1 A/g along with excellent durability, retaining 92% of its original capacity after extended charge-discharge 5,000 cycles within a potential range of -0.2 to $+0.6$ V, signifying its strong potential for better performance supercapacitor applications.

2. EXPERIMENTAL DETAILS

2.1. Synthesis of Pristine TMX and TMXP

$\text{Ti}_3\text{C}_2\text{T}_x$ MXene was synthesized following our previously reported LiF/HCl etching protocol for the preferential extraction of the Aluminum layers from the Ti_3AlC_2 precursor. The obtained multilayered $\text{Ti}_3\text{C}_2\text{T}_x$ was thoroughly washed and delaminated to yield few-layer MXene nanosheets [14]. Subsequently, polyaniline (PANI) was incorporated into the $\text{Ti}_3\text{C}_2\text{T}_x$ nanosheets in various weight ratios to form $\text{Ti}_3\text{C}_2\text{T}_x\text{/PANI}$ hybrid composites, enabling intimate interfacial contact between the conductive polymer chains and the MXene layers [15-16]. Based on optimized electrochemical behavior, the $\text{Ti}_3\text{C}_2\text{T}_x\text{/PANI}$ nanocomposite with the selected composition was dispersed in deionised water, ultrasonicated to ensure homogeneous distribution, and dried at room temperature for subsequent electrode fabrication (Figure 1)

2.2. Fabrication of the working Electrode

Modified working electrodes were fabricated using nickel foam as the current collector, benefiting from its conductive nature and interconnected porous framework. A homogeneous slurry consisting of 80% of TMX/ TMXP 10wt% conductive carbon and 10 wt% PVDF binder, thoroughly mixed in NMP solvent. The obtained homogenous slurry was evenly coated onto a pre-treated nickel foam collector. The coated electrode was then air-dried at room temperature overnight to eliminate residual solvent and strengthen the mechanical integrity of the coating. The final nickel form-coated mass of the active material was approximately 1.3 mg [17].

3. RESULTS AND DISCUSSION

Figure 2 (a) presents the XRD patterns of parent source Ti_3AlC_2 precursor, pristine $\text{Ti}_3\text{C}_2\text{T}_x$ MXene, PANI, and the $\text{Ti}_3\text{C}_2\text{T}_x\text{/PANI}$ nanocomposite. The Ti_3AlC_2 precursor shows a prominent diffraction peak at $2\theta = 38.77^\circ$, confirming the presence of the (104) plane, which represents the Al atomic layers, along with the (002) reflection at 9.40° . The careful etching of Al layers from the Ti_3AlC_2 MAX phase is intrinsically challenging due to strong metallic bonding;

however, after LiF/HCl etching, the total absence of the (104) peak specifies the effective elimination of the Al layer from the precursor. Simultaneously, the (002) peak shifts from 9.40° to 7.03° , representing a significant increase in the interlayer spacing resulting from surface termination ($-\text{O}$, $-\text{OH}$, $-\text{F}$) and ion intercalation, thereby confirming the active

formation of pristine TMX. Upon integration of PANI, the (002) reflection of TMX further shifts to a lower angle of 5.11° , suggesting additional interlayer expansion due to polymer intercalation and robust interfacial connections between TMX layers and PANI chains.

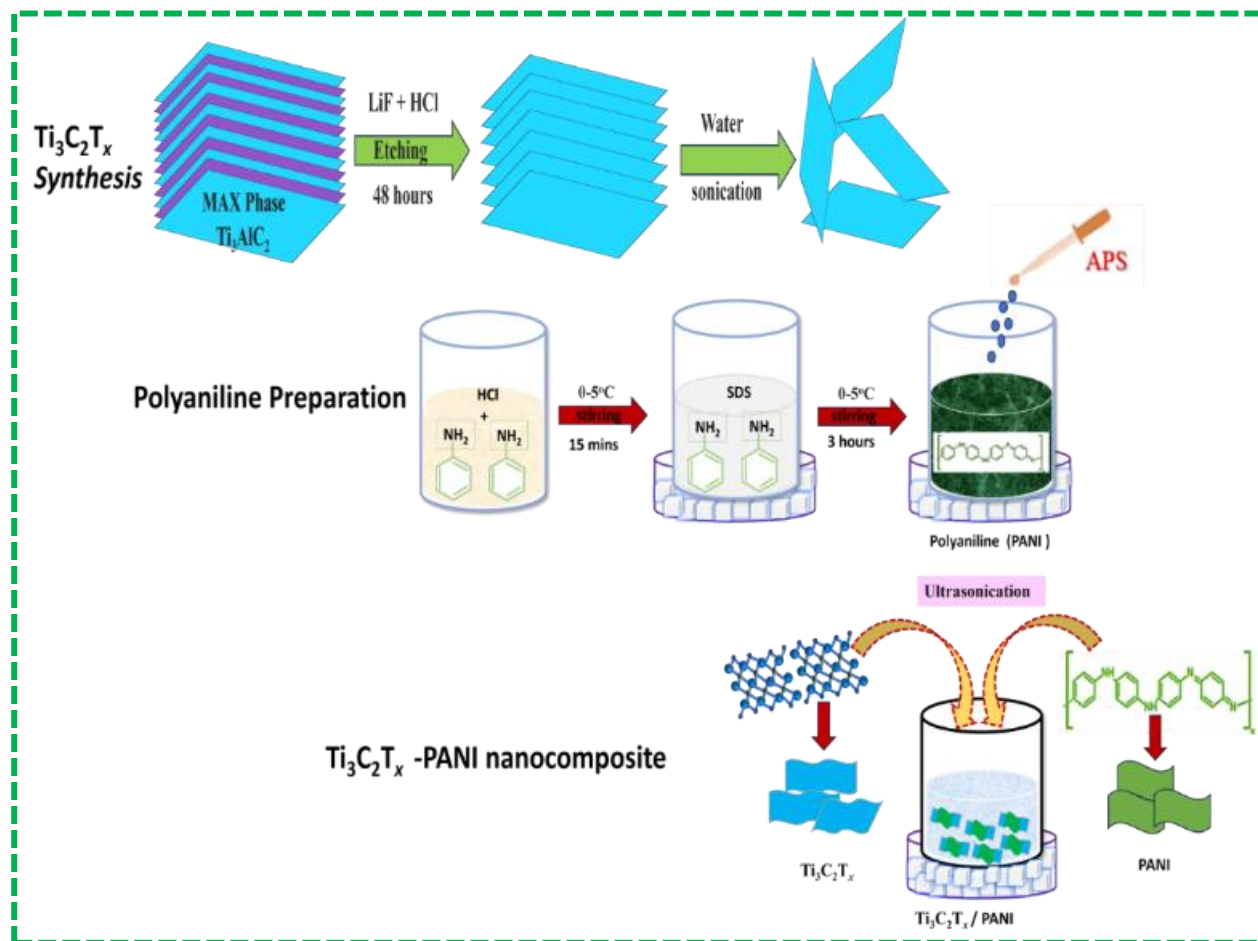


Fig. 1. Illustration of the preparation steps of TMX and TMXP nanocomposite.

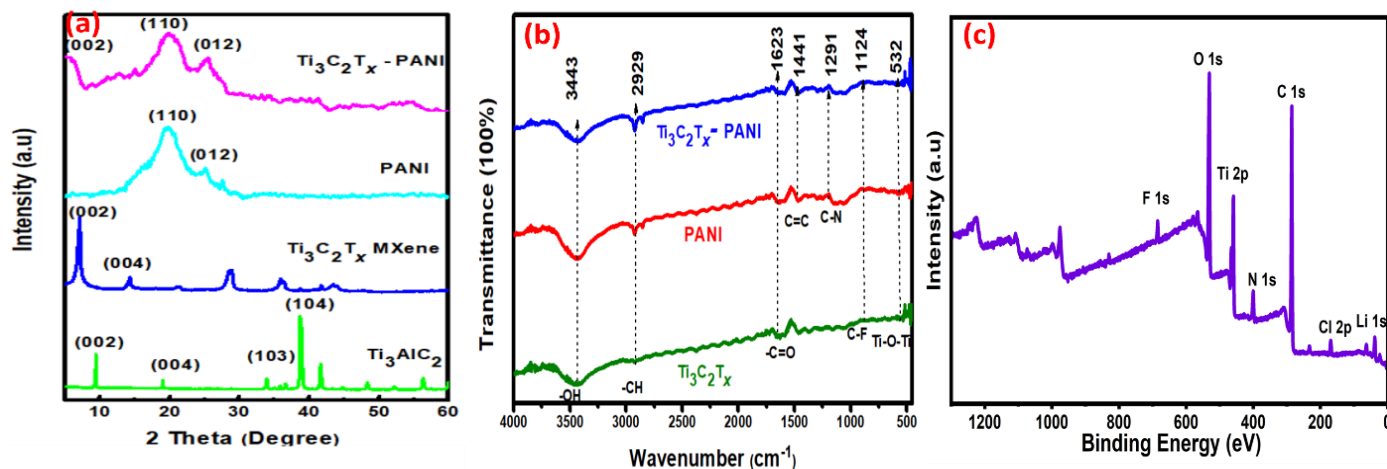


Fig. 2. (a) XRD patterns, (b) FTIR analysis, (c) XPS survey spectra of TMX and TMXP nanocomposite.

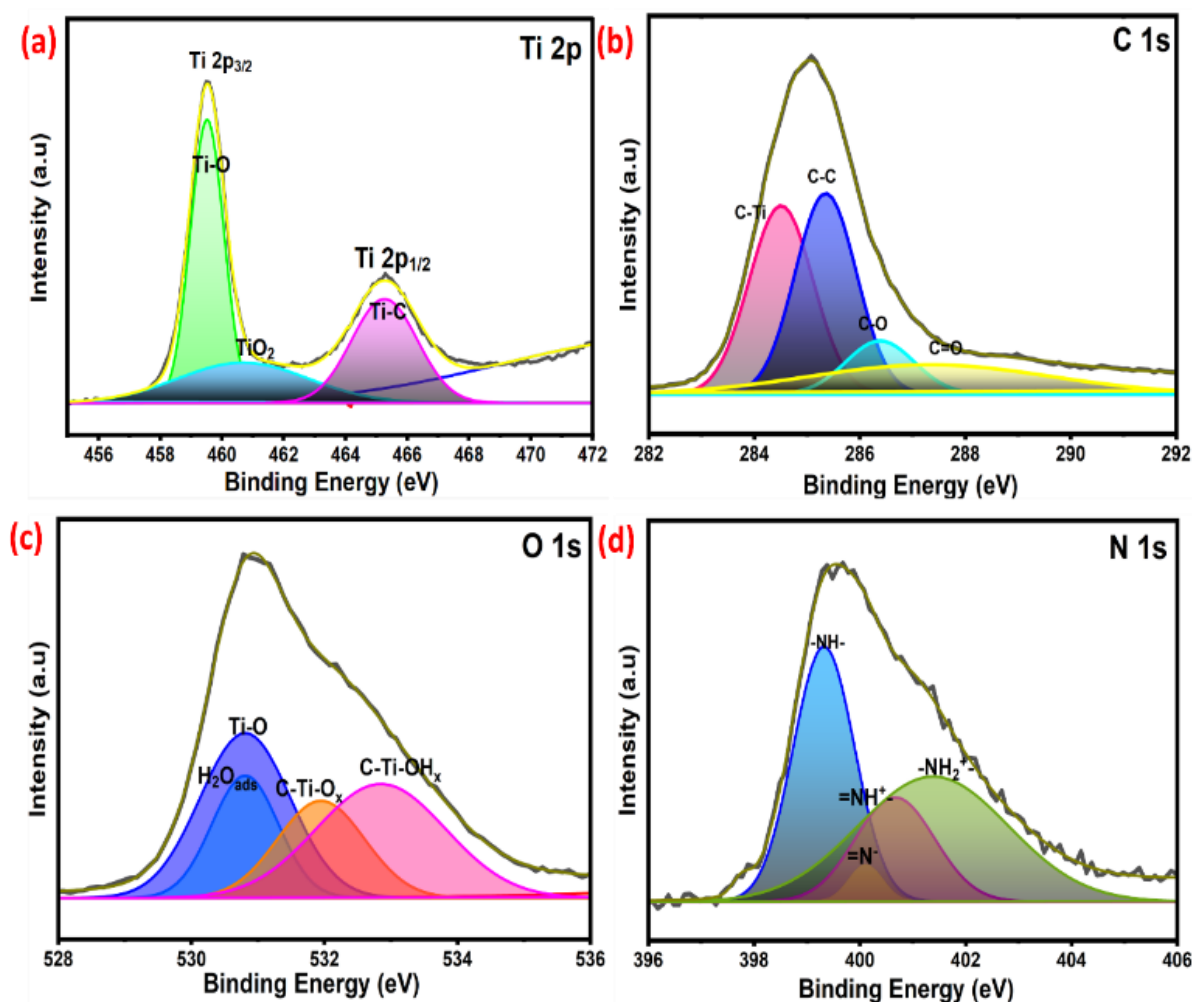


Fig. 3. (a) Ti 2p (b) C 1s (c) O 1s (d) N 1s XPS spectra of TMX and TMXP nanocomposite.

Moreover, the TMXP nanocomposite displays distinct diffraction peaks at 19.55° and 25.50° , equivalent to the (110) and (012) planes of PANI, respectively. The coexistence of MXene and PANI diffraction features confirms the effective development of the PANI incorporated TMX nanostructure [18].

Figure 2 (b) shows the FTIR spectra of TMX, PANI, and PANI-integrated TMX nanocomposite. The peak observed at 3443 cm^{-1} , indexed to $-\text{OH}$ stretching, arising from surface terminations, while the band at 2929 cm^{-1} is attributed to $-\text{CH}$ stretching vibrations. The distinctive vibrational bands of $\text{Ti}_3\text{C}_2\text{T}_x$ observed at 1124 cm^{-1} and 532 cm^{-1} are attributed to $\text{C}-\text{F}$ bending and $\text{Ti}-\text{O}-\text{Ti}$ stretching modes. After the introduction of PANI, additional peaks appear at 1441 cm^{-1} , related to the quinonoid ring ($\text{C}=\text{C}$ stretching), and 1291 cm^{-1} , conforming the benzenoid ring ($\text{C}-\text{N}$ stretching), confirming the effective integration of PANI within the MXene matrix and the development of TMXP nanocomposite through strong interfacial interactions [9, 19]. Figure 2 (c) displays the XPS survey spectrum of the TMXP nanocomposite. The spectrum reveals the existence of F 1s, O 1s, Ti 2p, N 1s, C 1s, Cl 2p and Li 1s 686.2 eV , 530.5 eV , 549.6 eV , 399.5 eV ,

285.5 eV , 167.7 eV , and 37.8 eV . The detection of Ti, O, N, C, F and Cl confirms the MXene framework with surface terminations, while the appearance of the N 1s peak provides a strong indication of successful incorporation of PANI. Residual Cl and Li signals originate from the etching and delamination processes, further corroborating the LiF/HCl synthesis route.

Figure 3 (a) demonstrates the Ti 2p spectrum, which can be deconvoluted into the characteristic Ti $2p_{3/2}$ and Ti $2p_{1/2}$ spin-orbit components located at 459.4 eV and 465.3 eV , respectively, indicative of $\text{Ti}-\text{O}$ bonding arising from surface oxidation and termination groups. Additional contributions observed at $\sim 460.6\text{ eV}$ and $\sim 465.2\text{ eV}$ are ascribed to $\text{Ti}-\text{C}$ bonds within the TMX lattice and minor TiO_2 species, signifying partial surface oxidation of $\text{Ti}_3\text{C}_2\text{T}_x$ during synthesis and composite formation. Figure 3 (b) presents the deconvoluted C 1s spectrum, revealing multiple chemical states of carbon. The bond at 284.5 eV agrees with $\text{C}-\text{Ti}$ bonds, confirming the preservation of the MXene carbide framework. The dominant peak at 285.3 eV is related to $\text{C}-\text{C}/\text{C}=\text{C}$ bonds, originating mainly from the PANI backbone. Peaks located at 286.3 eV and 287.3 eV are responsible for

C–O and C=O bonds, respectively, which result from surface terminations and polymer–MXene interfacial interactions. The N 1s spectrum (Figure 3 (c)) provides a direct indication of effective integration of PANI into the MXene matrix. The peaks at 399.1 eV, 400.0 eV, 400.6 eV, and 401.1 eV are consistent with $-\text{NH}-$ (benzenoid amine), $=\text{N}-$ (quinonoid imine), $=\text{NH}^+-$ (protonated imine), and $-\text{NH}_2^+$ (protonated amine) species, respectively. These nitrogen configurations confirm the presence of both benzenoid and quinonoid structures of PANI and indicate partial protonation, which is beneficial for enhanced electrical conductivity and electrochemical activity. Figure 3 (d) displays the deconvoluted peaks of the O 1s spectrum at 530.7 eV, 530.8 eV, 531.9 eV, and 532.8 eV, corresponding to Ti–O bonds, adsorbed H_2O , C–Ti– O_x , and C–Ti– OH_x surface terminations, respectively. These oxygen-containing functional groups play a crucial role in facilitating ion transport and interfacial charge transfer during electrochemical sensing [20].

Figure 4 (a, b) shows the FESEM morphology of pure $\text{Ti}_3\text{C}_2\text{T}_x$ MXene, revealing a layered and accordion-like morphology with stacked nanosheets at different magnifications, indicative of successful exfoliation after etching. Upon the incorporation of PANI (Figure 4 (c, d)), the MXene layers appear more interconnected and partially covered by polymer chains, suggesting effective intercalation and surface anchoring of PANI within the $\text{Ti}_3\text{C}_2\text{T}_x$ interlayers. The HRTEM images, shown in Figure 4 (e, f), further confirm the nanosheet architecture of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene and the uniform distribution of PANI within the MXene

matrix, demonstrating intimate interfacial contact between the two components. Such a hybrid structure is expected to improve electrical conductivity, increase the electroactive surface area leads to better electrochemical properties.

4. ELECTROCHEMICAL PROPERTIES

The electrochemical capacitive characteristic of pure TMX and the TMXP nanocomposite was systematically investigated in the potential window of 0.02–0.6 V using aqueous KOH electrolyte. Initially, the electrochemical property of pure TMX and TMXP nanocomposite electrode was measured in both 1 M and 2 M KOH electrolytes using cyclic voltammetry (CV) (Figure 5 (a)). Notably, the electrode showed a higher current response and enlarged CV area in 2 M KOH, attributable to its enhanced ionic conductivity. Consequently, 2 M KOH was optimized and employed for subsequent electrochemical evaluations.

Figure 5 (b) compares the cyclic voltagrams of TMX and PANI functionalized TMXP analysed at a scan rate of 0.02 V/s. Upon the incorporation of PANI within the interlayer galleries of TMX, the composite electrode demonstrates a substantially increased current response and a larger integrated CV area, particularly in the 0.2–0.6 V potential range, indicating an enhanced charge storage capability arising from synergistic pseudocapacitive and double-layer contributions.

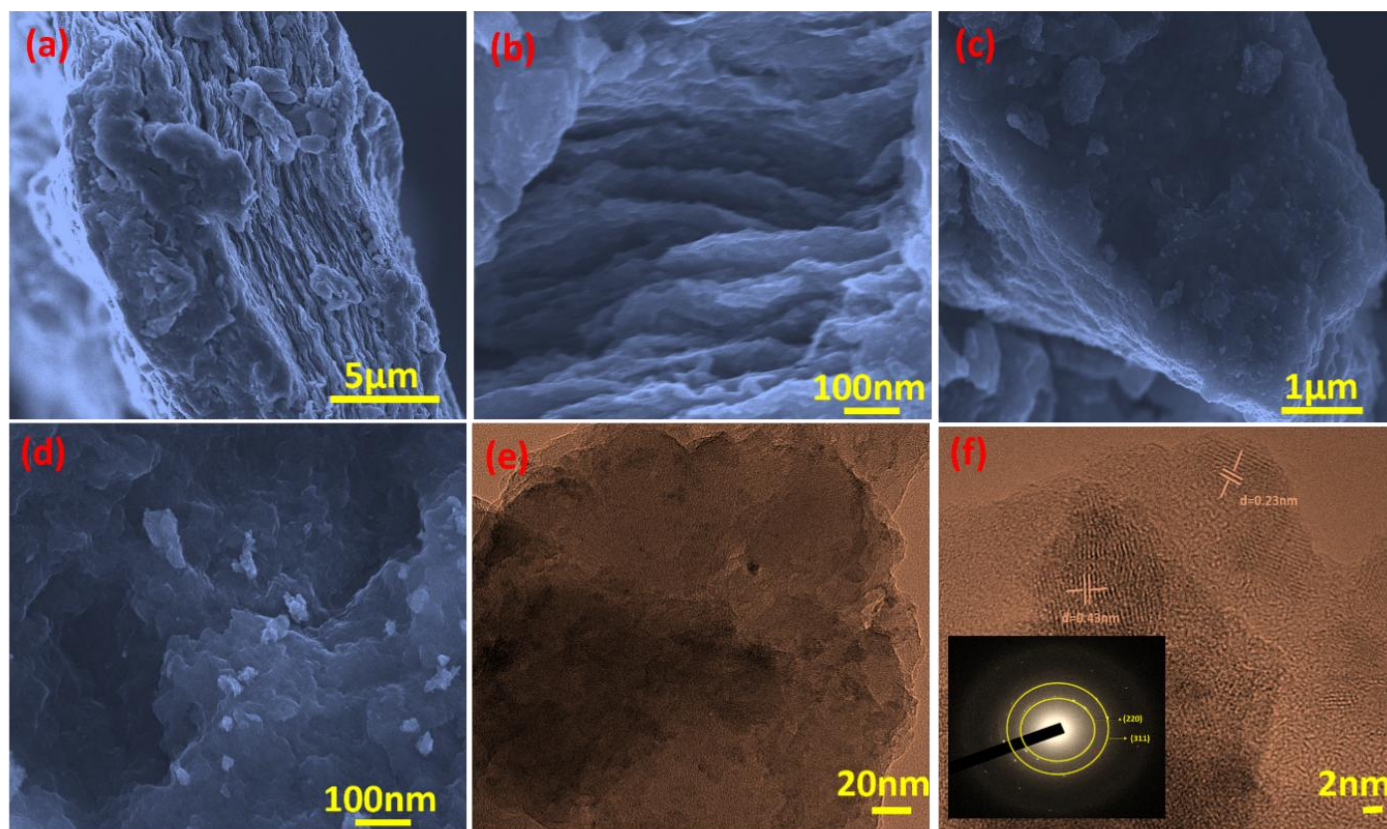


Fig. 4. FESEM images of (a–b) TMX, (c–d) TMXP, (e–f) HRTEM morphology of TMXP.

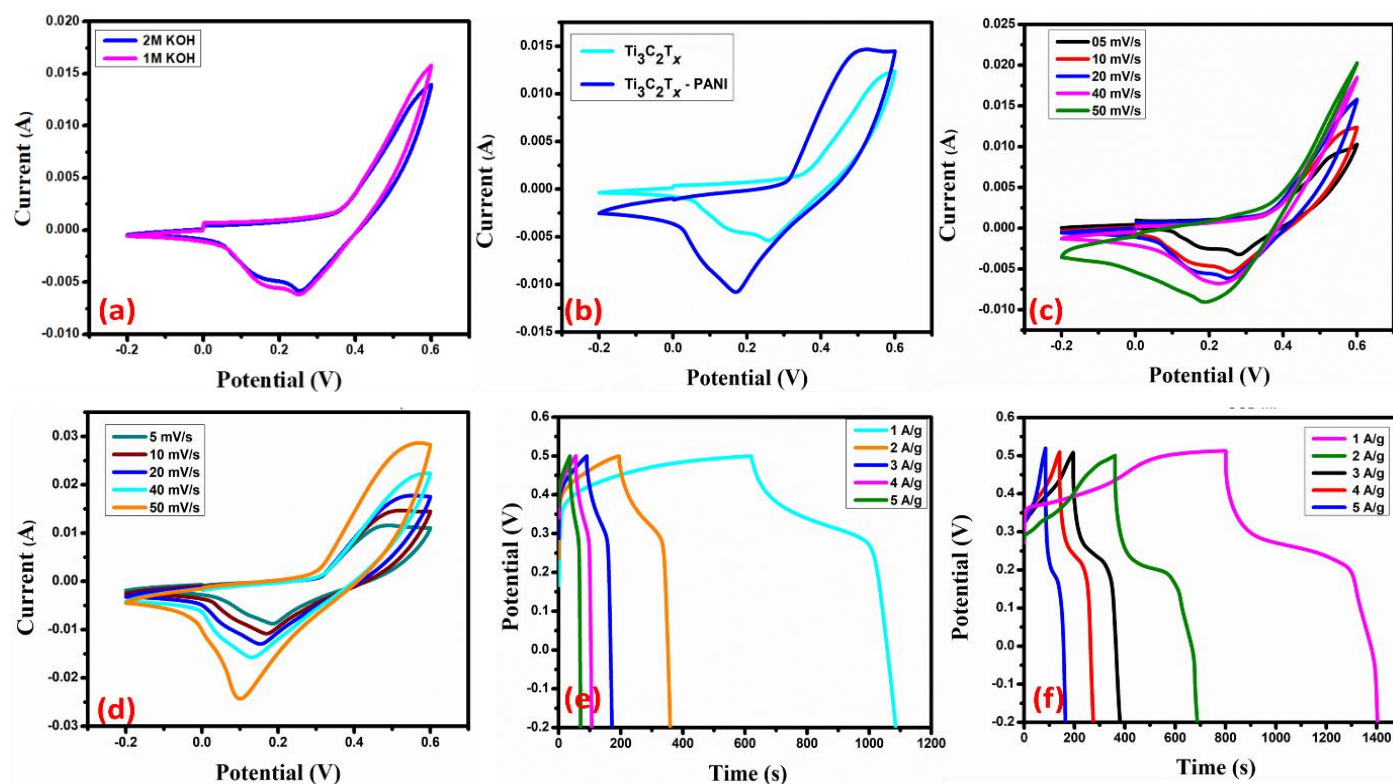


Fig. 5. Cyclic voltammograms of (a) 1 M and 2 M KOH, (b) at 60 mV/s, (c–d) at scan rates of 0.005–0.050 mV/s, of pure TMX and TMXP electrodes and (e–f) GCD curves with varying current densities of TMX and PANI integrated TMX electrodes.

Figure 5 (c, d) presents the cyclic voltammograms of TMX and TMXP electrodes measured at numerous scan rates varying from 0.005 to 0.050 V/s in the presence of 1 M KOH. With enhanced scan rate, a progressive rise in current density and CV area is observed, confirming the excellent electrochemical reversibility and rate capability of both electrodes. However, at higher scan rates, the ion diffusion becomes increasingly surface-dominated, resulting in relatively lower ion adsorption efficiency due to limited electrolyte penetration into the active material. During the anodic scan, PANI undergoes reversible oxidation to PANI^{2+} , while titanium species participate in redox transitions between Ti(II) and Ti(IV), which are reversed during the cathodic sweep. The TMXP nanocomposite delivers a better specific capacitance of 862.8 F/g at 0.001 V/s, significantly superior to that of pristine PANI (626.5 F/g), emphasizing the beneficial contribution of MXene–polymer interfacial interactions.

Figure 5 (e–f) illustrate the galvanic charge and discharge characteristics of TMX and TMXP electrodes at various 1 to 5 A/g current densities. The non-linear nature of the GCD profiles confirms the dominance of faradaic redox processes. At lower current densities, the redox reactions occur more uniformly and under controlled conditions, thereby producing a narrower working potential range. In contrast, at higher current densities, pronounced faradaic contributions necessitate higher operating potentials to sustain rapid charge-transfer kinetics. The calculated specific

capacitance values of 862.8 F/g for TMXP and 628.5 F/g for TMX further validate the higher energy storage performance of the nanocomposite electrode (Table 1). Figure 6 (a) illustrates the dependence of specific capacitance on current density for both pure TMX and TMXP electrodes. It is observed that a slow reduction in specific capacitance occurs with enhancing current density. The decline in the performance at elevated current densities arises from hindered ionic migration and incomplete utilisation of internal active sites due to limited electrolyte accessibility.

In contrast, the redox reactions occur in well controlled electrochemical kinetics, enabling effective application of the electroactive material and resulting in higher capacitance values. Figure 6 (b, c) presents the stability under prolonged cycling conditions of parent TMX and TMXP electrodes, evaluated over 5000 CV cycles at 0.01 V/s scan rate. The pure TMX electrode retains approximately 83% of its initial capacitance after prolonged cycling. Particularly, the TMXP nanocomposite exhibits a significantly improved capacitance retention of ~92%, demonstrating superior cycling durability. This enhanced stability arises from the intercalation of PANI within the TMX interlayers, which increases the interlayer spacing, exposes additional electroactive sites, and facilitates efficient ion diffusion. Moreover, the polymer-modified layered architecture effectively suppresses structural degradation and prevents restacking of TMX sheets in alkaline KOH electrolyte.

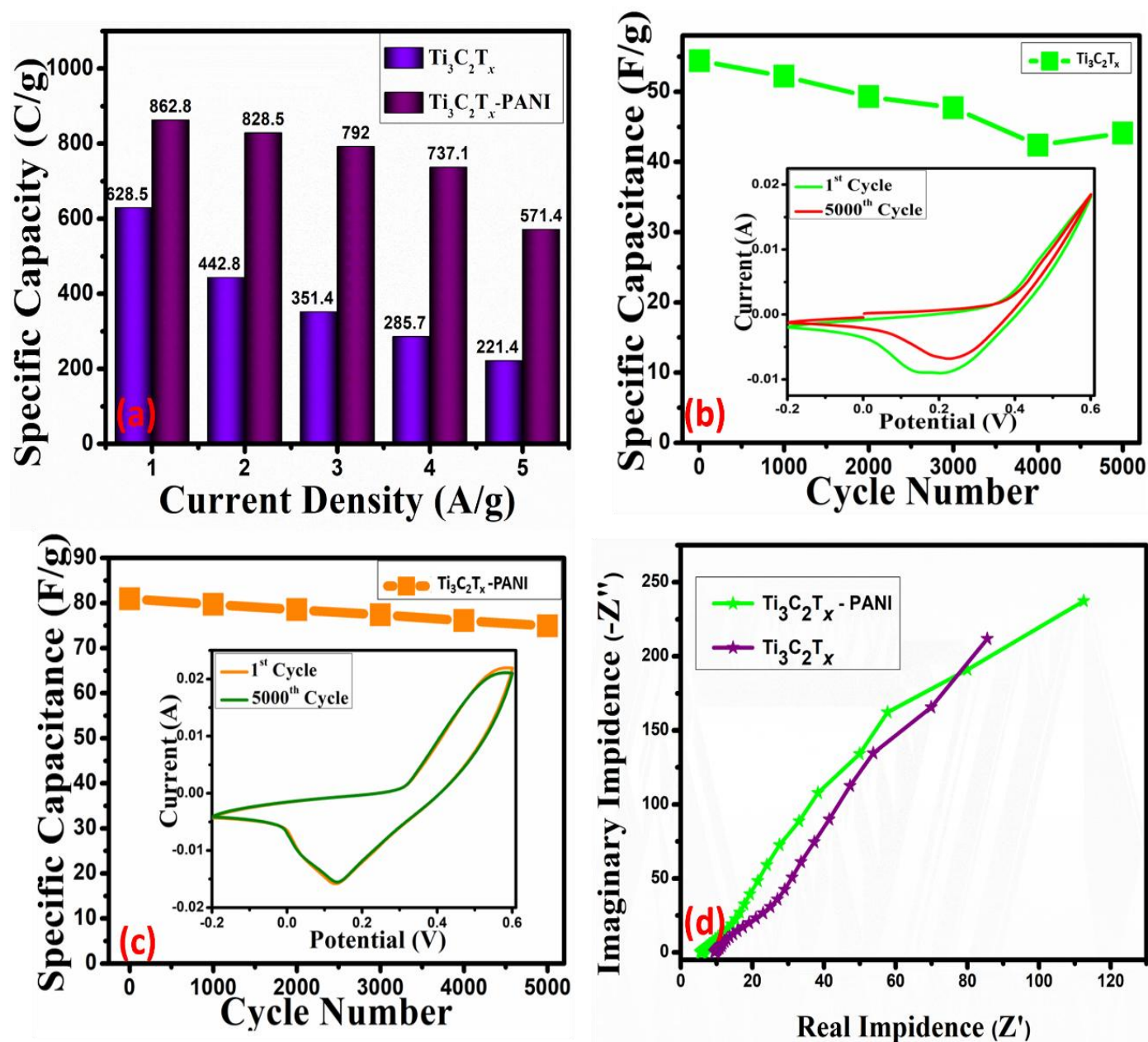


Fig. 6. (a) Bar graph of specific capacitance vs. current density (A/g), (b, c) cycling stability over 5000 CV cycles at 10 mV/s, and (d) EIS Nyquist plots of TMX and TMXP electrodes.

Table 1. Performance comparison of the fabricated electrode with previous reports

S. No	Working Electrode Material	Specific capacitance	Retention /cycles	Ref.
1	$\text{Ti}_3\text{C}_2\text{T}_x\text{@PANI}$	301.0 F/g	90.74% -10000	10
2	graphene-decorated $\text{Ti}_3\text{C}_2\text{T}_x\text{@PANI}$	452 F/g	80.4% - 5000	11
3	MXene/PANI hybrid electrodes	503 F/g	98.3% -10000	12
4	MXene/PANI fiber	510 mF cm^{-2}	94.4%	13
5	MXene/PANI electrode	458.3 Fg^{-1}	95.5%-10000	19
6	$\text{Ti}_3\text{C}_2\text{T}_x$ (TMX)	628.5 F/g	83%-5000	This work
7	$\text{Ti}_3\text{C}_2\text{T}_x\text{/PANI}$ (TMXP)	862.8 F/g	92%-5000	This work

The excellent cycling stability reveals that the TMXP electrode is highly adequate for practical supercapacitor applications requiring long-term operational reliability. The charge transfer kinetics of TMX and TMXP electrodes were studied using electrochemical impedance spectroscopy Figure 6 (d). The Nyquist plot represents a high-frequency semicircle followed by an inclined line at low-frequencies. The smaller semicircle diameter observed for the PANI integrated TMX shows a lower charge-transfer resistance (R_{ct}) compared to parent TMX, reflecting improved interfacial charge transport. These EIS results further confirm the enhanced electrochemical kinetics and accelerated electron/ion transport pathways provided by the synergistic interaction between MXene layers and the conductive PANI network.

5. CONCLUSIONS

In this study, pristine TMX and TMXP nanocomposite-based modified electrodes were successfully prepared through in-situ (LiF/HCl) HF etching and sodium dodecyl sulfate-assisted oxidative polymerization. The phase, chemical, and morphological characteristics of the developed materials were systematically confirmed through XRD, FTIR, XPS, FESEM, and TEM. Electrochemical evaluation revealed that the NF-supported TMX electrode displayed an enhanced specific capacitance of 628.6 F/g at 1 A/g, while the TMXP nanocomposite electrode delivered a specific capacitance of 862.8 F/g at 1 A/g. The improved performance originates from the efficient electron transport provided by the TMX backbone, along with the pseudocapacitive behavior of PANI. Furthermore, the TMXP electrode confirmed superior cycling stability with 92% retention of initial capacitance, compared to 83% for pristine $\text{Ti}_3\text{C}_2\text{T}_x$ after prolonged GCD cycling tests.

DECLARATIONS

Ethical Approval

We affirm that this manuscript is an original work, has not been previously published, and is not currently under consideration for publication in any other journal or conference proceedings. All authors have reviewed and approved the manuscript, and the order of authorship has been mutually agreed upon.

Availability of data and material

The datasets generated and/or analyzed during the current study are available from the corresponding author upon reasonable request.

Conflicts of Interest

The authors declare that they have no financial or personal interests that could have influenced the research and findings presented in this paper. The authors alone are responsible for the content and writing of this article.

Authors' contributions

All authors contributed equally to this work.

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