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Nb₂CT_x MXene integrated DyMn₂O₅ composites: tailored particle size and enhanced capacitance for high performance pseudocapacitors†

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Nb₂CT_x, 2D niobium carbide, belongs to the MXene family obtained from Nb₂AlCT_x through selective etching of the Al layer using a non-toxic aqueous KOH solution as an alternative to the hazardous HF etching process. MXene is an emerging material for supercapacitor electrodes due to its distinctive layered structure, large surface area, superior electrical conductivity, and exceptional chemical stability. Owing to the significant energy of the MXene layers, they have a tendency to restack, which limits the effective utilization of the interlayer space for energy storage. Consequently, expanding the interlayer spacing of MXene has become a key research focus to improve its electrochemical performance. Currently, the interlayer spacing of MXene is typically expanded by incorporating nanoparticles. Although numerous bi-metallic transition metal oxides have been explored for this application, only a limited number of rare earth-based bi-metallic oxides have been investigated. Nowadays, the interlayer spacing of MXene is enhanced through the incorporation of nanoparticles. Different bi-metallic transition oxides have been used for this purpose, but very few rare earth-based bi-metallic oxides have been investigated. So in this study, we have synthesized a bi-metallic DyMn₂O₅/Dy₂O₃/MXene nanocomposite using a hydrothermal method. X-ray diffraction (XRD) analysis was conducted to determine the crystalline structure and phase purity of the materials. In addition, Fourier-transform infrared spectroscopy (FTIR) and X-ray photoelectron spectroscopy (XPS) were performed to investigate the functional groups, chemical bonding and multiple oxidation states in the composites, respectively, providing further confirmation of successful synthesis. Scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDX) was employed to analyze the surface morphology and confirm the cubic-shaped pellet-like nanoparticles on the MXene layers, as well as to verify the elemental composition and uniform distribution of constituent elements. The optimized electrode material demonstrates a marvelous specific capacity of 362.92 C g⁻¹ at a current density of 1 A g⁻¹, along with excellent cycling stability. An asymmetric supercapacitor is fabricated using the DyMn₂O₅/Dy₂O₃/MXene nanocomposite as the anode and activated carbon as the cathode. The assembled device achieves an energy density of 46.25 W h kg⁻¹ at a power density of 705 W kg⁻¹, retaining 70.56% of its initial capacitance after 10 000 charge discharge cycles at 2 A g⁻¹. This study introduces an efficient hydrothermal method for the synthesis of an innovative rare earth and MXene-based nanocomposite with controlled particle size for the electrode of supercapacitors.

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1. Introduction

Overpopulation, excessive consumption of renewable energy sources, industrial expansion, and the rapid depletion of fossil fuels have raised significant concerns regarding the global energy crisis. Energy sources that are effective, green, clean, affordable, sustainable and renewable are urgently needed to replace traditional energy sources in order to address these issues. Although there are plenty of natural resources,

including wind, solar power, thermal, tidal, and hydroelectric, the scientific community still has a difficult time in storing and utilizing them.^{1–4} As a result, the need for advanced energy storage and conversion technologies has significantly increased. Many advanced energy storage devices, capacitors, batteries, fuel cells, sensors and super capacitors that store energy electrically and chemically are the major solutions to mitigate energy challenges.^{5–9} Batteries and supercapacitors¹⁰ are the most reliable and practical methods of electrochemical energy storage at the interface between a conducting electrode and an electrolyte.^{11–15} Supercapacitors have attracted the interest of battery makers due to their ability to reduce the gap between power density and energy density. High energy density while maintaining higher power density are both essential in the field of energy storage and transmission. On the basis of the types of electrode materials and electrochemical process, supercapacitors are commonly divided into two types, electric double layer capacitors (EDLC) and pseudocapacitors.¹⁶ Carbon or activated carbon-based materials are used as electrode materials in the first type and metal oxides/conducting polymers in the second type of supercapacitor.^{17,18} However, a significant effort is being made by researchers to find suitable pseudocapacitive materials and methods of production in order to build such storage units. The optimal electrode material for pseudocapacitors (PCs) with superior power and energy densities must satisfy the following stringent criteria: (i) exceptional electrical conductivity, an extensive specific surface area, and enhanced ion transport pathways to facilitate efficient charge storage and rapid electrochemical response; (ii) abundant redox-active sites coupled with ultrafast charge-transfer kinetics to enable high-rate electrochemical reactions and maximize energy storage efficiency.^{19–21} Transition metal oxides (TMOs) exhibit exceptional electrochemical properties, characterized by high theoretical capacities and versatile redox activity, making them highly suitable for energy storage applications. However, their practical deployment is constrained by inherent drawbacks such as limited electrical conduction, slow ion diffusion, and structural degradation during cycling. To mitigate these limitations, integrating TMOs with carbon-based materials such as graphene, carbon nanotubes, or activated carbon or novel MXene carbides—has proven to be a highly effective strategy. Sometimes, one metal in binary TMOs can be replaced by a rare earth metal because rare earth metals such as La, Dy, Er, and Ce have multiple valence states, and a high value of conductivity. These conductive matrices enhance charge transport, reduce internal resistance, and provide mechanical stability, thereby counteracting volume fluctuations during charge–discharge processes. This synergistic combination significantly improves electrochemical performance by enhancing specific capacity, rate capability, and cycling stability.^{22,23} Composites based on rare earth metal oxides are an example that satisfy those requirements and may be utilized to develop electrode materials with a high energy and power density, where the various valence states of the rare earth metals may be fully used for increased charge storage capacity together with a maximum

operating voltage.²⁴ The two primary chemical processes that rare earth compounds undergo are chemical redox reactions and acid base reactions. Rare earth-based metal oxides are thought to be possible pseudocapacitors because of their redox characteristics and preferred conductivity. In addition, they possess maximum bulk density, which is thought to be particularly beneficial in producing outstanding volumetric capacitance.^{25–30} There are a few reports of rare earth metal-based oxides functioning as the electrode of supercapacitors. The specific gravimetric capacitance of rare earth compounds is quite low, ranging from 200 to 300 F g^{–1}, compared to other transition metal oxides including nickel oxides, cobalt oxides, and manganese oxides. However, due to their significantly higher density, rare earth compounds have a very high volumetric capacitance.^{31–33} As examples, LaMnO₃,³⁴ SrMnO₃,³⁵ Y₂NiMnO₆,³⁶ and LaCrO₃³⁷ electrodes exhibit pseudocapacitance behavior with efficient specific capacitance and remarkable cycling stability. Among these, dysprosium-based manganese oxides (DyMn₂O₅) offer intriguing properties due to the synergistic combination of manganese's rich redox chemistry and dysprosium's ability to stabilize the crystal structure and enhance electrical conductivity. While DyMn₂O₅ has been studied for its magnetic and structural properties, its electrochemical behavior remains largely unexplored. Pseudocapacitance is produced by the oxygen-containing ions in the electrolyte by manganese oxidation/reduction and direct absorption into the voids of the perovskite structure. Manganese-based metal oxides were shown to be a good substitute because to their favourable electrochemical properties, natural availability, affordability, strong redox activity, eco-friendliness, & substantial potential capacitance.^{38–40} Also, to further enhance the conductivity and cycling performance of DyMn₂O₅, MXene and other carbon-based materials can be added. MXenes have emerged as highly promising materials in energy storage research due to their advanced two-dimensional framework, rich surface chemistry, excellent mechanical properties, and exceptional electrical conductivity. Their general chemical composition is represented as M_{n+1}X_nT_x where n denotes integer values (e.g., 1, 2, 3), and T corresponds to surface functional groups such as –OH, –O, and –F. Extensive investigations have led to the discovery of more than 20 MXene variants, including VC₄, Ti₂C, Nb₂C, and Nb₄C₃, among others.^{41–43} These different types of MXene composite with metal oxides are used in energy storage devices and electrochemical sensors also. N. Prabhakar *et al.* studied the Nb₄C₃T_x @WO composite for a high performance asymmetric supercapacitor, delivering a specific capacitance of 1045 F g^{–1} at 1 A g¹.⁴⁴ Furthermore, P. Varatharajan *et al.* fabricated a sensor system with remarkable selectivity, stability and excellent reproducibility for the sensing of *p*-nitrotoluene using MnCo₂O₄ @ Ti₃C₂T_x.⁴⁵ U. Rajaji *et al.* used MoS₂/S-Ti₃C₂/LGE as an electrochemical sensing medium for the detection of poisonous roxarsone and aristolochic acid.⁴⁶ Similarly, MXene base composites with metal oxides e.g. Dy-WO₃/PCNFs, δ -MnO₂@Ti₃C₂T, MnO₂/MXene, CoCr₂O₄@MXene, ZrO₂-doped V₂CT_x MXene, cobalt ion-doped V₂CT_x MXene, Ni-Mn(OH)₂@V₂CT_x

$\text{Mn}_3\text{O}_4@\text{MXene}$, and $\text{Ti}_3\text{C}_4@\text{Mn}_3\text{O}_4@\text{carbonized iron}$, are used as electrode materials of supercapacitors.^{47–54} Structurally, MXenes comprise alternating layers of transition metals and carbon, imparting high electrical conductivity. Their characteristic of an accordion-like morphology facilitates the reversible intercalation of ions and molecules, effectively increasing the interlayer spacing and specific surface area, making them ideal candidates for electrochemical energy storage systems. Among various MXene compositions, Nb_2CT_x has been extensively studied due to its layered architecture, enabling its application as an electrode material with outstanding electrochemical properties.⁵⁵ However, strong interlayer van der Waals forces lead to significant stacking, reducing the accessible surface area and limiting charge storage efficiency. Consequently, expanding the interlayer spacing and preventing restacking are critical research priorities to enhance MXene's performance in energy storage applications.⁵⁶

Multiple literature have been reported on the preparation of DyMn_2O_5 that have looked at their magnetic characteristics and crystal structure.⁵⁷ Despite extensive studies on individual pseudocapacitive materials, there is a critical gap in the research regarding the integration of rare earth metal oxides with MXenes for electrochemical energy storage. Based on the available literature, there are no reported studies on the synthesis and electrochemical investigation of $\text{DyMn}_2\text{O}_5/\text{MXene}$ (Nb_2CT_x) composites. The rational design of these composites leverages the high redox activity of DyMn_2O_5 and the exceptional electrical conductivity of Nb_2CT_x MXene, addressing the limitations of both materials. The synergistic effect of these components is expected to enhance the charge transfer kinetics, improve structural stability, and increase overall electrochemical performance.

The selection of DyMn_2O_5 and Dy_2O_3 among rare earth oxides was based on their synergistic electrochemical and structural characteristics, which are particularly advantageous for energy storage systems. DyMn_2O_5 , a mixed metal oxide containing transition and rare earth metals, provides multiple accessible oxidation states of manganese, enabling fast and reversible faradaic redox reactions. Dysprosium within this compound also contributes to improved electronic conductivity and stability through its 4f orbitals.⁵⁸ Moreover, they possess high bulk density, which significantly contributes to achieving

superior specific capacitance. To optimize their combined performance, various compositions were systematically investigated by adjusting the weight or molar ratios.

In this study, $\text{DyMn}_2\text{O}_5/\text{MXene}$ (Nb_2CT_x) composites were synthesized using a hydrothermal method and evaluated as electrode materials for supercapacitor applications. The incorporation of Nb_2CT_x MXene is anticipated to enhance the conductivity and electrochemical stability of DyMn_2O_5 , leading to improved power and energy density. This work not only introduces a novel electrode material, but also contributes to the broader field of MXene-based hybrid materials for advanced energy storage technologies.

2. Experimental procedure

2.1. Materials

Dysprosium(III) nitrate ($\text{Dy}(\text{NO}_3)_3$, 99% purity), manganese(II) nitrate ($\text{Mn}(\text{NO}_3)_2$, 99% purity), potassium hydroxide (KOH, 98% purity) and MAX (Nb_2AlC , 99% purity) all were obtained from Sigma Aldrich. Deionized water and ethanol (99.9% purity) and no other procedures were required to clean the materials, because all materials used in the experiment were analytical quality.

2.2. Synthesis of MXene (Nb_2CT_x)

Initially, 0.2 g of Nb_2AlCT_x (MAX phase powder) was dispersed in 1 mL of distilled water in a beaker. In a separate beaker, 2 g of potassium hydroxide (KOH) was dissolved in 2 mL of distilled water under stirring. The prepared KOH solution was then gradually added to the Nb_2AlCT_x dispersion while maintaining continuous stirring. The resulting mixture was transferred to a mortar and pestle and finely ground for 1 hour until a thick paste was formed. This paste was then placed into a stainless steel autoclave and subjected to thermal treatment at 150 °C for 24 hours. After the heating process, the autoclave was removed, and the obtained precipitates were collected and thoroughly washed with ethanol and distilled water multiple times to eliminate any residual impurities. The final product was dried in a vacuum oven at 80 °C for 24 hours, yielding a black colored Nb_2CT_x MXene powder, as shown in Fig. 1.

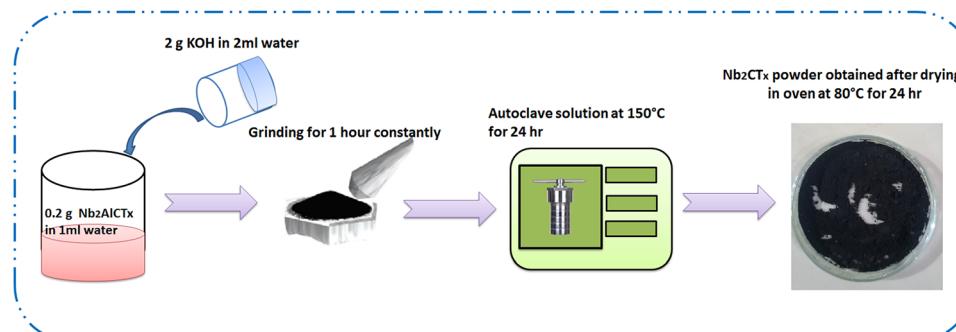


Fig. 1 Schematic illustration of the synthesis of Nb_2CT_x MXene from $\text{Nb}_2\text{AlC}_2\text{T}_x$.

2.3. Preparation of DyMn_2O_5 $\text{Dy}_2\text{O}_3/\text{MXene}$ (Nb_2CT_x) and $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$ composites

$\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$ (Nb_2CT_x) was synthesized *via* a hydrothermal method. Initially, 1 mmol of dysprosium nitrate ($\text{Dy}(\text{NO}_3)_3$) and 2 mmol of manganese nitrate ($\text{Mn}(\text{NO}_3)_2$) were dissolved in 30 mL of distilled water under magnetic stirring for 1 hour. Here, 0.1 g of synthesized MXene (Nb_2CT_x) in 5 mL distilled water was added dropwise into the precursor solution under constant stirring. Separately, 2 g of potassium hydroxide (KOH) was dissolved in 20 mL of distilled water, and the resulting solution was gradually added dropwise to the stirred precursor solution until the pH was adjusted to 7–8. The obtained solution was then transferred into a Teflon-lined stainless steel autoclave and subjected to thermal treatment at 180 °C for 12 hours to facilitate solvent evaporation. When the solution in the autoclave was cooled down at room temperature, it was washed multiple times with ethanol and distilled water, and subsequently subjected to centrifugation five times at 4000 rpm for 20 minutes to obtain pure precipitates. The collected precipitates were dried in an oven at 80 °C for 12 h. Finally, a light yellowish powder was obtained and annealed at 750 °C for 6 hours in a furnace to enhance its structural and functional properties. A schematic diagram of the synthesis technique for the preparation of the $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$ (DMO/DO/MX) composite is shown in Fig. 2.

For comparison, the $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$ (DMO/DO) composite was synthesized by following the same amount of precursors, and the same synthesis protocols as mentioned above without adding MXene (Nb_2CT_x) in solution.

2.4. Preparation of the electrode

A three-electrode configuration was employed for electrochemical measurements. The system consisted of a reference electrode (Ag/AgCl), a counter electrode (platinum wire), and a working electrode composed of nickel foam coated with $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$ (Nb_2CT_x). The fabrication of the working electrode involved grinding of 0.1 g of $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$ (DMO/DO/MX) powder in a mortar and pestle for approximately one hour to achieve a fine particle dispersion. A few drops of water were then added to form a homogeneous aqueous slurry. The prepared slurry was uniformly applied onto a 1 cm × 1 cm nickel foam substrate, followed by drying in an oven at 75 °C for 12 hours. Using the same procedure, a $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$ (DMO/DO) electrode was fabricated. The active loaded mass of $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$ and $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$ was 2.5 mg and 2.8 mg, respectively.

3. Results and discussion

3.1. Structural analysis

3.1.1. XRD analysis. The crystal structure and phase purity of the synthesized samples were analyzed using X-ray diffraction (XRD). Fig. 3 presents the diffraction patterns that were indexed using X'Pert Highscore software. Fig. 3(a) presents the XRD pattern of the synthesized Nb_2CT_x MXene, where the characteristic diffraction peaks at $2\theta = 33.4^\circ$, 36.4° , 38.2° , 59.6° , and 72.7° correspond to the (100), (002), (101), (110), and (201) planes, respectively, as indexed to the

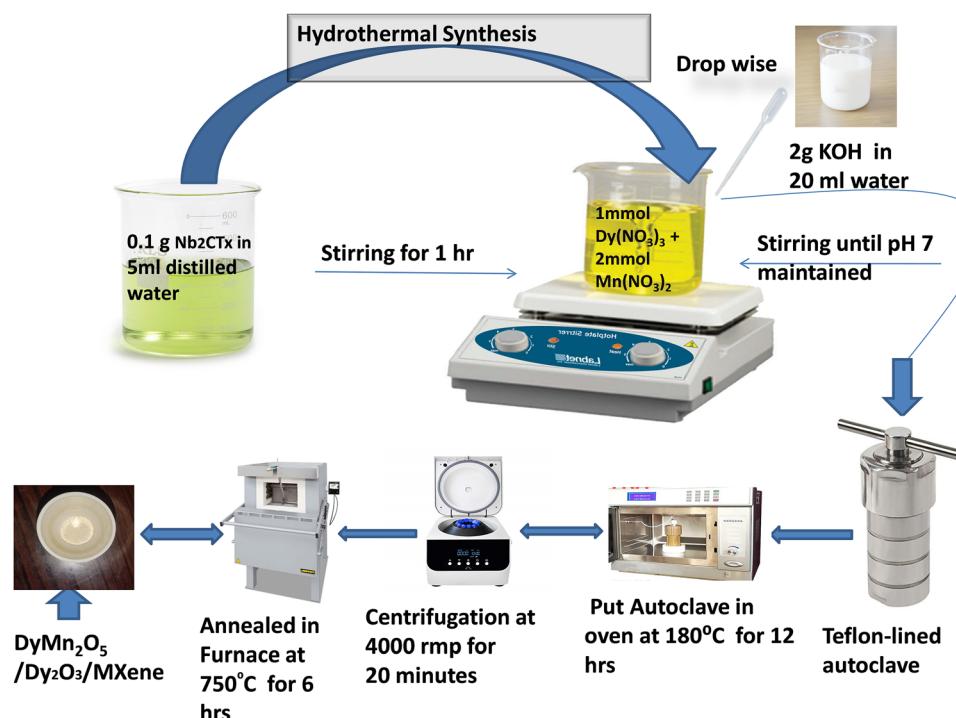


Fig. 2 Hydrothermal synthesis of nanocomposite $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$.

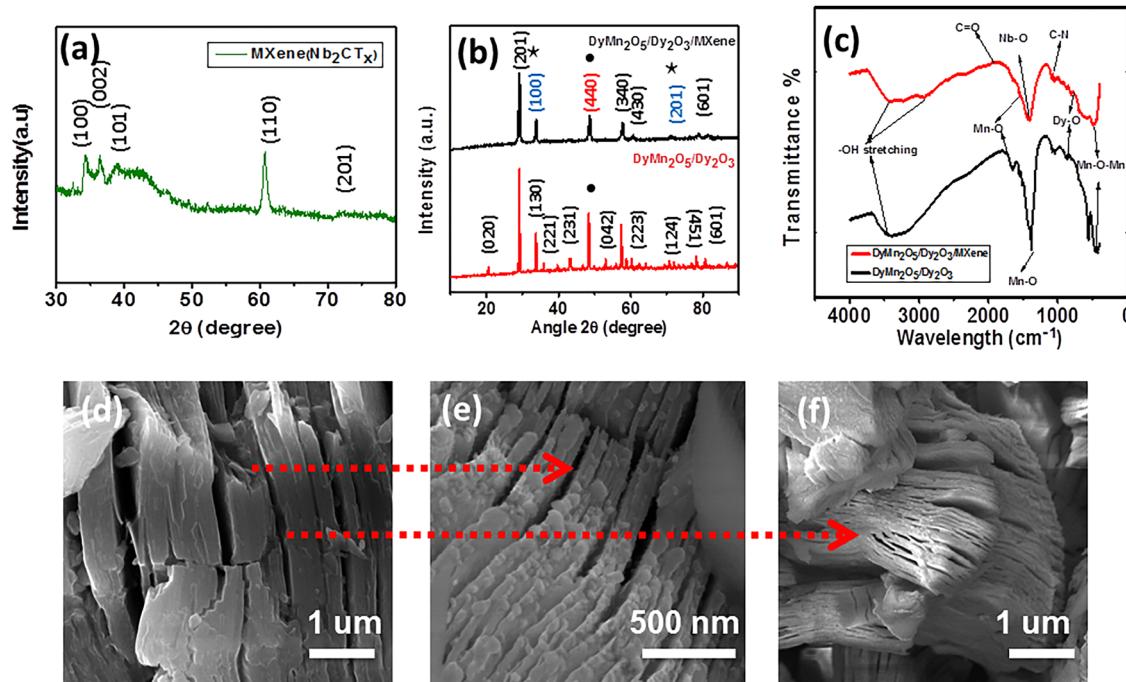


Fig. 3 (a) XRD pattern of Nb_2CT_x , (b) XRD spectrum of $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$ and $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$, (c) FTIR spectrum of both the $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$ and $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$ composite at room temperature, (d) SEM pictograph of the MAX phase of Nb_2AlCT_x at 1 μm magnification, and (e) and (f) SEM images of the layered structure of MXene at high magnification of 500 nm and 1 μm , respectively.

PDF card #15-0127. These peaks confirm the successful formation of Nb_2CT_x MXene.

Fig. 3(d–f) illustrates the morphological characteristics of Nb_2AlCT_x and Nb_2CT_x . As shown in Fig. 3(d), Nb_2AlCT_x exhibits a well-ordered ternary layered structure with no visible delamination, and its surface remains clean and intact. In contrast, the SEM images of Nb_2CT_x MXene in Fig. 3(e and f) captured at resolutions of 500 nm and 1 μm respectively, reveal a distinct stacked structure with well separated layers. This transformation indicates the effective removal of the Al atomic layer from Nb_2AlCT_x through KOH etching, resulting in the characteristic layered morphology of Nb_2CT_x MXene. These observations are consistent with the XRD results, further validating the successful synthesis of Nb_2CT_x MXene.

Furthermore, Fig. 3(b) exhibits the XRD pattern of both synthesized samples. The characteristic peaks in the prepared sample $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$ observed at $2\theta = 20.6^\circ, 29.1^\circ, 33.7^\circ, 35.9^\circ, 43.0^\circ, 53.1^\circ, 57.3^\circ, 58.9^\circ, 70.9^\circ, 78.4^\circ$, and 80.7° correspond to the (020), (201), (130), (221), (231), (042), (340), (223), (124), (451), and (601) crystal planes of orthorhombic DyMn_2O_5 (JCPDS # 01-07-1696). The refined lattice parameters, $a = 7.290 \text{ \AA}$, $b = 8.633 \text{ \AA}$, and $c = 5.626 \text{ \AA}$, exhibit strong agreement with standard values (7.2940, 8.5551, and 5.6875 \AA , respectively). Additionally, a distinct peak at $2\theta = 48.44^\circ$ (●) corresponds to the (440) plane of cubic Dy_2O_3 (JCPDS 00-022-0612), confirming the presence of Dy_2O_3 as a secondary phase. The XRD pattern of the synthesized $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$ composite, shown in Fig. 3(b), was recorded over a broad 2θ range (10° – 90°). The peaks at $2\theta = 29.1^\circ, 57.8^\circ, 60.7^\circ$, and 80.7°

correspond to the (201), (340), (430), and (601) planes of orthorhombic DyMn_2O_5 (JCPDS 01-07-1696). Additionally, the peaks observed at $2\theta = 33.9^\circ$ and 72.7° (★) correspond to the (100) and (201) planes of hexagonal niobium carbide (Nb_2C), indexed to JCPDS # 00-015-0127, confirming the incorporation of the MXene phase. The distinct peak at $2\theta = 48.44^\circ$ (●) is also present in the composite sample, further verifying the coexistence of Dy_2O_3 . The XRD pattern of the DyMn_2O_5 composite with MXene exhibits characteristic peaks corresponding to DyMn_2O_5 , Dy_2O_3 , and Nb_2CT_x , confirming the successful synthesis of the $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$ composite. The Debye–Scherrer formula was employed to assess the crystallite size:

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (1)$$

where D is crystallite size, $K = 0.9$ (Scherrer constant), $\lambda = 0.15406 \text{ nm}$ (wavelength of $\text{K}\alpha$ X-ray sources), θ is peak position in radians and β is full width and half maximum (FWHM). The obtained values of average crystallite size of $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$ and $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$ composite size from eqn (1) are 61.9 nm and 39.4 nm, respectively, which confirms the nanoparticle (1–100 nm) behavior of both samples. In addition, the crystallite size has been calculated by the Williamson–Hall (W–H) model (eqn (2)) and modified Scherrer relation (eqn (3)) using eqn (2) and (3), respectively.

$$\beta_t \cos \theta = \varepsilon(4 \sin \theta) + \frac{K\lambda}{D} \quad (2)$$

$$\ln(\beta) = \ln\left(\frac{1}{\cos \theta}\right) + \ln\left(\frac{K\lambda}{D}\right) \quad (3)$$

Both equations are the equations of a straight line, where $\frac{K\lambda}{D}$ and $\ln\left(\frac{K\lambda}{D}\right)$ respectively, are the y -intercepts of both equations from which crystallite size is measured by W-H plots and modified W-H plots as in Fig. S1(a-d) (ESI[†]). Also, it is very helpful to find strain by extracting the slope and y -intercept of the linear fit as demonstrated in Fig. S1(a and b) (ESI[†]). The estimated values of strain, crystallite size, dislocation density and crystallinity for both samples $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$ and the $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$ composite are given in Table 1.

The crystallite size of $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{Nb}_2\text{CT}_x$ is smaller than that of $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$, indicating a higher surface area, which can contribute to enhanced specific capacitance. Furthermore, the crystallite size estimated using the Williamson–Hall (W-H) plot method is observed to be smaller than that determined by Scherrer's equation. This discrepancy arises because the W-H model accounts for microstrain and instrumental broadening effects, making it a more accurate approach for crystallite size determination.⁵⁹ Dislocation density can provide details about the material's mechanical characteristics and deformation processes. The dislocation density of $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$ is more than that of $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$, which informs us that the mechanical properties of the composite, especially its hardness and strength, have been improved.⁶⁰ Furthermore, the $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$ composite exhibits higher crystallinity compared to $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$, indicating an increase in structural order. Crystallinity is a fundamental parameter that has a significantly impact in determining the overall electrochemical functionality of materials. Highly crystalline structures typically facilitate enhanced charge/discharge kinetics, improved electrical conductivity, and greater structural stability. Therefore, precise control over crystallinity through optimized synthesis and processing techniques is essential for tailoring the electrochemical properties of materials in energy storage and conversion applications.⁶¹

3.1.2. FTIR analysis. The FT-IR spectral analysis of the $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$ and $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$ composite at room temperature is presented in Fig. 3(c) to elucidate its functional group characteristics and chemical bonding interactions in wavenumber range 0–4000 cm^{−1}. The FTIR spectrum of both synthesized nanocomposites displays a broad absorption band around 3456 cm^{−1}, attributed to the stretching vibrations of hydroxyl groups from adsorbed water molecules, while the peak at approximately 1674 cm^{−1} corresponds to the bending vibrations of water molecules.⁶² Similarly, in both

composites, the peak positions at 447 cm^{−1} can be assigned to the stretching vibrations of Mn–O–Mn bonds, while the peak at 803 cm^{−1} can be ascribed to stretching vibration of Dy–O.^{63,64} The presence of well-defined peaks at 1386 and 1668 cm^{−1} corresponds to metal oxide (Mn–O) bonds in the crystal structure. The peak position at 1870 cm^{−1} can be attributed to stretching vibrations of carbonyl groups C=O. A characteristic C–N stretching vibration has been detected at 1125 cm^{−1} in the MXene structure.⁶⁵ Also, the vibration frequency at 1507 cm^{−1} is characteristic of the Nb–O stretching mode.⁶⁶ The FT-IR spectrum of the composite material may offer critical insights into its electrochemical characteristics. The observed slight shift in peak position relative to the pristine $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$ spectrum suggests modifications in the electronic structure arising from the incorporation of Nb_2CT_x . These structural alterations likely influence the material's electrochemical characteristics and efficiency by enhancing its redox activity by introducing additional redox-active sites.

3.1.3. XPS analysis. X-ray photoelectron spectroscopy (XPS) is conducted to elucidate the elemental makeup and oxidation states of the constituent elements in the $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$ composite. The acquired XPS survey spectrum, presented in Fig. 4(a), displays a wide-spectrum graph of DMO/DO/MX (in binding energy range 0–1200 eV) that distinctly confirms the presence of Dy, Mn, Nb, O, and C. Notably, no additional peaks corresponding to other elements were detected, confirming the excellent compositional purity of the prepared material. Furthermore, the Gaussian fitting method was employed to accurately analyze the oxidation states of the constituent elements. The high resolution XPS spectrum of Dy 4d (Fig. 4(b)) exhibits sharp peaks at B.E of 154.5 and 155.6 eV, attributed to Dy 4d_{5/2} and Dy 4d_{3/2}, respectively, proving the characteristic electronic states of dysprosium. One peak associated with 157.1 eV represents the existence of Dy_2O_3 .^{67,68}

The high-resolution XPS spectrum of Mn 2p (Fig. 4(c)) exhibits distinct peaks at 642.9 and 654.03 eV characteristic of Mn 2p_{3/2} and Mn 2p_{1/2} spin-orbit components, respectively, while the peak at 647.6 eV represents a satellite peak. The measured spin-orbit splitting of 11.15 eV between these states is characteristic of Mn³⁺ based materials, further confirming the oxidation state of manganese in the synthesized sample.⁶⁹ Fig. 4(d) presents the high-resolution XPS spectrum of Nb 3d, where the deconvolution of the Nb⁴⁺ 3d_{5/2} and Nb⁴⁺ 3d_{3/2} peaks reveals Nb⁴⁺ oxidation states. The characteristic peak at 206.1 eV is attributed to Nb–C, while the characteristic peak at 208.9 eV corresponds to Nb–O bonding.^{70,71} Fig. 4(e) presents

Table 1 A comparison in different parameters, measured from XRD data, for $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$ and $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$

Sample	Crystallite size by Scherrer (nm)	Crystallite size by W-H model (nm)	Crystallite size by modified Scherrer's equation (nm)	Micro strain $\varepsilon \times 10^{-3}$	Dislocation density $\delta \times 10^{-3} (\text{nm})^{-2}$	Crystallinity (%)
$\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$	61.9	50.6	58.6	1.5	0.44	62.5
$\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{Nb}_2\text{CT}_x$	39.4	29.8	24.6	2.5	0.96	73.4

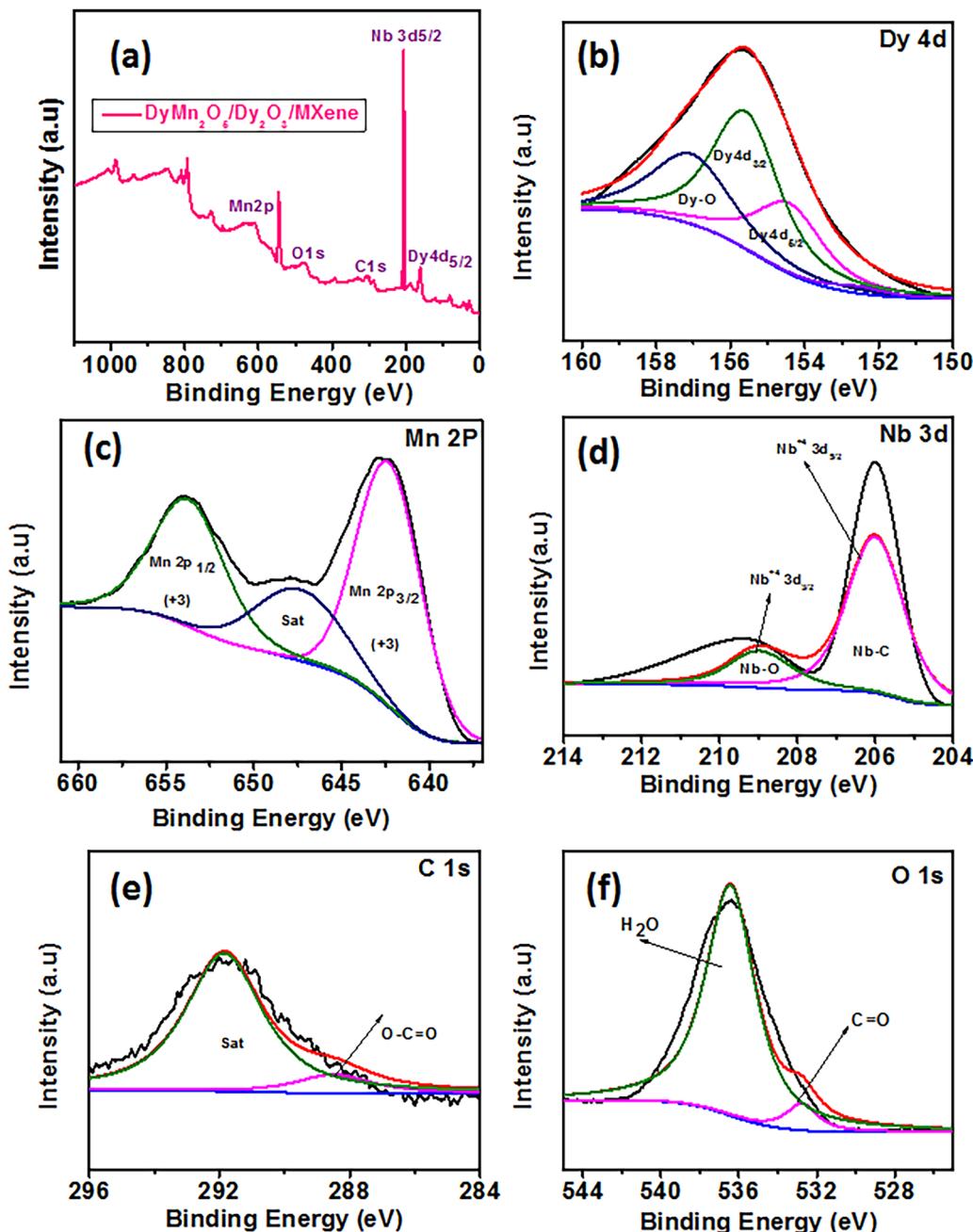


Fig. 4 (a) XPS wide spectrum graph of $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$, and (b) high resolution spectra of Dy 4d, (c) of Mn 2P, (d) of Nb 3d, (e) of C 1s, and (f) of O 1s.

the high resolution XPS spectrum of C 1s, where the deconvolution reveals distinct spectral components. The peak observed at 288.2 eV corresponds to $\text{O}-\text{C}=\text{O}$, indicating modifications in the chemical state of carbon bonded to oxygen. Additionally, the peak at 291.2 eV is identified as a satellite peak, attributed to the presence of C–O functional groups, reflecting the electronic interactions within the material.⁷² Fig. 4(f) depicts the high resolution XPS spectrum of O 1s, where one peak at 532.8 eV is due to chemisorbed oxygen and is also indicative of carbonyl ($\text{C}=\text{O}$) functional groups. Meanwhile, the peak at 536.4 eV is associated with adsorbed molecular oxygen (O_2) or

surface-bound water (H_2O), suggesting the presence of physically adsorbed species on the material's surface.⁷³

3.2. Morphological and compositional analysis

The surface morphology of the $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$ and $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$ composites was systematically analyzed using scanning electron microscopy (SEM), and the corresponding micrographs, captured at varying magnifications, are depicted in Fig. 5 and 6. As observed in Fig. 5(a–c), the $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$ sample exhibits a distinct assembly of peanut-shaped micro-particles that are interconnected, forming a compact structure.

Fig. 5(d) demonstrates the energy-dispersive X-ray (EDX) spectrum of the as-synthesized DyMn_2O_5 , where characteristic peaks confirm the presence of Dy (dysprosium), Mn (manganese), and O (oxygen), with their respective atomic and weight percentages detailed in the inset. The EDX analysis unequivocally verifies the compositional integrity of the synthesized material, further corroborated by X-ray diffraction (XRD) analysis. Moreover, statistical evaluation using ImageJ software, as depicted in the histogram of Fig. 5(e), indicates that the average length of the peanut-shaped $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$ particles is approximately 0.34 μm .

Fig. 6(a and b) presents the SEM micrographs of the $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$ composite at magnifications of 500 nm and 2 μm . The images reveal the presence of small, variably sized cubic-shaped platelets, which are uniformly distributed across the two-dimensional Nb_2CT_x (MXene) layer, with multiple interstitial voids between them. Fig. 6(d) displays the energy-dispersive X-ray (EDX) spectrum of the composite, confirming the presence of Dy (dysprosium), Mn (manganese), O (oxygen), Nb (niobium), and C (carbon), thereby substantiating the elemental composition of the as-synthesized material. Furthermore, statistical analysis performed using Image J software, as represented in the histogram of Fig. 6(e), indicates that the average particle size of the $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$ composite is approximately 78 nm. The extensive surface area of the MXene layers enhances heterogeneous nucleation, thereby promoting the formation of small nanoparticles. Additionally, the hydrophilic nature of MXene and the abundance of surface-anchored functional groups (e.g., $-\text{OH}$, $-\text{F}$, $-\text{O}$) facilitates strong interactions with precursor ions, effectively regulating their diffusion dynamics and constraining particle growth, ultimately leading to a reduction in overall nanoparticle size. The surface morphology and small particle size of $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$ as compared to $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$ gives active sites and

facilitate diffusion of ions, which may ultimately improve electrochemical properties.

3.3. Electrochemical analysis

The electrochemical properties of the synthesized materials (DMO/DO and DMO/DO/MX) were systematically investigated using cyclic voltammetry (CV), galvanostatic charge discharge (GCD), and electrochemical impedance spectroscopy (EIS). Initially, the intrinsic electrochemical behavior of both samples was assessed through CV measurements employing a three-electrode system. In this setup, an Ag/AgCl electrode served as the reference, a platinum (Pt) wire functioned as the counter electrode, and the working electrode was composed of the synthesized material coated onto a nickel (Ni) foam substrate. Cyclic voltammetry (CV) measurements were conducted for all synthesized materials MXene (Nb_2CT_x), $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$ and $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$ in a 3 M KOH electrolyte across a range of scan rates of 10, 20, 30, 40, 50 and 100 mV s^{-1} , within a potential window of 0.1–0.7 V. The corresponding CV profiles are presented in Fig. 7(a–c), illustrating the well-defined sharp redox peaks, which indicates pseudo capacitive behavior of all prepared samples. MXene CV curves, in this positive potential window (0.1–0.7 V), display pronounced redox peaks, indicating pseudocapacitive behavior. This is attributed to reversible faradaic reactions between the surface functional groups of the Nb_2C MXene (e.g., $-\text{OH}$, $-\text{O}$, $-\text{F}$) and the electrolyte ions. Nb-based MXenes, like other transition-metal carbides, can undergo surface redox reactions due to multiple oxidation states of Nb (e.g., $\text{Nb}^{5+}/\text{Nb}^{4+}$), contributing to charge storage *via* pseudocapacitance. Meanwhile, DMO/DO and DMO/DO/MX also depict sharp redox peaks, confirming pseudocapacitive behavior. With increasing scan rates, redox peaks exhibit a shift towards higher potentials, indicating an enhancement in ion transport resistance, while the same shape of the CV curves

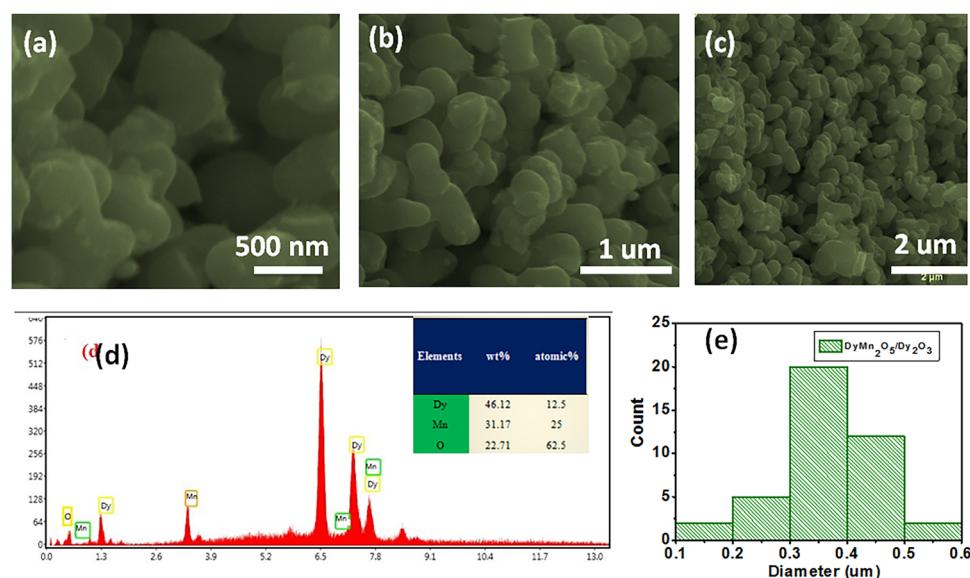


Fig. 5 (a)–(c) Scanning electron microscope (SEM) images of sample $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$ at different magnifications (500 nm, 1–2 μm respectively). (d) EDX image of $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$. (e) Histogram of the statistical distribution of lengths of particles of $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$.

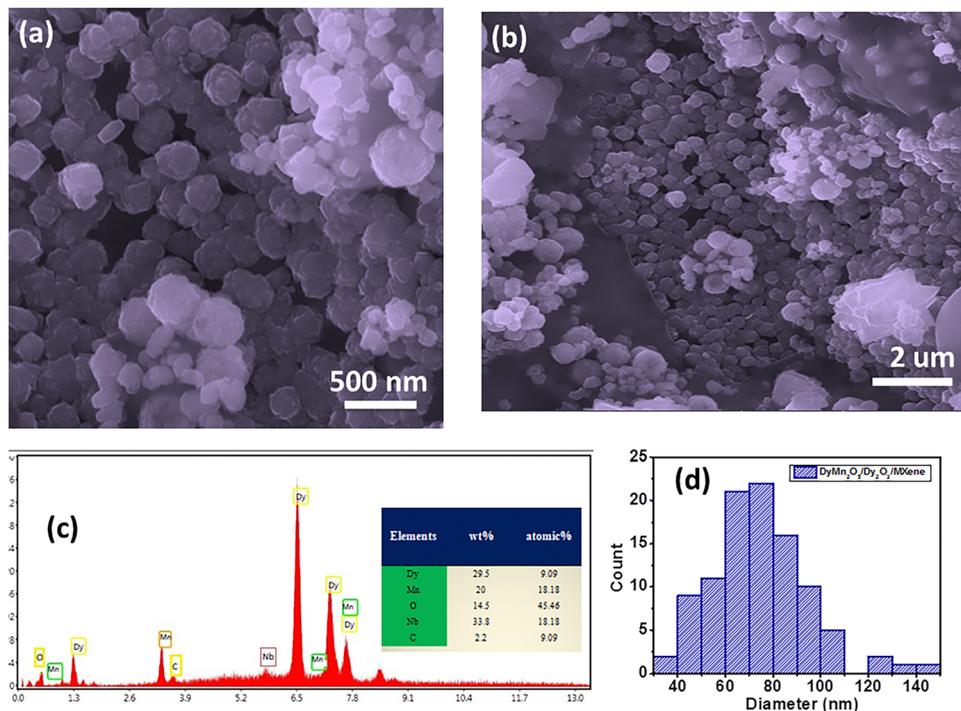
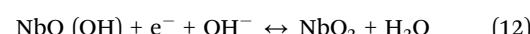
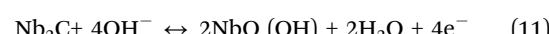
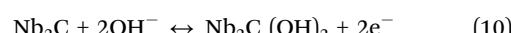
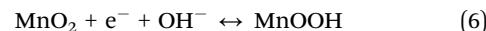
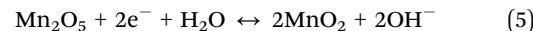


Fig. 6 (a) and (b) SEM pictographs of composite $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$ at different magnifications. (c) EDX image of $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3$ MXene. (d) Histogram of statistical distribution of lengths of particles in composite $\text{DyMn}_2\text{O}_5/\text{Dy}_2\text{O}_3/\text{MXene}$.

even at higher scan rates suggests efficient charge transfer dynamics and stable electrochemical behavior of the electrode material.^{74,75} DMO/DO/MX demonstrates a large integrated area under the CV curve at 10 mV s^{-1} , indicating a significantly enhanced specific capacitance relative to DMO/DO as shown in Fig. 7(d). The specific capacitance of all electrodes, MXene, DMO/DO and DMO/DO/MX is determined through the following equations.⁷⁶

$$C_s = \frac{\int I \times dv}{m \times k \times (\Delta V)} \quad (4)$$

where $\int I \times dv$ is the integral sweep area of the curve, m is the active mass of the electrode, (ΔV) is potential window and k denotes scan rate. The calculated specific capacitances of MXene are 105.9 F g^{-1} , 61.13 F g^{-1} , 50.24 F g^{-1} , 44.30 F g^{-1} , 39.10 F g^{-1} , and 23.92 F g^{-1} , for DMO/DO and 703.9 F g^{-1} , 394.5 F g^{-1} , 285.9 F g^{-1} , 241.2 F g^{-1} , 208.6 F g^{-1} and 130.6 F g^{-1} , and for DMO/DO/MX are 863.1 F g^{-1} , 629.36 F g^{-1} , 530.2 F g^{-1} , 413.9 F g^{-1} , 368.3 F g^{-1} and 213.9 F g^{-1} at scan rates of 10 mV s^{-1} , 20 mV s^{-1} , 30 mV s^{-1} , 40 mV s^{-1} , 50 mV s^{-1} and 100 mV s^{-1} , respectively (Table S1, ESI†). DMO/DO/MX has significantly high specific capacitance as compared to DMO/DO, which may be governed by the presence of Nb_2CT_x MXene. DyMn_2O_5 undergoes redox reactions involving $\text{Mn}^{3+}/\text{Mn}^{2+}$ and $\text{Mn}^{4+}/\text{Mn}^{3+}$ transitions, while Dy_2O_3 dissolves in an alkaline medium, forming hydroxylated species and niobium carbide undergoes surface hydroxylation and redox reactions contributing to charge storage.



The electrochemical performance of the DMO/DO/MX system is predominantly governed by faradaic redox transitions involving Mn, Dy, and Nb species, which contribute to charge storage. Additionally, the surface hydroxylation of Nb_2C enhances its pseudocapacitive behavior, while the 3 M KOH alkaline electrolyte supplies hydroxyl ions, facilitating ion transport and interfacial redox reactions. Various methodologies have been proposed for distinguishing between diffusion-controlled and capacitive (non-diffusion-limited) processes. Dunn (2007) introduced a normalization formula for cyclic voltammetry (CV) kinetics analysis, as represented in eqn (13). Using this approach, the contributions of capacitive and diffusion-controlled processes in the DMO/DO/MX electrode were quantified based on the given equation.⁷⁷

$$i(v) = k_1(V) + k_2(V)^{1/2} = i \text{ (capacitive)} + i \text{ (diffusion)} \quad (13)$$

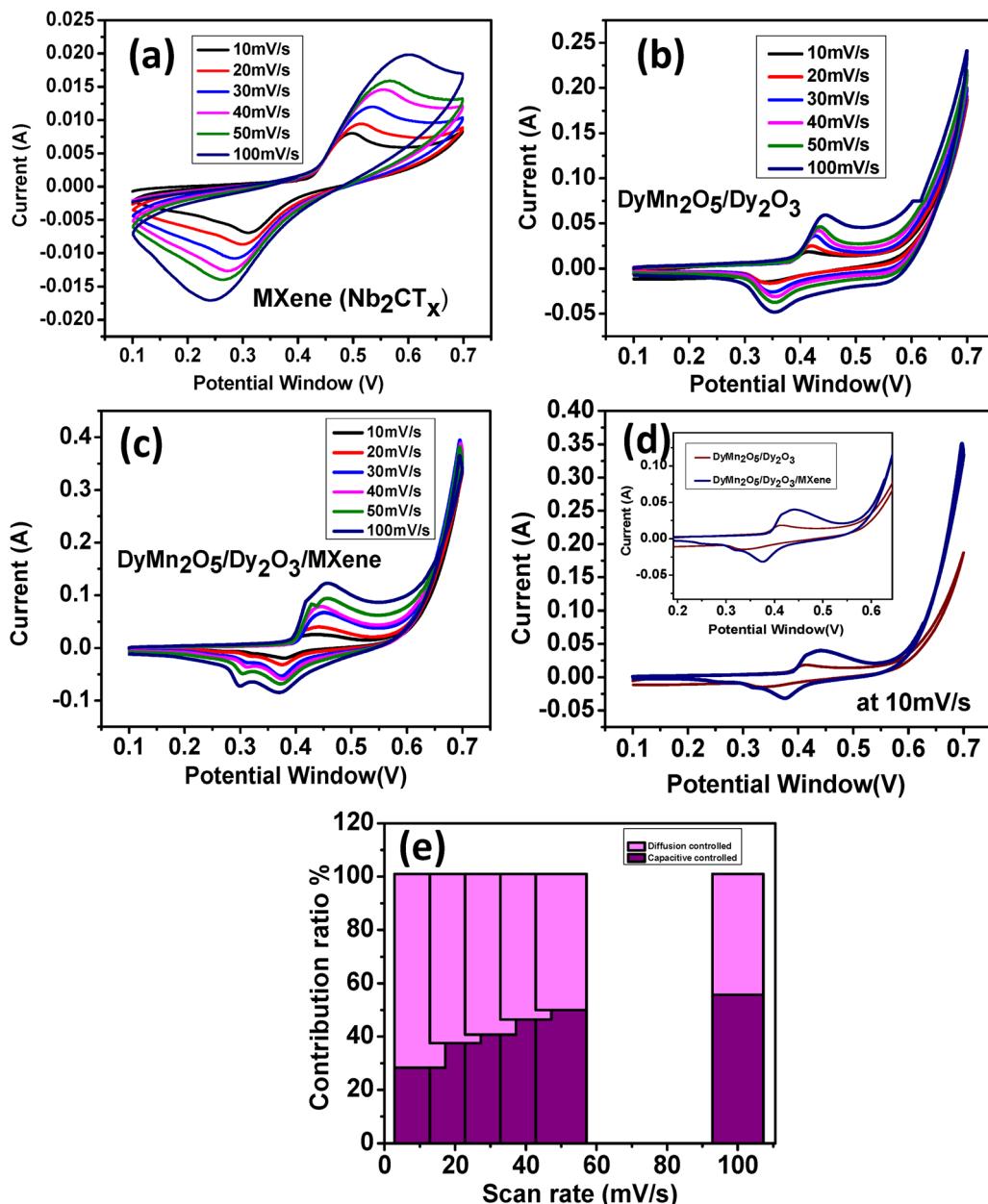


Fig. 7 (a) CV curves of MXenes at different scan rates, (b) and (c) CV curves of DMO/DO and DMO/DO/MX at different scan rates ($10\text{--}100\text{ mV s}^{-1}$), (d) comparison of the CV curves of DMO/DO and DMO/DO/MX at 10 mV s^{-1} and (e) capacitive and diffusion-controlled contributions in DMO/DO/MX at different scan rates.

In this context, $i(V)$ represents the current at a given potential V , while $k_1(V)$ and $k_2(V)^{1/2}$ correspond to the contributions from diffusion limiting (surface capacitive effects) and diffusion-controlled processes, respectively. The graphical representation highlights the dual influence of capacitive effects and ion diffusion on the total capacitance. Furthermore, Fig. 7(e) depicts the total capacitance contribution of the DMO/DO/MX electrode at a scan rate of 20 mV s^{-1} . At scan rates of 10, 20, 30, 40, 50, and 100 mV s^{-1} , the electrode DMO/DO/MX exhibits diffusion controlled contributions of 72.61%, 63.41%, 60.28%, 54.59%, 51.01%, and 45.26%, respectively. Notably, the diffusion controlled contribution of the DMO/DO/MX electrode

dominates, which confirms the pseudo capacitive behavior. At higher scan rates, the time available for ion diffusion decreases, thus enhancing the capacitive controlled contribution.

Fig. 8(a) shows the galvanostatic charge discharge (GCD) curves of Nb_2CT_x MXene at $1, 2, 3$ and 5 A g^{-1} . Fig. 8(b) presents the comparison of the GCD curves of DMO/DO and DMO/DO/MX electrodes recorded across a current density of 1 A g^{-1} within the potential window ($0\text{--}0.5\text{ V}$), providing insights into their electrochemical performance under charge discharge conditions. The observed reduction in the potential window during GCD measurements as compared to CV curves can be attributed to internal resistance, kinetic constraints of redox

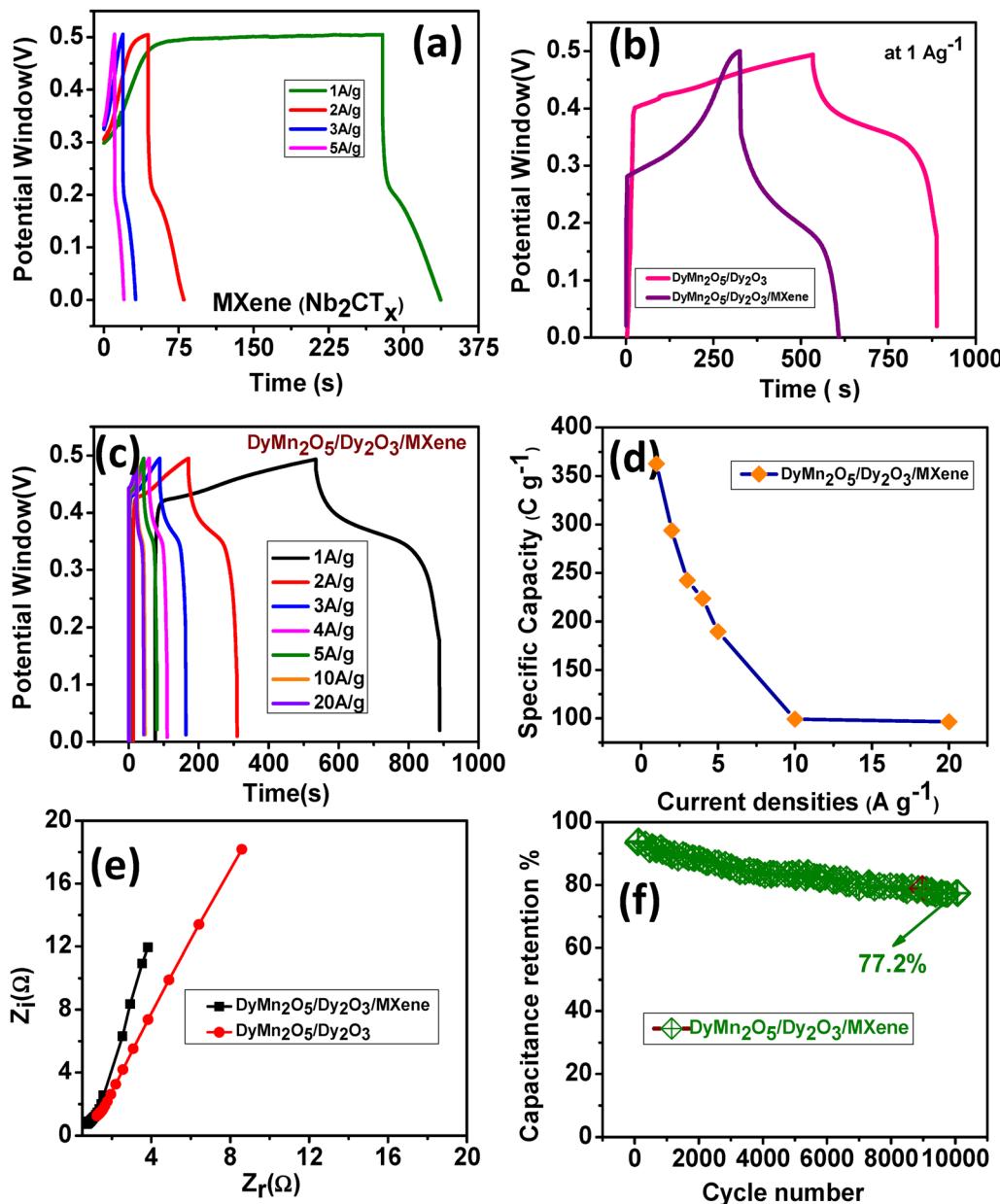


Fig. 8 (a) GCD curves of MXene (Nb₂CT_x) at different current densities, (b) comparison of the GCD curves of DMO/DO and DMO/DO/MX at 1 A g⁻¹, (c) GCD curves of DMO/DO/MX at different current densities (1–20 A g⁻¹), (d) trend depicting that C_s decreases as current density increases, (e) Nyquist plot of DMO/DO and DMO/DO/MX, and (f) cycling stability of DMO/DO/MX at 5 A g⁻¹ over 10 000 cycles.

reactions, and the need to avoid electrolyte decomposition at higher potentials. All electrodes demonstrate a nonlinear charge-discharge curve, confirming the characteristic of purely faradaic behavior of both materials, and also supported the CV results by suggesting a diffusion-controlled redox process. Notably, the DMO/DO/MX electrode exhibits the longest discharge duration as compared to DMO/DO, indicating its superior specific capacity. MXene, DMO/DO and DMO/DO/MX exhibit specific capacity of 70.23 C g⁻¹, 285.41 C g⁻¹ and 362.92 C g⁻¹ respectively at 1 A g⁻¹ within a potential window of 0–0.5 V.

Fig. 8(c) exhibits GCD curves of DMO/DO/MX at different current densities 1, 2, 3, 4, 5, 10 and 20 A g⁻¹ within a potential

window of 0–0.5 V. The specific capacity (C_s) from GCD curves of DMO/DO/MX at different current densities is calculated using the following formula^{78,79}

$$C_s = \frac{I \times t}{m} \quad (14)$$

where C_s denotes the specific capacity (C g⁻¹), I is the discharge current in amperes (A), t is the discharge time (s), and m represents the mass of the active electrode material (g). The specific capacity obtained from eqn (14) is 362.92, 293.78, 242.43, 223.44, 189.55, 99.21, and 96.4 C g⁻¹ at 1, 2, 3, 4, 5, 10 and 20 A g⁻¹, respectively. The nonlinear nature of the GCD curves, along with the evident potential drops at elevated

current densities, suggest a limited availability of active sites within the nanocomposites DMO/DO/MX ultimately leading to a reduction in specific capacity from 362.92 to 96.4 C g⁻¹ (Table S2, ESI†). DMO/DO/MX exhibits prolonged discharge duration and high specific capacity, which can be attributed to a synergistic effect arising from DyMn₂O₅, Dy₂O₃ and Nb₂C, improved electrical conductivity due to MXene layers, efficient and deep electrolyte penetration, large surface area due to MXene layers and also the multiple oxidation states of all metals as confirmed by XPS. With increasing current density, the specific capacity of the DMO/DO/MX electrode gradually declines (Fig. 8(d)). This reduction can be attributed to the limited faradaic response and the intrinsic resistance of the synthesized material under high current conditions.⁸⁰ Table 2 presents a comparative analysis of the specific capacitance of the DMO/DO/MX sample synthesized *via* a simple hydrothermal method in relation to recently reported materials.

Electrochemical impedance spectroscopy was used to study the impedance behavior of both electrode materials in the frequency range of 0.1 to 10⁵ Hz with a small amplitude of 5 mV to ensure system linearity and maintain data quality during measurement. Fig. 8(e) exhibits Nyquist plots of both electrodes. In general, a Nyquist plot consists of two parts, the first part consists of a semicircle in the high frequency range and the other part represents an inclined line in the low frequency range.⁸¹ The resistance of the electrode material, electrolyte and interface of the electrolyte and electrode material can be determined by the *x*-intercept at a high frequency range, termed as solution resistance “R_s”. The charge transfer resistance “R_{ct}” at the electrode material is determined by the diameter of the semicircle.⁷³ The DMO/DO/MX electrode exhibits a relatively low charge transfer resistance (R_{ct} = 10.1 Ω) as compared to DMO/DO (13.9 Ω) indicating the enhanced capacitive performance of the DMO/DO/MX electrode. Furthermore, the Nyquist plot for DMO/DO/MX exhibits a steeper slope in the low-frequency region, with a greater inclination toward the imaginary axis.

This characteristic suggests a lower diffusion resistance compared to the other electrodes, indicating improved ion transport dynamics.⁸² These results indicate that the DMO/DO/MX electrode possesses inherently lower resistance, facilitating more efficient ion transport and electron transfer. This enhanced conductivity contributes to its high specific capacitance and stable cycling performance. The cycling lifespan of a supercapacitor is a critical parameter that significantly

influences its practical applicability and long-term performance. Fig. 8(f) exhibits that the DMO/DO/MX electrode maintained 77.2% of its initial capacitance after 10 000 charge-discharge cycles at a current density of 5 Ag⁻¹. These findings demonstrate that the DyMn₂O₅/Dy₂O₃ composite with MXene significantly gives a high specific capacitance with long-life stability, making it a promising candidate for pseudo capacitor applications.

The enhanced specific capacitance and cycling stability of the DyMn₂O₅/Dy₂O₃ decorated MXene composite can be attributed to several inter-related mechanisms. Firstly, the presence of dysprosium (Dy) ions plays a crucial role in improving capacitive behavior through reversible redox reactions involving Dy³⁺/Dy²⁺ and Mn⁴⁺/Mn³⁺ couples. These redox transitions contribute additional faradaic charge storage, boosting the overall capacitance. Secondly, DyMn₂O₅ and Dy₂O₃ nanoparticles provide multiple active sites for electrochemical reactions, while their stable crystal structures enhance cycling durability. When integrated with MXene, a highly conductive and layered 2D material, a strong interfacial synergy emerges. MXene acts as a conductive backbone that accelerates electron transport and minimizes internal resistance, while also serving as a mechanically robust support that accommodates volume changes during charge-discharge cycles. This synergistic interaction between the redox active rare earth oxides and the conductive MXene matrix leads to improved charge storage kinetics, higher specific capacity, and excellent long term cycling stability.^{58,83,84}

3.4. Assembly of an asymmetric super capacitor (ASC) using DyMn₂O₅/Dy₂O₃/MXene||AC

The remarkable electrochemical performance of the DyMn₂O₅/Dy₂O₃/MXene composite necessitated a comprehensive evaluation of its energy storage potential for practical applications. To assess its practical viability, an asymmetric supercapacitor (ASC) was fabricated, integrating DMO/DO/MX as the positive electrode and activated carbon (AC) as the negative electrode (designated as DMO/DO/MX||AC ASC). The 2 M KOH electrolyte was utilized to enhance ionic conductivity and facilitate efficient charge transfer between the electrodes. The active loaded mass of DMO/DO/MX was approximately 3.2 mg. Fig. 9(a) presents the cyclic voltammetry (CV) curves of the DMO/DO/MX and Activated carbon (AC) at different potential windows and at the same scan rate of 10 mV s⁻¹. CV experiments were performed over progressively wider potential windows ranging

Table 2 Comparison of the electrochemical performance of the synthesized DMO/DO/MX in this work and recent reported work

S. no.	Material	Specific capacitance (F g ⁻¹)	Morphology	Synthesis method	Ref.
1	SrMnO ₃	446 F g ⁻¹ at 1 A g ⁻¹	Nano fibers	Sol gel	85
2	CeO ₂ /LaMnO ₃	262 F g ⁻¹ at 1 A g ⁻¹	Spherical nanoparticles	Co-precipitation	86
3	DyMnO ₃	531 F g ⁻¹ at 1 A g ⁻¹		Microwave-assisted synthesis	87
4	Dy ₂ NiMnO ₆	395.2 F g ⁻¹ at 0.5 A g ⁻¹	Mesoporous sphere	Hydrothermal	88
5	La ₂ CuMnO ₆	206 F g ⁻¹ at 1 A g ⁻¹	Nanostructures	Hydrothermal	89
6	Y ₂ NiMnO ₆	78 F g ⁻¹ at 1 A g ⁻¹	Nanowires	Hydrothermal	90
7	α-MnO ₂	111 F g ⁻¹ at 5 mV s ⁻¹	Nanorods	Hydrothermal	91
8	DyMn ₂ O ₅ /Dy ₂ O ₃ /MXene	725.8 F g ⁻¹ at 1 A g ⁻¹	Cubic pallets	Hydrothermal	This work

from 1.1 to 1.5 V at 20 mV s^{-1} as depicted in Fig. 9(b). As the potential window increased, the enclosed area of each CV curve expanded, reflecting the greater energy stored at the enlarged operating potential window of 1.5 V. Above 1.5 V, oxygen evolution occurs, limiting the stability of the device. Consequently, 1.5 V was adopted as the maximum operating potential window for all subsequent electrochemical measurements of the ASC device. As anticipated, the fabricated asymmetric device achieves an extended operating voltage of 1.5 V, attributed to the synergistic integration of the electrochemically stable DMO/DO/MX positive electrode and the robust AC negative electrode.⁹² CV curves of DMO/DO/MX//AC were recorded at scan rates ranging from 3 to 80 mV s^{-1} within

an operational voltage window of 0–1.5 V, as illustrated in Fig. 9(c). The presence of distinct redox peaks at 1.27 V and 0.80 V signifies a predominantly faradaic charge storage mechanism indicating pseudocapacitive behavior. A progressive increase in peak current was recorded with no significant distortion in the shape of the CV curves, even at higher scan rates, indicating the fabricated ASC's excellent rate capability and superior electrochemical reversibility. The GCD curves presented in Fig. 9(d) exhibit a non-linear quasi triangle shaped curve, reflecting efficient charge storage dynamics and favorable pseudocapacitive characteristics. The specific capacitance of the fabricated device, determined from the GCD curves was measured as 148.2, 84.5, 76.38, 72.4, 70.02, 68.1 and 60.2 F g^{-1} at current

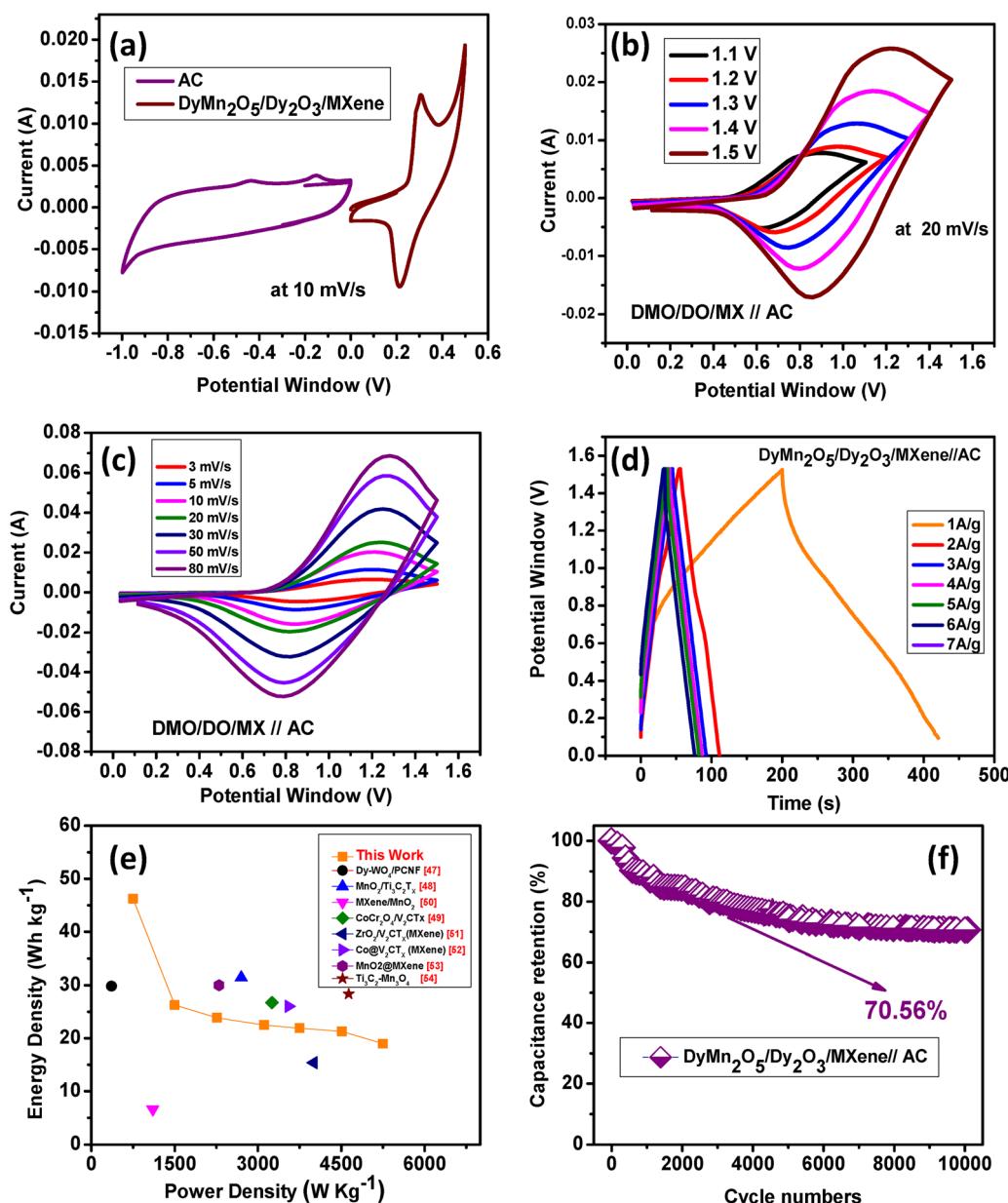


Fig. 9 (a) CV curves of AC and DMO/DO/MX at different potential windows at 10 mV s^{-1} , (b) CV curves of DMO/DO/MX//AC at different potential windows from 1.1–1.5 V at 20 mV s^{-1} and (c) CV curves of DMO/DO/MX//AC at different scan rates from 3–80 mV s^{-1} . (d) GCD curves of DMO/DO/MX//AC at 1, 2, 3, 4, 5, 6 & 7 A g^{-1} . (e) Ragone plot of DMO/DO/MX//AC. (f) Cyclic stability of DMO/DO/MX//AC during 10 000 cycles.

densities of 1, 2, 3, 4, 5, 6 and 7 A g⁻¹, respectively. The energy density (E_d) and the critical performance metric, power density (P_d), serve as fundamental parameters for assessing the energy efficiency of the fabricated asymmetric device and they are calculated from the GCD curves using the following equations.⁹³

$$E_d = (C_s \times \Delta V^2) / (2 \times 3.6) \quad (15)$$

$$P_d = \frac{E}{\Delta t} \quad (16)$$

Fig. 9(e) illustrates the Ragone plot of the DyMn₂O₅/Dy₂O₃/MXene//AC ASC, demonstrating that the fabricated device achieves a high energy density of 46.25 W h kg⁻¹ at a power density of 750 W kg⁻¹. Furthermore, it retains an energy density of 18.96 W h kg⁻¹ even at a maximum power density of 5250.20 W kg⁻¹. This performance is comparable to, and in certain aspects surpasses, previously reported transition and rare earth-based metal oxides and MXene-based supercapacitors such as MnO₂@CNT//AC (13.4 W h kg⁻¹ at 602 W kg⁻¹),⁹⁴ Ti₃C₂T_x-δ-MnO₂//AC (8.2 W h kg⁻¹ at 400 W kg⁻¹),⁹⁵ λ-MnO₂//Ti₃C₂T_x (15.5 W h kg⁻¹ at 100 W kg⁻¹),⁹⁶ Ni dope CeO₂@d-Ti₃CN (4.4 W h kg⁻¹ at 495 W kg⁻¹),⁹⁷ Dy doped CoFe₂O₄ (15.23 W h kg⁻¹ at 450 W kg⁻¹)⁹⁸ and CeO₂/LaMnO₃//AC (17.2 W h kg⁻¹ at 1015 W kg⁻¹).⁸⁶ The cycling stability of the DMO/DO/MX//AC ASC was evaluated at a current density of 2 A g⁻¹ over 10 000 charge-discharge cycles (Fig. 9(f)), demonstrating a capacitance retention of 70.56%. This high retention exhibits the device's excellent stability and highlights its potential for high performance asymmetric supercapacitor (ASC) applications.

In this study, the fabricated system introduces a straightforward approach for synthesizing Nb₂CT_x-based MXene and rare earth metal-based oxide composites with outstanding cycling stability and high specific capacitance. This research offers innovative approaches for the development of flexible electrode materials with superior electrochemical performance, paving the way for next-generation portable and wearable electronic devices.

Conclusions

In this study, a novel rare earth-based bi-metallic oxide DyMn₂O₅/Dy₂O₃/MXene nanocomposite was successfully synthesized using a hydrothermal method to enhance the interlayer spacing of MXene for improved electrochemical performance. The incorporation of DyMn₂O₅/Dy₂O₃ nanoparticles effectively mitigated the restacking issue of MXene, leading to an optimized electrode material with a high specific capacitance of 362.92 C g⁻¹ at 1 A g⁻¹ with 77% capacitance retention over 10 000 discharge cycles. Furthermore, an asymmetric supercapacitor assembled with the DyMn₂O₅/Dy₂O₃/MXene electrode and an activated carbon cathode exhibited a commendable energy density of 46.825 W h kg⁻¹ at a power density of 750 W kg⁻¹, retaining 70.56% of its initial capacitance after 10 000 cycles at 2 A g⁻¹. These findings highlight the potential of rare earth-based MXene nanocomposites as high-

performance electrode materials for next-generation supercapacitors, demonstrating the effectiveness of the hydrothermal approach in tailoring nanostructured materials for energy storage applications.

Author contributions

Komal Ali Rao: the conceptualization, formal analysis, investigation, methodology, data curation, software, writing original draft, editing and review, Javed Ahmad, Muhammad Bilal: resources and review Muhammad Ehsan Mazhar, Muhammad Imran Khan, Muhammad Suleman Ahmad, Muhammad Aziz and Hafeez ur rehman: review.

Data availability

All data generated or analyzed during this study are included in this article. Furthermore all the related data are available from the authors upon request.

Conflicts of interest

The authors declare that they have no known competing financial interest or personal relationships that could have appeared to influence the work reported in this paper.

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