Structural, Morphological, and Electrochemical Properties of MXene/MoS$_2$-based Supercapacitor

Muhammad Akmal Kosnan$^a$, Mohd Asyadi Azam$^{a,b}$, Rose Farahiyan Munawar$^a$, Alicja Klimkowicz$^b$ and Akito Takasaki$^b$

$^a$Fakulti Teknologi dan Kejuruteraan Industri dan Pembuatan, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia.
$^b$Department of Engineering Science and Mechanics, College of Engineering, Shibaura Institute of Technology, 3-7-5 Toyosu, Koto-Ku, Tokyo 135-8548, Japan.

*Corresponding author. Tel.: +606-270 2571; Fax: +606-270 1047; e-mail: asyadi@utem.edu.my

ABSTRACT

Two-dimensional (2D) materials, inclusive of molybdenum disulfide (MoS$_2$), are auspicious due to their structure providing absorption sites and shorter diffusion paths. However, their sheet restacking affects the functional properties, resulting in the device’s low efficiency. A strategy is proposed to combine MoS$_2$ with MXene material. X-ray Diffraction data revealed peaks at d-spacings of 6.07, 2.72, 2.49, 2.27, 1.82, and 1.53 Å. X-ray photoelectron spectroscopy shows prominent peaks for C 1s and Ti 2p, with no signal for Al detected in the pristine Ti$_3$C$_2$ MXene. This indicates successful etching of the Al layer. Electron Microscopes were used to confirm the samples’ morphology and showed that the hybrid sample has a layered flake-like structure with a small sphere attached to the surface. While the HR-TEM confirmed the layered structure of Ti$_3$C$_2$ after exfoliation from Ti$_3$AlC$_2$, consistent with prior studies. Prior, the electrochemical performances of the MXene/MoS$_2$ supercapacitor in 6M KOH aqueous electrolyte were examined through cyclic voltammetry and galvanostatic charge–discharge. The highest specific capacitance reached was 139.71 Fg$^{-1}$, attributed to heterostructures of an equally distributed MoS$_2$ and MXene. Furthermore, the device retained about 83% of its initial capacitance after 10,000 cycles of cyclic stability testing. The results demonstrated that the MXene material improved the capacitive performance of MoS$_2$ and conversely. Further enhancements can be expected as a result of a major omission in electrode synthesis, particularly the importance of delamination in MXene preparation.

Keywords: MXene, MoS$_2$, hybrid electrode compositions, characterization, supercapacitor, specific capacitance, electrochemical performance

1. INTRODUCTION

A supercapacitor (SC) is an electrochemical energy storage system that has great power density and specific capacitance. Unlike normal capacitors that store energy electrostatically, different SCs rely on various electrical charge storage mechanisms such as electric double-layer capacitance (EDLC), pseudocapacitance, or hybrid mechanisms to store and release energy [1], [2]. Supercapacitors are known for their ability to deliver high power density, but advancements in materials and design have also led to improvements in energy density, allowing supercapacitors to store more energy per unit mass or volume [3]. They are known for their ability to withstand a significantly higher number of charge-discharge cycles compared to normal batteries. This is due to the electrostatic charge storage mechanism in supercapacitors, which involves the physical movement of ions within the electrolyte and between the electrodes, rather than the chemical reactions involved in batteries [4], [5]. Electricity plays a major role in our daily lives, but it is not easy to store it quickly. So, the focus will be on proficient energy storage for effective consumption or utilization in any needed field [6]. Nowadays, industry 4.0 demands portable grid-less energy devices, including supercapacitors. As the reports stated, a supercapacitor is a great energy storage device, that is reusable over many cycles [7]. However, for future development, supercapacitors require an enhancement in the amount of specific capacitance and an even better rate for cycling stability. The supercapacitor electrode material plays a vital part in both requirements to increase the device performance [8], [9].

2D materials are of great interest nowadays. For example, molybdenum disulphide (MoS$_2$) is a bright material because of its structure, providing adsorption sites and short ion diffusion paths. However, restacking among MoS$_2$ sheets showed a reduction of their functional properties and resulted in low efficiency of the device [10-12]. In this study, a strategy to tackle this problem is to design novel heterostructures composed of MoS$_2$ with MXene as a hybrid material [13]. MXene materials are two-dimensional compounds made up of transition metals, carbides, and nitrides that have shown great promise for usage in supercapacitor applications [14]. MXenes are typically synthesised by selectively eliminating the A-group individual, which refers to the elements in the first column of the periodic table, also called alkali metals, from MAX phases. While MAX phases are layered ternary compounds containing carbides and nitrides. This technique yields MXene materials with a distinct layered structure, high specific surface area, outstanding electrical
conductivity, and remarkable mechanical flexibility, which is desirable from the application viewpoint [15]. The appeal of MXene materials for supercapacitor applications stems from their distinct physicochemical properties, which facilitates electrochemical reactions, efficient electrical conductivity, mechanical flexibility, which makes them suitable for flexible and wearable supercapacitor devices, and hydrophilic nature, which allows for easy dispersibility in water and other solvents [16]. MXene materials store electric charge using a novel mechanism that involves electrochemical ion adsorption on their surfaces and subsequent charge transfer. Recent research has mostly focused on creating composite materials for supercapacitor electrodes that incorporate MXenes, as well as carbon nanomaterials, metal oxides, and conductive polymers. For example, Ti₃C₂Tx MXene, a representative member of the developing 2D MXene family, has demonstrated remarkable promise as a supercapacitor electrode material [17]. Comprehensive evaluations of latest advances in Ti₃C₂Tx-based supercapacitor electrodes have underlined the critical role of Ti₃C₂Tx MXene in obtaining exceptional electrochemical performance and improving our understanding of the underlying mechanisms.

2D MXene has good electrical conductivity, and it can host many different cations between its layers [18]. These special properties have attracted attention from various fields. MXene-based materials, such as Titanium Carbide (Ti₃C₂), showed varied chemical properties and structures, offering a competitive performance. However, even though stacking on MXene sheets showed good results, surface oxidation remains a challenge. Hence, the creation of hybrid material is suggested as a solution to compensate for the disadvantage of individual materials and possibly improve properties due to a synergic effect. In this study, the prime objective is to combine Ti₃C₂ MXene and MoS₂ through a straightforward hydrothermal synthesis method, followed by the establishment of a relationship between the structural attributes of MXene/MoS₂ and their performance as supercapacitors. This correlation was investigated through analytical techniques, including FESEM, TEM, XPS, and XRD. The focus of this study is not an exhaustive optimization of the synthesis procedure but rather an exploration of the synergic effect of the composite material. The principal aim was to introduce specific enhancements that were strategically crafted to build upon existing knowledge and address identified limitations without undertaking a complete overhaul of the synthesis process.

2. MATERIALS AND METHODS

2.1. Preparation of Ti₃C₂ MXene and MoS₂

To prepare MXene Ti₃C₂, the MAX phase powders, which in this case is titanium aluminum carbide (Ti₃AlC₂), were slowly submerged in a 40% HF solution, and then was constantly stirred for 24 h at room temperature [19]. Then the resultant was washed many times over with deionized water and centrifuged at 3500 rpm for 5 min until the pH value exceeded 6.0. This process was conducted to remove the aluminum. The final resultant was then dried in the vacuum oven at 50 °C for 12 h.

For MoS₂ preparation, thiourea and ammonium molybdate tetrahydrate, (NH₄)₆Mo₇O₂₄·4H₂O, were solvate in deionized water and stirred vigorously to obtain a homogeneous solution [20]. Then, the mixture was transferred to a Teflon-lined stainless-steel autoclave. The precursor was heated to 210 °C for 40 min and kept for 18 h. Then, the reaction system was naturally cooled to room temperature.

2.2. Preparation of MoS₂/Ti₃C₂ MXene Hybrid Electrode

As-prepared MoS₂ powder was mixed with 0.13 g Ti₃C₂ MXene in the N-methylpyrrolidone (NMP) solution and constantly stirred. The homogeneous resultant was transferred into a Teflon-lined stainless-steel autoclave (28 mL capacity). After that, the experimental procedure is the same to synthesize MoS₂ including drying. The mass of as-synthesized MoS₂/Ti₃C₂ MXene powder was about 0.63g.

A variety of pre-prepared chemicals were readied in advance, including polyvinylidene fluoride (PVDF) binder, acetylene black conductive agent, NMP, and potassium hydroxide (KOH) electrolyte. The slurry was fabricated using a fixed weight ratio set at 80:10:10, allocating 80% to the active material, 10% to the binder, and 10% to the conducting additive. When the active material used was MXene/MoS₂, the individual materials were mixed in equal proportions, as prior research has determined it to be the most optimal ratio. Table 1 below presents the weight ratios for the materials used to create the electrode slurry.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Weight (mg)</th>
<th>Weight composition (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MXene powder</td>
<td>2.72</td>
<td>40</td>
</tr>
<tr>
<td>MoS₂ powder</td>
<td>2.72</td>
<td>40</td>
</tr>
<tr>
<td>PVDF</td>
<td>0.68</td>
<td>10</td>
</tr>
<tr>
<td>Acetylene Black</td>
<td>0.68</td>
<td>10</td>
</tr>
</tbody>
</table>

Once all the samples were prepared, they were mixed and agitated in NMP solvent for a minimum of 5 h to ensure the slurry was well dispersed. The final slurry was achieved after continuous stirring for 2–3 h. Using a conventional slurry coating technique, the slurry was applied onto nickel foam, which served as the current collector. Subsequently, the electrodes were allowed to dry. Additionally, material characterization was done using pristine MXene or MoS₂ or the hybrid and sometimes without conducting additive or acetylene black.
2.3. Materials Characterization (XRD, XPS, FESEM, HR-TEM)

X-ray diffraction (XRD; Rigaku MiniFlex) measurement was carried out by using an X-ray beam with a wavelength of 1.5406 Å. The diffraction angle, denoted as \( \theta \), was from 5° to 90°. XRD is a suitable technique for assessing the crystallinity of the MXene/MoS\(_2\) hybrid material. The XRD database employed in this research is the XPERT-PRO. In this investigation, we aim to evaluate the crystallinity of pristine MoS\(_2\) and the MXene/MoS\(_2\) hybrid electrode. An X-ray photoelectron spectroscopy (XPS) machine (Kratos AXIS Ultra DLD) was employed to assess the surface chemical characteristics of Ti3C\(_2\) MXene.

Morphological analyses were done to delve into the intricate details of material surfaces and internal structures and link them with the properties, functionalities, and prospective applications of the studied materials. The field emission scanning electron microscope (FESEM; JEOL JSM-7600F) was used for general observation. The micro-nano sheet/layers analysis was done by a high-resolution transmission electron microscope (HRTEM; JEOL JEM 2100).

2.4. Coin Cell-Type Supercapacitor Fabrication and Electrochemical Performance Evaluation

The fabricated electrodes were assembled in a coin cell to produce a symmetric supercapacitor. 6M of KOH solution was selected as the electrolyte and polypropylene (PP) as the separator. The machine used to perform cyclic voltammetry (CV) and galvanostatic charge–discharge (GCD) measurements was WonAtech WMPG1000. Electrode loading or average active mass per electrode for this work was 6.20 mg.

For cyclic voltammetry (CV) testing, the scan rates were 1, 10, 50, 100, and 250 mVs\(^{-1}\) at 0.0 to 1.0 V potential window. The achieved CV curves are then discussed and used to calculate the specific gravimetric capacitance (Csp) value. The equation to calculate the Csp is shown below.

\[
C_{sp1} = \frac{A}{mR(V_2 - V_1)}
\]  

(1)

Where \((V_2 - V_1)\) = potential window, \(m\) = average active mass per electrode, \(R\) = scan rates, and \(A\) = area under the curve.

For galvanostatic charge–discharge (GCD) testing, the current applied was set at 1.0, 3.0, 6.0, 9.0, and 15.0 mA from a 0.0–1.0 V potential window, which gave a current density of 0.15, 0.48, 0.96, 1.61, and 2.42 Ag\(^{-1}\), respectively. From the GCD results, the equation to calculate specific gravimetric capacitance \(C_{sp2}\) is shown below.

\[
Specific\; Capacitance, \; C_{sp2} = \frac{l \times \Delta t}{m \times \Delta V} = \frac{l_m(\Delta t)}{\Delta V}
\]  

(2)

where \(l\) = current applied, \(m\) = average active mass of electrode, \(\Delta t\) = time difference, \(l_m\) = current densities, and \(\Delta V\) = voltage difference.

The cyclic stability of the MXene/MoS\(_2\)-based supercapacitor was evaluated by GCD at a relatively high current density of 2.42 Ag\(^{-1}\). To confirm the MXene/MoS\(_2\)-resilience long-term cycle life in the supercapacitor application, capacity retention is crucial. Both techniques provide information on the capacitance of the electrode material. While CV is a relatively quick technique, making it suitable for initial assessments and screening of electrochemical properties, GCD can provide insights into the stability and performance of your device over longer periods, as it simulates the conditions the device would face during practical use. In this study, the \(C_{sp}\) obtained from CV is employed to assess the performance of the devices.

3. RESULTS AND DISCUSSION

3.1. Structural Properties Detected During XRD and XPS Analyses

Figure 1 shows the XRD patterns obtained from pristine MoS\(_2\) and MXene/MoS\(_2\) electrodes. Nevertheless, there are apparent variations in peak heights that correspond to the individual peaks, and fortunately both samples have good crystallinity [21]. The XRD spectrum pattern of an exfoliated pure MoS\(_2\) electrode is shown. The data shows peaks at angles of 14.6°, 33°, 36.1°, 39.7°, 50°, and 60.6°, with corresponding d-spacing of 6.07, 2.72, 2.49, 2.27, 1.82, and 1.53Å. Minor peaks emerge at angles of 14.6, 33, 39.7, and 60.6°, which correspond to the MoS\(_2\) crystal planes (002), (101), (103), and (110), correspondingly; this is consistent with the standard reference. A reduction to a lower intensity of peak at 2θ of 32.9°, 36.1°, and 50° from MoS\(_2\) patterns can be detected by observing the achieved results of peaks of data varying from pure MoS\(_2\) electrode up to MXene/MoS\(_2\) hybrid electrode [22]. This shows the ability of MXene material to unload on the exfoliated MoS\(_2\) electrode. The lack of the Al peak around ~38° indicates the effective removal of the Al layer from the MAX phase material.
MoS$_2$ and Ti$_3$C$_2$ MXene's surface chemical properties were evaluated using XPS; the complete spectra are displayed in Figures 2(a) and (b), respectively. The XPS graph shown in Figure 2(b) shows notable peaks that are associated with Ti 2p and C 1s, but no signal indicative of Al is found in the pure Ti$_3$C$_2$ MXene. This monitoring highlights how the HF acid treatment successfully removed the Al layers and concurrently introduced fluorine and oxygen [23]. The C 1s high-resolution spectra associated with Ti$_3$C$_2$ all arise in the relatively narrow region of 281.9–282.0 eV, regardless of the type of termination present. This prompt derives from C atoms occupy in the Ti octahedra [24]. The Ti 2p spectrum exhibits a dual-peaked structure, as shown in Figure 2(d), roughly around 455 eV and 463 eV, which correspond to Ti 2p3/2 and Ti 2p1/2, respectively. This splitting results from interactions between spin and orbit, and both peaks contain valuable information about the bonding conditions of titanium [25]. Furthermore, Figure 2(e) confirms the formation of MoS$_2$ resolution XPS spectra of Mo 3d. In the Mo 3d spectrum, two prominent peaks are found at 229.5 and 232.6 eV, corresponding to the 3d5/2 and 3d3/2 states of MoS$_2$, respectively. Into the bargain, a peak observed at 226.7 eV can be attributed to the S 2s state of MoS$_2$, while another peak situated at a greater energy level, around 235.8 eV, is associated with Mo6+. This Mo6+ peak signifies a slight oxidation of the Mo edges within MoS$_2$, transitioning from the Mo4+ state to the Mo6+ state [26]. In addition, Figure 2(f) shows the S 2p spectra, wherein two distinct peaks are fitted at 162.31 and 161.5 eV, correlates to the S 2p1/2 and S 2p3/2 states, respectively. These findings provide additional confirmation of the presence of MoS$_2$ on the MXene layers.

Figure 1. XRD spectra of pure MoS$_2$ (black) and MXene/MoS$_2$ electrode (blue).

Figure 2. (a) XPS profiles of the MoS$_2$ samples; (b) XPS profiles of the Ti$_3$C$_2$ samples; (c) C 1s spectra of the Ti$_3$C$_2$ MXene and; (d) Ti2p spectra of Ti$_3$C$_2$ MXene; (e) Mo3d spectra of MoS$_2$ and; (f) S2p spectra of MoS$_2$.

3.2. Morphological Analysis Based on FESEM and HRTEM

The FESEM images shown in Figure 3 (a) and (b) revealed that the Ti$_3$C$_2$ MXene exhibits a layered structure, consistent with its synthesis from the selective etching of aluminium layers from Ti$_3$AlC$_2$. The layered nature is a hallmark feature of MXenes and is evident as stacked sheets or flakes [27]. The surface topography appears relatively smooth, with some observable wrinkles and undulations. These features are attributed to the inherent flexibility and mechanical properties of MXene nanosheets [28]. Figure 3 (d) and (e) depict the morphological characteristics of pure MoS$_2$. These samples showcase the presence of lamellar nanoflower-like MoS$_2$ structures. Lastly, Figure 3 (c) and (f) present images detailing the morphological features of hybrid MXene/MoS$_2$ structures. These images vividly illustrate the attachment of MoS$_2$ to the MXene, which plays an important role in effectively preventing the restacking of individual MoS$_2$ layers. This phenomenon enhances the utility of MXene in optimizing the structural integrity of MoS$_2$, a point that is critical for further comprehensive understanding [29].

HR-TEM was employed to further examine the micro-nano structures of Ti$_3$C$_2$ MXene and MoS$_2$, and the data are presented in Figure 4. From the TEM image of Ti$_3$C$_2$, as shown in Figure 4 (a), it is confirmed that Ti$_3$C$_2$ exhibits a layered structure following the exfoliation of Ti$_3$AlC$_2$.
aligning with previous research findings [30]. The thickness of a single layer of Ti$_3$C$_2$. Notably, the observed $d$-spacing value of the core's lattice plane was approximately 2.44 nm, which was calculated using the Gatan software, closely matching the (310) plane of TiO$_2$, as shown in Figure 4 (c). As XRD characterization did not provide substantial evidence of oxidation, this implies that partial oxidation of Ti$_3$C$_2$ MXene may have took place during the exfoliation process.

As shown in Figure 4 (d), the MoS$_2$ material, as synthesized, exists in the configuration of aggregated layers rather than in an isolated form.

**Figure 3.** (a) FESEM image of Ti$_3$C$_2$ powder after HF treatment and (b) with lower magnification; (c) FESEM image of MXene/MoS$_2$ hybrid material; (d) FESEM image of MoS$_2$ powder and (e) with lower magnification; (f) MoS$_2$ sphere on the MXene/MoS$_2$ hybrid.

**Figure 4.** (a) TEM image of Ti$_3$C$_2$ after HF treatment and (b) high-resolution image (c) TEM image of MoS$_2$ and (d) high-resolution image.
3.3. Electrochemical Measurement

Figure 5 shows the cyclic voltammetry (CV) curves of the MXene/MoS\textsubscript{2} symmetrical supercapacitor at different scan rates. The calculated \( C_{sp} \) values are presented in Figure 5 and tabulated in Table 2. It is clear that there are no redox reaction peaks recorded, which indicates that the electrode exhibited good capacitive behaviour for a symmetric supercapacitor. The figure also shows the relatively not-so-high calculated \( C_{sp} \) value as compared to the literature data is most probably due to the high electrode loading or high active mass on the current collector [31]. Possibly, minimizing the electrode active mass might be a potential solution for achieving a high specific capacitance value. When the electrode loading is minimized, the whole or complete potential of each element inside the electrode may play a crucial role in enhancing the storage ability. Another matter to consider would be the coating technique applied during the preparation of the electrode [32]. Enhancing coating techniques in material synthesis holds significance for bolstering the future performance, durability, and functionality of coated materials.

Table 2 Specific gravimetric capacitance of MXene/MoS\textsubscript{2} hybrid electrode from CV analysis, calculated using (1)

<table>
<thead>
<tr>
<th>Area under curves</th>
<th>Scan rates (mV/s\textsuperscript{-1})</th>
<th>( C_{sp} ) (Fg\textsuperscript{-1})</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.95 x 10\textsuperscript{-3}</td>
<td>1</td>
<td>139.71</td>
</tr>
<tr>
<td>3.03 x 10\textsuperscript{-3}</td>
<td>10</td>
<td>44.59</td>
</tr>
<tr>
<td>7.39 x 10\textsuperscript{-3}</td>
<td>50</td>
<td>21.74</td>
</tr>
<tr>
<td>8.52 x 10\textsuperscript{-3}</td>
<td>100</td>
<td>12.53</td>
</tr>
<tr>
<td>11.00 x 10\textsuperscript{-3}</td>
<td>250</td>
<td>6.47</td>
</tr>
</tbody>
</table>

The supercapacitor exhibited a quasi-rectangular-shaped curve for its CV, also confirming that this hybrid electrode shows good electrochemical double-layer capacitance (EDLC) behaviour [33]. When a lower scan rate was applied, the ion diffusion became slower; thus, the interaction between electrodes and electrolytes could be maximized. Also, the high-concentration electrolyte provides a higher number of ions, and when measured at a lower scan rate, the electrolyte ions have sufficient time to penetrate electrode pores and finally yield a better electrical charge storage for a good capacitance value [34]. Combining MXene and MoS\textsubscript{2} helped in increasing the active area, thus improving the charge–discharge process. Another reason for a lower \( C_{sp} \) is probably the synthesis method not involving the delamination of Ti\textsubscript{3}C\textsubscript{2} and oxidation, which is known to hinder the performance of MXene. On the other hand, higher scan rates allowed more current flow and led to quick ion diffusion, during which electrolyte ions were unable to fully penetrate electrode pores but only on the surface instead [35]. In contrast, when a lower value of scan rates is applied, the capability to capture the details is more accurate; thus, it takes longer to complete testing and yield a more accurate result.

![Figure 5: CV curves of the MXene/MoS\textsubscript{2} hybrid electrode at 1–250 mV/s in 6M KOH electrolyte.](image)

The galvanostatic charge–discharge data are depicted in Figures 6 (a) and (b). The charging and discharging were found to be symmetric at around 77 secs for both charge and discharge. The unequal charge–discharge time indicated that the electrodes in such compositions are not really stabilized [36]. Furthermore, the \( C_{sp} \) values calculated using Equation (2) were very low, because of the high current applied, leading to too rapid a charge–discharge process, and this is common for symmetric supercapacitor. As referred to in Table 3, the \( C_{sp} \) values for 3.0, 6.0, 9.0, and 15.0 mA are not greater than 20 Fg\textsuperscript{-1}. Therefore, instead of studying the insignificant value achieved from the current density of 0.15 to 2.42 Ag\textsuperscript{-1}, the GCD result at 2.42 Ag\textsuperscript{-1} is taken for further discussion. Generally, at 15.0 mA, when a higher current density is applied, increasing the applied current causes the charge–discharge process to happen too rapidly and results in a vast time difference, which leads to very low \( C_{sp} \).

Another possible reason for relatively low capacitance is due to the huge iR drop of approximately ±50% (up to 0.5V), as observed in the majority of the GCD curves (Figure 6). The high resistances owned by the whole supercapacitor system including electrode, electrolyte resistances caused the iR drop, and therefore, the potential value was used to compensate the resistivity. Another possible reason is the oxidation of MXene surfaces. Oxidation can introduce impurities and alter the desired properties of the material [37]. The process of oxidation can lead to the formation of additional functional groups on the electrode surface, which can enhance the electrochemical activity and improve the capacitance performance of the supercapacitor. For example, the initial oxidation of graphene-oxide precursors has been demonstrated to directly correlate with the final supercapacitor performance. It is crucial to prevent raw materials from oxidizing during the synthesis process, for example, to perform material synthesis in an inert gas environment, such as nitrogen (N\textsubscript{2}) or argon (Ar). Nevertheless, these inert gases displace oxygen and moisture, creating a low-oxygen environment, which prevents oxidation.
Table 3: Specific capacitance of MXene/MoS₂ hybrid electrode from GCD analysis

<table>
<thead>
<tr>
<th>Discharging time (sec)</th>
<th>Applied Current (mA)</th>
<th>Current Density (Ag⁻¹)</th>
<th>Specific Capacitance (Fg⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>154.96</td>
<td>1.0</td>
<td>0.15</td>
<td>23.24</td>
</tr>
<tr>
<td>28.10</td>
<td>3.0</td>
<td>0.48</td>
<td>13.49</td>
</tr>
<tr>
<td>14.10</td>
<td>6.0</td>
<td>0.96</td>
<td>13.54</td>
</tr>
<tr>
<td>3.60</td>
<td>9.0</td>
<td>1.61</td>
<td>5.80</td>
</tr>
<tr>
<td>1.90</td>
<td>15.0</td>
<td>2.42</td>
<td>4.56</td>
</tr>
</tbody>
</table>

90.75%, respectively. Measurements were also conducted at current levels of 9.0 mA and 15.0 mA over a span of 1,000 cycles to evaluate the samples’ ability to maintain significant capacitance retention under high current conditions. The findings indicate that, despite a declining trend at 15.0 mA, the samples still achieved a cycle retention of 79.34% after 1,000 cycles, while at 9.0 mA, the retention was 83.34%, as shown in Figure 7. This demonstrates that the samples are capable of enduring high current levels during extensive cycle testing.

In cyclic tests for supercapacitor applications, it is common for the first 100 to 200 cycles to show instability and noise. This may be attributed to the formation of a solid electrolyte interphase (SEI) layer. During the initial charge-discharge cycles, the inauguration of an SEI layer on the electrode-electrolyte interface can cause instability and noise in the voltage profile [38].

After 10,000 cycles of capacitance retention testing, a thorough examination of the cell’s physical and electrochemical characteristics revealed no noticeable changes [39]. It may be due to the sample’s stored energy from the physical separation of charges, avoiding the chemical reactions that occur commonly in batteries during charge and discharge cycles. Chemical reactions can lead to material degradation and reduced performance over time. Figure 8 displays a FESEM picture of the sample with EDX taken both before and after the capacitance retention test. Apart from the potassium element, the EDX values following cycle retention revealed no discernible alterations. Here, the presence of potassium is attributed to the use of 6M KOH as the electrolyte.
Figure 8. FESEM and EDX analyses: samples (a) before and (b) after 10,000 cycles capacitance retention evaluation.

Figure 9 shows the morphology of MXene/MoS₂ before and after 10,000 cycles. There is no significant difference, indicating that the sample can withstand high capacitance retention testing, which is good for supercapacitor applications. Further, the image indicates that the sample has retained its appearance and continues looking like a crumpled piece of paper, symbolizing the freshly crumpled sheet and structural integrity during the retention tests. However, despite those signs of long-term stability and durability, a decrease in sheets' thickness was observed, as expected [40].

The reliable and stable capacitance retention observed after an extensive testing period demonstrates the cell’s strong and lasting performance, making it a great option for applications where long-term and dependable cycle retention is crucial, such as in advanced energy storage systems [41]. The images show a layered structure in line with its synthesis from Ti₃AlC₂ selectively etched aluminium layers visually, thus proving the sample’s ability to withstand the harsh conditions of retention testing, which is a major consideration for the development of high-performance supercapacitors.
4. CONCLUSIONS

The synthesis of MXene/MoS\textsubscript{2} hybrid electrode was accomplished through a hydrothermal process, followed by conventional slurry coating. The XRD patterns confirmed the successful deposition of MXene Ti\textsubscript{3}C\textsubscript{2} onto the exfoliated MoS\textsubscript{2} electrode, as well as proved that this composite heterostructure exhibits a crystalline structure. The element compositions and surface chemical states were confirmed by the XPS analysis. FESEM showed that the Ti\textsubscript{3}C\textsubscript{2} MXene possesses a layered flake-like structure, aligning with its formation through the selective removal of aluminium layers from Ti\textsubscript{3}AlC\textsubscript{2}. Also, HR-TEM confirmed that Ti\textsubscript{3}C\textsubscript{2} exhibits a layered structure following the exfoliation of Ti\textsubscript{3}AlC\textsubscript{2}, which is in good agreement with the literature. The electrochemical performance of the MXene/MoS\textsubscript{2} supercapacitor was evaluated by cyclic voltammetry (CV) and galvanostatic charge-discharge (GCD) tests, with the highest specific capacitance of 139.71 Fg\textsuperscript{-1}. Additionally, the device was subdue to 10,000 cycles of cyclic testing at 6.0 mA applied current, equivalent to 0.96 Ag\textsuperscript{-1}, with a capacitance retention of 82.58% of its initial discharge capacitance. Furthermore, even after 10,000 retention cycles, the cell remained unchanged, indicating its strong cyclic stability. Further optimization of the MXene synthesis process, specifically incorporation of the delamination step, is necessary to unlock the full potential of MXene/MoS\textsubscript{2} hybrid for energy storage applications.

ACKNOWLEDGMENTS

The authors are grateful to Universiti Teknikal Malaysia Melaka and Shibaura Institute of Technology, Japan for the facilities support. Special thanks to the Ministry of Higher Education for financially supporting this research under the Fundamental Research Grant Scheme (FRGS) research grant numbered FRGS/1/2021/TK0/UTEM/01/3/F00476.

REFERENCES


