The Potential of MXene Materials as a Component in the Catalyst Layer for the Oxygen Evolution Reaction

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Abstract

MXenes are a class of 2D/layered materials which are highly conductive, hydrophilic, have a large electrochemical surface area and are easily processible into electrodes for energy applications. Since the discovery of MXenes over ten years ago, these materials have been mainly used in the preparation of electrodes for batteries and supercapacitors. However, due to their aforementioned properties, MXenes could potentially be utilised as a component in the catalyst layer for the Oxygen Evolution Reaction (OER). This opinion piece will discuss some of the recent literature in the area of hybrid catalysts consisting of various Transition Metal Oxides (TMOs) and MXenes for the OER. We will also discuss current drawbacks and future outlook in this new area of research.

Keywords: MXene, Transition Metal Oxide, Oxygen Evolution Reaction
1. Introduction

In an electrolyser device, the catalyst layer can contribute to losses in the overall running of the electrolyser due to inactive OER sites and low conductivity of the materials.[1,2] To achieve next generation inexpensive OER electrolyser catalysts, the catalysts themselves must be electrically conductive, mechanically and chemically stable under operating conditions, exhibit a high electrochemical surface area, and contain a high concentration of active sites for the evolution of O\textsubscript{2}. This has not been achieved to date for Proton exchange Membrane (PEM) and Alkaline Anion Exchange Membrane (AAEM) water electrolysis. One avenue to explore to make a catalyst that possesses all these characteristics would be to essentially combine different materials that exhibit these properties individually and make a ‘super’ catalyst.

MXenes are a family of 2D materials, which are made up of transition metal carbides and nitrides, produced from MAX phases by various etching and delamination processes, Figure 1A. [3,4] A MAX phase has the general formula of M\textsubscript{\textit{n+1}}AX\textsubscript{n} where the M is an early transition metal, the A is an element from group 13 and 14 of the periodic table, and the X represents a carbon or nitrogen.[5] During the etching process, done in a fluoride ion based solution, the element from group 13/14 is removed from the MAX structure causing the carbide layers to become terminated by OH\textsuperscript{−}, O\textsuperscript{−}, Cl\textsuperscript{−} or F\textsuperscript{−} groups which are subsequently called ‘surface groups or edge sites’. [5] The resulting structure is known as a ‘MXene’. [5,6]

MXenes are known to be highly conductive, hydrophilic, and tuneable which are all advantageous properties that could lead to improving pure metal oxides when combined in a catalyst layer for OER. [6] As metal oxide materials lack high conductivity, which adds to the overall losses in an electrolytic cell, the addition of MXene materials could provide a high conductive support network making the hybrid material into a superior OER catalyst. Additionally, the hydrophilic nature of the MXenes will allow for the full coverage of OH\textsuperscript{−} ions on the surface of the catalyst from the electrolyte which should help in the formation of O\textsubscript{2}. Finally, being able to tune the various MXene materials could significantly improve the conductivity and the hydrophilic nature of the MXene, hence further improving the ability to evolve O\textsubscript{2}. However, to date MXenes are not known to contain active sites for the OER, as no MXenes with metals for promoting the OER (e.g. Ni, Ru, Ir, Co, Mn or Fe) have been successfully synthesised (however MXenes containing Mn and Fe have been theoretically reported).[7,8] Interestingly, for the opposite water splitting reaction, the hydrogen evolution reaction (HER), MXene materials have been proven to be promising through computation calculations and experimentally methods.[9-11]

On the other hand, Transition Metal Oxides (TMOs) are an exciting group of materials, that possess various intriguing physical properties that can change depending on the oxidation state of the material and are known to be active OER catalysts, Figure 1B.[12,13] However, these TMO materials exhibit instability and dissolution during operation which renders these materials unsuitable for deployment in large-scale electrolyser devices.[14]

By combining inexpensive, active TMO catalysts with the conductive MXenes, most of the characteristics of the ‘super’ catalysts described previously, i.e. active site density due to the metal oxides and high conductivity due to the MXenes, in theory can be achieved.[15] The
MXene materials may also provide a high surface area network for the TMO materials, similar to what has been attempted with carbon nanotubes (CNT) for OER composite materials, in order to improve electron transfer properties. However, CNT based materials are known to contain metal impurities that enhance the OER (unaware to most) and corrodes under anodic OER potentials therefore are not the ideal composite materials with TMOs for the OER. This high surface area network of a TMO/MXene composite will hopefully improve the operational stability of the catalyst layer due to improved mechanical properties.

Finally, combining MXenes and the TMOs may even result in a lower loading of the TMOs, further lowering the catalyst costs. Hence, the combination of TMOs and MXenes into heterostructured layers or functionalising the MXenes with TMOs is a new and exciting avenue to be explored for the generation of highly active OER catalysts in PEM and AAEM electrolysers.

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**Figure 1.**  
A. MXene fabrication (Reproduced with permission from Zhang et al. [20] Copyright Wiley (2014))  
B. Metal Oxide Suspensions (Reproduced with permission from Browne et al. [21] Copyright Royal Society of Chemistry (2021))  
C. Number of TMO/MXene papers for the OER (includes published literature from Scopus (01/02/2022) terms used ‘Oxygen Evolution Reaction’ ‘MXene’ and ‘metal oxide’. One paper is a review and one paper is for batteries, not OER).

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**2. TMO/MXene OER Catalysts in Literature**

To date only a handful of papers have been published in this area, Figure 1C and Table 1. All of these papers have the same overarching conclusion that the addition of MXenes to transition metal oxides improves the initial OER performance compared to the TMO or the MXene alone.
Figure 2. Linear Sweep Voltammetry Curves in the OER region showing hybrid TMO/MXene materials outperform their TMO and MXene counterparts from research published by A. Lu et al. (Reproduced with permission from Lu et al. [22] Copyright Elsevier (2020)) B. Benchakar et al. (Reproduced with permission from Benchakar et al. [23] Copyright Wiley (2019)) and C. Yu et al. (Reproduced with permission from Yu et al. [24] Copyright Elsevier (2018)).

For example, Lu et al. synthesised a hybrid MXene composite with Co$_3$O$_4$ decorated on Ti$_3$C$_2$Tx MXene flakes by a solvothermal reaction at 150 °C for three hours.[22] The resulting Co$_3$O$_4$/Ti$_3$C$_2$Tx hybrid exhibited an OER overpotential of ~300 mV at a current density of 10 mA cm$^{-2}$ (the current benchmark used in literature when reporting the performance of OER materials) from linear sweep voltammetry measurements, Figure 2A.[22] Under the same OER conditions, the authors reported that a Co$_3$O$_4$ only material reached the same current density at overpotentials of 390 mV, while the Ti$_3$C$_2$Tx can be deemed OER inactive as the current density at an overpotential of ~400 mV is virtually zero.[22] Furthermore, in this study, Lu and co-workers investigated the effect of the ratio of metal oxide:MXene on the OER. The four ratios of metal oxide:MXene prepared were 1:0.1, 1:0.4, 1:1 and 1:10. The results showed that the lowest amount of MXene to metal oxide (i.e. 1:0.1) exhibited the best OER results in terms of overpotential at a current density at 10 mA cm$^{-2}$.

Benchakar and co-workers have observed a similar phenomenon for a Co layered double hydroxide (LDH)/Ti$_3$C$_2$Tx material fabricated by a polyol and solvothermal process.[23] In this particular study, the Co LDH/Ti$_3$C$_2$Tx outperformed the unsupported Co LDH for the OER by 50 mV, Figure 2B.[23] The authors have also reported that the MXene structure can be preserved for oxidation during synthesis and the OER by the well distributed Co LDHs on the surface of the MXene. Interestingly, the authors also reported that this Co LDH/Ti$_3$C$_2$Tx hybrid synthesized by chemical routes outperformed a material which consisted of the Co LDH and the Ti$_3$C$_2$Tx, mechanically mixed. This increase in performance of the chemically synthesized hybrid compared to the mechanically mixed catalyst was hypothesised to be due to the higher charge transfer resistance due to the close proximity of the Co LDHs and the Ti$_3$C$_2$Tx or the lower amount of active sites in the mechanically mixed material.[23]

Additionally, the integration of MXene materials into a composite with bimetallic TMOs has been shown to be advantageous for the OER. Yu et al. reported that the synthesis of an FeNi-LDH/Ti$_3$C$_2$Tx material, fabricated by the co-precipitation of Fe$^{2+}$ and Ni$^{2+}$ from metal salts with already exfoliated MXene flakes under reflux, which also outperformed its individual counterparts under OER conditions, Figure 2C. The authors attributed the superior OER performance of the composite material to the increase in the charge transfer properties from
electrochemical impedance measurements, a shift in the Ni redox peaks to more anodic potential that may induce the OER earlier and an enhancement in the O binding strength of the FeNi LDH due to an electron extraction as a result of the coupling with the MXene. [24]

| TMO            | MXene       | Synthesis method                                      | Initial OER performance (Overpotential at 10 mA cm⁻²) | Experimental reasoning for OER activity from postmortem or in-situ analysis | Ref  |
|----------------|-------------|-------------------------------------------------------|------------------------------------------------------|--------------------------------------------------------------------------|------|
| NiFe₂O₄        | Ti₃C₂Tx     | Hydrothermal                                          | 266 mV                                               | None given                                                               | [25] |
| NiFe           | Ti₃C₂Tₓ/    | Metal oxides were precipitated and MXene/rGO mixed in  | 235 mV                                               | None given                                                               | [26] |
|                | reduced     |                                                       |                                                      |                                                                          |      |
|                | graphene    |                                                       |                                                      |                                                                          |      |
|                | oxide       |                                                       |                                                      |                                                                          |      |
| Co LDH         | Ti₃C₂Tₓ     | Polyol synthesis                                      | Mixed: Over 520 mV                                    | None given                                                               | [23] |
|                |             | Mixed                                                 |                                                      |                                                                          |      |
| Co₂O₄          | Ti₃C₂Tₓ     | Solvothermal                                          | 300 mV                                               | None given                                                               | [22] |
| PtO-PdO NP     | Ti₃C₂Tₓ     | Solution Plasma modification                         | 310 mV                                               | None given                                                               | [27] |
| FeNi LDHs      | Ti₃C₂Tₓ     | ionic hetero-assembly – metal salts and MXene refluxed | 298 mV                                               | None given                                                               | [24] |

3. **Current Drawbacks and Potential Avenues for Success (Outlook)**

It is evident from literature that MXenes do significantly enhance the initial performance of TMO catalysts for the OER. However, MXene materials in a water-based solution are known to be unstable in air, Figure 3A.[28] The edge sites/surface groups of the MXene materials will oxidise first (due to deoxygenated species) to produce TiO₂ which will then lead to the whole flake becoming oxidized and hence decreasing the conductivity of the materials, Figure 3A. This can potentially be a huge problem for any electrochemical energy application including electrolysis.
Unfortunately, the electrochemical instability of hybrid TMO/MXene materials have been already observed in the small amount of literature published in the area to date. For example, Lu et al. multi-cycled their Co$_3$O$_4$/Ti$_3$C$_2$Tx hybrid in the OER region for 2000 times and observed a significant decrease in activity over time, Figure 3B. [22] Furthermore, a FeNi-LDH/Ti$_3$C$_2$Tx material on Ni foam, synthesized by Yu and co-workers, also exhibited a decrease in activity over time during a chronopotentiometry test at 10 mA cm$^{-2}$ for 60 hours, Figure 3C.

The instability of these materials at such low current densities presents a major drawback in the potential for MXenes to be incorporated as a component of an OER catalyst layer. If these hybrid materials are to be employed in large scale electrolysis devices, the materials must remain stable at high current densities of 1 A cm$^{-2}$ and higher. In order to alleviate major instabilities in these hybrid materials, investigations using in-situ or operando measurements in conjunction with electrochemical techniques need to be carried out in order to determine the reasoning for the instability of the materials, which is likely to be related to the degradation of the MXene to TiO$_2$ under the extreme oxidative conditions present during the OER. The controlled synthesis of hybrid materials that can hinder the instability of the MXene component by covering the edge sites with active materials for the OER is another avenue which can be explored. Benchakar and co-workers have reported that their polyol/solvothermal synthesis method did fabricate a TMO/MXene hybrid material that is OER stable by covering the MXene edge sites with Co LDHs, however no stability tests were conducted to determine the long-term stability, which is needed to learn about possible TMO/MXene instabilities if superior such composite materials are to be designed in the future.[23]

A second route which could be undertaken to improve the OER performance and stability of TMO/MXene hybrids is to explore the (continuously expanding) world of MXenes materials. In literature the only MXene which has been utilised to synthesise TMO/MXene materials is Ti$_3$C$_2$Tx. There are numerous MXene materials now available and all exhibit different chemical and physical properties. [3,29,30] These other MXenes may be more stable and/or active for
the OER when compared to the most common MXene, Ti$_3$C$_2$Tx. For example, it is well known that the incorporation/presence of Fe based materials/impurities into metal oxides improves the parent oxide towards the OER.[31] Therefore if the theoretically proposed Fe$_2$CT$_2$ MXene materials could be synthesized,[32] this may lead to an enhancement in the OER activity, while also attaining high conductivity and hydrophilicity of the combined TMO/MXene materials compared to when a non-Fe MXene (e.g. Ti$_3$C$_2$Tx) is present in the hybrid material.

A third route which could be undertaken to improve the performance of the metal oxide/MXene hybrid is to further investigate what ratio of TMO:MXene is optimum for the OER. Lu and co-workers showed that a ratio of 1:0.1 metal TMO:MXene is the best ratio for the OER in their study. However, this 1:0.1 TMO:MXene material contained the lowest ratio of MXene. Therefore, further studies into lower amounts of MXene in the hybrid need to be undertaken to determine if even less MXene is favourable for the OER when combined with metal oxides.

Furthermore, Gogotsi and co-workers have recently reported that MXene materials containing more than one metal can be fabricated with different stoichiometries and can all be tuned in respect to the chemical properties they exhibit.[3] If the chemical properties of the aforementioned mixed metal MXenes can be tuned, this opens up a huge space to investigate the suitability of these TMO/MXene hybrid materials for the OER.

Due to the large volume of TMO/MXene combinations, Density Functional Theory (DFT) will also play a vital role in the screening of the most promising TMO/MXene OER catalysts, as it has done for the HER.[11] DFT calculations, such as free energy and surface Pourbaix diagrams, could be utilised to predict stable surface terminations under OER conditions which could help experimental synthesis of stable TMO/MXene OER catalysts.

Finally, for these hybrid TMO/MXene catalysts to reach a stage of commercialisation, the TMO/MXene materials would need to outperform the current state-of-the-art materials in terms of activity and stability in actual electrolyser devices and not just in a conventional three-electrode cell used in OER studies throughout academia. The reason behind this, is that in a conventional three-electrode cell, it has been shown that OER catalysts do not behave the same as in an electrolyser device.[21,33,34] However, to reach a stage of testing TMO/MXene catalysts in an electrolyser device, first more fundamental issues must be tackled including the oxidation of MXenes under OER potentials, the optimum metal oxide to MXene ratio for OER and improving the overall activity of the hybrid catalysts by synthetic/in-situ characterisation feedback mechanisms.

Declaration of interest

Nothing declared

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