

A new era in energy storage: *In Situ* and *Operando* characterization of 2D materials-based devices

Kotturu V.V. Chandra Mouli ^{a,1}, Durai Mani ^{b,c,1}, Tholkappiyan Ramachandran ^d,
Yedluri Anil Kumar ^e, Avijit Ghosh ^f, Prathap Somu ^{g,*}, Ramanadha Mangiri ^{h,**}

^a Department of Mechanical and Industrial Engineering, College of Engineering, Majmaah University, Al- Majmaah, 11952, Saudi Arabia

^b Department of Civil Engineering, Yeungnam University, Gyeongsan, 38541, Republic of Korea

^c Centre for Promotion of Research, Graphic Era (Deemed to be University), Clement town, Dehradun, Uttarakhand, 248002, India

^d Department of Physics, Saveetha School of Engineering, Saveetha Institute of Medical and Technical Sciences (SIMATS), Thandalam, Chennai, 602105, India

^e Department of Chemistry, Lovely Professional University, Phagwara, 144411, Punjab, India

^f Department of Electrical and Electronic Engineering, Begum Rokeya University, Rangpur, 5400, Bangladesh

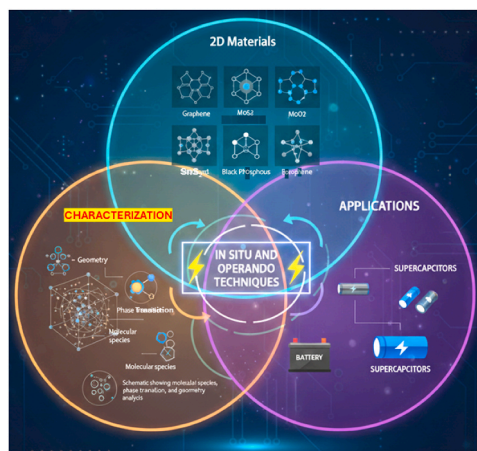
^g Department of Biotechnology and Chemical Engineering, School of Engineering, Faculty of Science, Technology and Architecture (FoSTA), Manipal University Jaipur, Dehmi Kalan, Off. Jaipur-Ajmer Expressway, Jaipur, 303007, Rajasthan, India

^h Department of Physics and Semiconductor Science, Gachon University, Seongnam-si, 13120, Republic of Korea

HIGHLIGHTS

- 2D materials enable high-performance batteries and supercapacitors.
- Review covers synthesis, functionalization, and surface engineering methods.
- In situ and operando tools reveal real-time structural and electrochemical changes.
- Advanced studies expose degradation, phase transitions, and interfacial processes.
- Future trends highlight scalable, durable 2D-based energy storage solutions.

GRAPHICAL ABSTRACT



ARTICLE INFO

Keywords:

2D materials

In situ characterization

Operando techniques

ABSTRACT

Two-dimensional (2D) materials have emerged as promising candidates for advanced electrochemical energy storage devices, such as batteries and supercapacitors, owing to their exceptional surface area, electrical conductivity, and tunable properties. This review provides a comprehensive overview of the synthesis and

* Corresponding author.

** Corresponding author.

E-mail addresses: prathaps1987@gmail.com (P. Somu), ramanadh@gachon.ac.kr (R. Mangiri).

¹ The authors (K V V C MOULI c.mouli@mu.edu and Durai Mani) contributed equally to this work.

Energy storage devices
Electrochemical performance
Surface modification
Solid-electrolyte interface

preparation methods of 2D materials, highlighting their critical roles in optimizing device performance. Central to this discussion is the exploration of *in situ* and *operando* characterization techniques, which enable real-time monitoring of structural, chemical, and electrochemical changes within 2D materials-based devices under working conditions. We systematically examine key electrochemical characterization methods, including Electrochemical Impedance Spectroscopy (EIS), Cyclic Voltammetry (CV), Galvanostatic Charge-Discharge (GCD), and Differential Capacity Analysis, alongside advanced *operando* approaches that capture dynamic processes during device operation. The review further delves into recent *operando* studies applied in real-time device applications, offering insights into degradation mechanisms, phase transformations, and interfacial phenomena. Special attention is devoted to the functionalization and surface engineering of 2D materials, underscoring strategies for performance enhancement and their *operando* monitoring. Challenges and limitations inherent to *in situ* and *operando* techniques are critically analyzed to guide future improvements in measurement accuracy and device integration. Finally, we discuss emerging trends and future perspectives in the field, emphasizing the technological outlook for next-generation energy storage solutions based on 2D materials. This review aims to provide a detailed understanding of the synergy between 2D materials and advanced characterization techniques, fostering progress towards more efficient, durable, and scalable energy storage technologies.

1. Introduction

The development of advanced electrochemical power storage systems is becoming widespread because of the increasing quest for renewable energy [1]. Power storage systems are becoming more and more important as the world becomes less dependent on fossil fuels and more on renewable energy sources such as wind and sunlight. Aside from the fact that fossil fuels have limited reserves of energy, unlike these energy resources, they can provide an energy source every day of the year. Batteries and supercapacitors, which are classified as electrochemical energy storage devices, are invaluable in extending the generation mix energy produced in excess during peak production times and drawing down the energy during low or peak demand periods. There is ongoing research looking for materials for these energy devices that do not sacrifice efficiency and reliability as the energy demand continues to rise [2–6]. In this regard, two-dimensional (2D) materials came among the best alternatives for energy storage systems of the next generation. Lot of attention in the world has been created with the advent of 2D materials after the isolation and separation of graphene, which is a single layer of a hexagonal structure of carbon atoms [8–10]. These materials tend to exhibit high surface area, high electrical functionality and adjustable surface chemistry. They also tend to have very few considerably thick layers. 2D materials are the best for electrochemical devices because there are layers in the structure that can accommodate layers of ions and combine high ionic conductivity and good electrical insulation while retaining some flexibility.

Electrochemical energy storage (EES) devices are complex systems governed by ion conduction, charge transfer, phase transitions, and solid–electrolyte interphase (SEI) formation [11–13]. These coupled, multiscale processes make it difficult to identify the key factors controlling the performance and stability of 2D materials [14–17]. SEI formation, in particular, strongly affects cycling stability and is highly sensitive to structural features of 2D materials. In MoS₂ and MXenes, for instance, defects, edge sites, and layer thickness influence SEI chemistry by dictating electrolyte decomposition, ion accessibility, and diffusion pathways. Conventional *ex situ* methods capture only pre- or post-cycling snapshots, highlighting the need for advanced *in situ* and *operando* techniques that reveal dynamic processes in real time [18–21]. *In situ* techniques probe materials under controlled, non-operational conditions (e.g., temperature, atmosphere), while *operando* techniques monitor structural and chemical changes under actual device operation, directly linking them to electrochemical performance [22–25]. Both approaches have advanced the understanding of 2D materials. For example, *in situ* TEM enables real-time observation of atomic defects and ion transport, while *in situ* XRD tracks structural evolution during intercalation [26–28]. *Operando* Raman and X-ray absorption spectroscopy (XAS) further provide insights into surface chemistry and redox processes during cycling [29,30]. A comparative overview of these methods for 2D materials is summarized in Table 1.

Due to the application of 2D materials in electricity storage, exhaustion is among the leading concerns, particularly during rate and discharge cycles when many procedures are carried out over and over again. The other contributing factor to batteries' durability is the creation of the SEI interface, which is an interface that comes into being after several charging and discharging cycles of an electrode [63]. There has been a massive progression in addressing the development and growth of the SEI layer that inhibited the performance of 2D materials by *in situ* and *operando* techniques. These effects have made it possible to devise ways the SEI could be avoided being adverse. These techniques include surface engineering and the addition of modified elements into electrolytes, intending to improve the operational period and performance of 2D materials used for the storage of energy [64]. Fig. 1 depicts the overview of 2D materials.

Another contributing aspect to the effectiveness of 2D materials is ionic intercalation and deintercalation. To use 2D materials in batteries, it is important to have a layered structure such that it allows ions to be inserted reversibly between the layers [65]. However, too many cycles of faithful insertion and extraction of the ions may lead to structural alterations such as swelling and shrinking of the layers that can ultimately compromise the structural integrity of the material. These changes in microstructure have now become achievable with the enhancement of *operando* and *in situ* techniques, specifically in real-time X-ray diffraction which has applications in understanding the ion incorporation phenomenon in 2D materials during the cycling process. This intelligence is very vital to the enhancement of the design of 2D materials for purposes of potential and charge performance and cycling stability improvement as well [66]. The domain of 2D materials, especially in terms of electrochemical energy devices, still has further elements that need to be researched. However, the performance of such materials depends on certain underlying standards aspired in the foregoing technology and this is where more sophistication is required [67]. The emergence of the MXenes family of 2D transition metal carbides and nitrides has been one of the most significant advances in the last decade. Thanks to the properties of MXenes including high electrical conductivity, hydrophilic character and tunable surface chemistry, the interest in their application for energy storage devices has grown considerably. Most if not all *in situ* and *operando* studies carried out on MXenes have contributed significantly to understanding their global cycling history changes and the processes responsible for their electrochemical activity [68]. These however are leaning towards research of other types of MXenes which can enhance energy storage even further. Besides batteries, 2D materials are also being incorporated into a different kind of energy storage device known as supercapacitors. Supercapacitors, on the other hand, do not make use of chemical changes to charge their electrodes. Instead, they apply electrostatic forces between the negative electrode and the electrolyte. 2D materials are considered the best for the construction of supercapacitor electrodes because these materials have very high conductivity (e.g., for MXenes: $\sim 10^4$ S/cm) and

Table 1Performance analysis of *Operando* and *In Situ*, characterization of 2D materials.

Performance Metric	Device Type	2D Material Example	<i>In Situ</i> Technique	<i>Operando</i> Technique	Synthesis Method	Key Findings	References
Capacitance	Supercapacitors	Graphene	CV	Galvanostatic Charge/Discharge	Chemical Vapor Deposition (CVD)	High surface area and conductivity yield excellent capacitance	[31]
Energy Density	Supercapacitors	MoS ₂	EIS	CV	Hydrothermal Synthesis	Layered TMD structure improves energy storage capacity	[32]
Power Density	Batteries	Ti ₃ C ₂ (MXene)	CV	Charge/Discharge Cycling	Selective Etching of MAX phases	Metallic conductivity drives high power density	[33]
Rate Capability	Batteries	WS ₂	CV	Galvanostatic Tests	Chemical Exfoliation	Layered WS ₂ supports rapid ion diffusion	[34]
Charge Transfer Resistance	Supercapacitors	MXenes	EIS	CV	Etching + Delamination	Low resistance aids rapid charge transfer	[35]
Cycle Stability	Batteries	Black Phosphorus	CV	Cycle Life Tests	Liquid Exfoliation	Stability limited by structural degradation	[36]
Reaction Kinetics	Supercapacitors	rGO	Raman Spectroscopy	CV	Reduction of Graphene Oxide	Surface functional groups accelerate redox reactions	[37]
Structural Integrity	Batteries	h-BN	TEM	XRD	Mechanical Exfoliation	Crystal structure maintained, ensuring long-term integrity	[38]
Ion Diffusion Coefficient	Supercapacitors	MoS ₂	EIS	Diffusion Tests	Hydrothermal Synthesis	Layered morphology enhances ion transport	[32]
Conductivity	Batteries	Graphene	Four-Probe Method	<i>In Situ</i> EIS	CVD	Exceptional electrical conductivity lowers device resistance	[31]
Voltage Window	Batteries	Ti ₃ C ₂ (MXene)	CV	Charge/Discharge Cycling	Selective Etching	Broad electrochemical stability window improves energy density	[33]
Electrochemical Stability	Supercapacitors	MXenes	EIS	Cycle Tests	Etching + Delamination	More stable in operando measurements	[39]
Surface Area	Batteries	rGO	BET Surface Area Analysis	XPS	Reduction of Graphene Oxide	Larger surface area observed <i>in situ</i>	[40]
Electrolyte Interaction	Supercapacitors	WS ₂	NMR	XPS	Chemical Exfoliation	Better <i>operando</i> understanding of electrolyte interactions	[41]
Capacitance Retention	Supercapacitors	Graphene	CV	Cycle Tests	CVD	Higher capacitance retention in <i>operando</i>	[42]
Morphological Changes	Batteries	Black Phosphorus	SEM	AFM	Liquid Exfoliation	More morphological changes observed <i>in situ</i>	[43]
Material Degradation	Supercapacitors	MoS ₂	TEM	CV	Hydrothermal Synthesis	More detailed insights on degradation in <i>operando</i>	[44]
Electrode/Electrolyte Interface	Batteries	Ti ₃ C ₂ (MXene)	XPS	EIS	Selective Etching	Clearer interface analysis <i>in situ</i>	[45]
Temperature Effects	Batteries	h-BN	Raman Spectroscopy	XRD	Mechanical Exfoliation	Temperature effects clearer in <i>operando</i>	[46]
Charge Storage Mechanism	Supercapacitors	MXenes	CV	EIS	Etching + Delamination	Mechanisms better resolved <i>in situ</i>	[47]
Performance Under Strain	Batteries	rGO	AFM	XPS	Reduction of Graphene Oxide	Performance variation clearer <i>in situ</i>	[48]
Particle Size Distribution	Supercapacitors	Graphene	DLS	TEM	CVD	Size distribution clearer in <i>operando</i>	[49]
Surface Functionalization	Batteries	MoS ₂	XPS	Raman Spectroscopy	Hydrothermal Synthesis	Functionalization clearer in <i>operando</i>	[50]
Voltage Hysteresis	Supercapacitors	WS ₂	CV	Galvanostatic Tests	Chemical Exfoliation	Lower voltage hysteresis <i>in situ</i>	[51]
Interface Stability	Batteries	Black Phosphorus	TEM	EIS	Liquid Exfoliation	Stability insights clearer in <i>operando</i>	[52]
Gas Evolution Studies	Batteries	Ti ₃ C ₂ (MXene)	NMR	CV	Selective Etching	Gas evolution clearer <i>in situ</i>	[53]
Stability of Electrolytes	Supercapacitors	Graphene	EIS	XRD	CVD	Electrolyte stability better in <i>operando</i>	[54]
Size Effects on Performance	Batteries	MoS ₂	TEM	SEM	Hydrothermal Synthesis	Size effects clearer <i>in situ</i>	[55]
Variability in Performance	Supercapacitors	MXenes	EIS	Galvanostatic Tests	Etching + Delamination	Better insights in <i>operando</i>	[56]
Reversible Capacity	Batteries	h-BN	CV	Charge/Discharge Cycling	Mechanical Exfoliation	Higher reversibility <i>in situ</i>	[57]
Doping Effects	Supercapacitors	rGO	XPS	Raman Spectroscopy	Reduction of Graphene Oxide	Doping effects clearer in <i>operando</i>	[58]
Ion Selectivity	Batteries	Ti ₃ C ₂ (MXene)	EIS	CV	Selective Etching	Selectivity insights clearer <i>in situ</i>	[59]
Comprehensive Characterization	Supercapacitors	MoS ₂	SEM	XPS	Hydrothermal Synthesis	More comprehensive <i>operando</i> analysis	[60]
Transition Metal Ion Interactions	Batteries	Graphene	NMR	Raman Spectroscopy	CVD	Better understanding <i>in situ</i>	[61]
Durability Under Cycling	Batteries	Black Phosphorus	CV	Cycle Tests	Liquid Exfoliation	Improved durability in <i>operando</i>	[62]

capacitance (e.g., MoS_2 : $\sim 200\text{--}300\text{ F g}^{-1}$, graphene: $\sim 100\text{--}150\text{ F g}^{-1}$) and a wide surface area, which is necessary to achieve high charge/discharge rates. The other area of study has been the assessment of the rate storage mechanisms of 2D materials such as how different structures and compositions will affect performances electrochemically and this has also been done *in situ* and *operando* [69]. These studies have suggested other approaches for the development of 2D-based long-cycle high-energy supercapacitors.

Using *in situ* and *operando* characterization techniques to enhance our understanding of 2D systems encountered some challenges. The primary challenge is the sophisticated experimental arrangements that are associated with these methods. The majority of *in situ* and *operando* experimentation includes a specialized system, such as synchrotron resources or expensive electron microscopes, which are mostly unavailable in many research centres. Also, given that the behaviour of 2D materials can be affected by many factors such as surface chemistry, ion diffusion, or even phase changes, it may be difficult to interpret the results obtained with such approaches [70]. Computational modelling and machine learning are more advanced examples of data analysis techniques that have already been implemented to help facilitate the study of the complex *in situ* and *operando* data. Another challenge faced is the quest for new active and *in situ* techniques capable of more effectively observing 2D materials. There have been incorporated systems that have provided an enormous amount of information related to the physical and chemical changes during the processes of operation within cycles. However, the principles that underline the working of 2D materials are rather still rudimentary. It is very difficult for example to comprehend the extent to which mechanical stress concerning the electrochemical performance of 2D materials, or the movement of ions

across their surfaces, can be achieved on the atomic level without the aid of real-time and crystal-clear imaging equipment of the processes taking place [71–73].

In situ and *operando* characterization techniques have revolutionized the understanding of 2D materials in electrochemical energy storage by enabling direct observation of structural, compositional, and electrochemical changes in real time. This review aims to highlight how these advanced methods have deepened insights into the dynamic behaviors of 2D materials within operating devices such as batteries and supercapacitors. By capturing the evolution of 2D structures during charge storage processes, these techniques pave the way for designing energy storage devices that are more cost-effective, efficient, and durable. As the field evolves, *operando* and *in situ* methods will become indispensable tools to unlock the full potential of 2D materials in next-generation energy storage technologies.

2. Overview of 2D materials in electrochemical energy storage

Since the invention of graphene, which turned into made viable through micromechanical cleavage using scotch tape, the manufacturing of a huge range of 2D substances with thicknesses ranging from one to 3 atoms has advanced appreciably [74]. These current materials generally fall into precise elemental groups, in general from Groups IV and V of the periodic table and all of them have features that lead them to be valuable for current makes use of. To give an instance, the electronic structures of the Group IV materials, which include stanene, germanene and silicene, are akin to those of carbon in graphene. Nonetheless, these Group IV materials generally show a buckled hexagonal association of their maximum stable conformation,

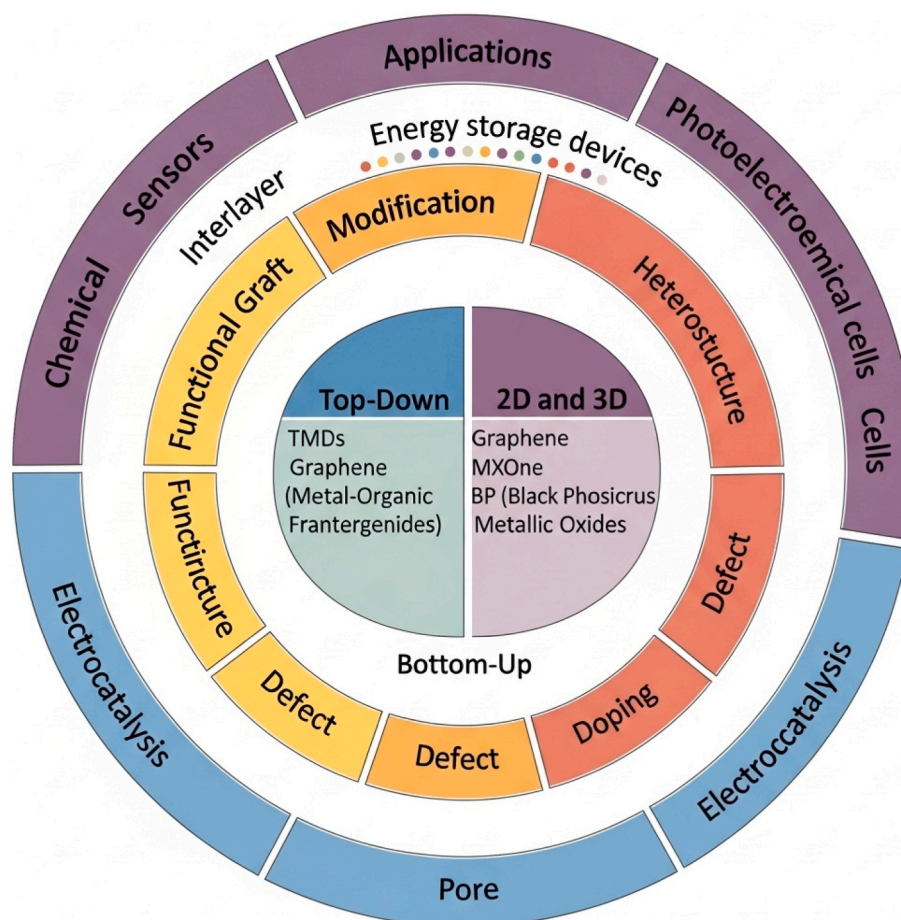


Fig. 1. Overview of 2D materials.

regardless of the truth that graphene has a planar and tessellated shape composed of carbon atoms [75]. Structure-sensible differentiation does not simplest effects in various digital characteristics, however, it additionally gives the possibility of massless Dirac fermions, which may be exploited in graphene-based totally optoelectronics. Furthermore, phosphorene, arsenide, antimonene and bismuthene are derived from group five factors in the usage of growing technology which include exfoliation techniques that entail the separation of bulks. Because of those materials layer-established digital residences, band gaps and conductivities can range, allowing for a huge variety of electrical and power garage programs. MXT_z, in which M denotes an early transition metal, X denotes carbon and/or nitrogen and T_z denotes terminal practical companies like -OH, -O, or -F gift on the surface, characterizes MXenes, some other type of 2D materials. MXenes are considered promising electrode materials with the capacity to grow the operating efficiency of batteries and supercapacitors because of their awesome electric and ionic conductivities [76].

In addition, transition metal dichalcogenides (TMDs) are every other essential sort of dimensional substance, denoted by using the MX₂ formulation which indicates that M is a metallic of the transition metals group including tungsten, molybdenum or titanium, whilst X stands for the chalcogen compounds including sulfur, selenium or tellurium. TMDs are interesting because they can exhibit extraordinary electronic delivery homes, including being semiconducting, metal and even superconductive, depending on the diploma of exchange within the chemical composition and layering of substances [77]. Such assets make TMDs of amazing significance in lots of fields, consisting of but not confined to electronics and optoelectronics, in addition to strength-related technology. They possess remarkable mechanical electricity and optical transparency with without difficulty modifiable electronic houses which has brought about plenty of interest in the fabrication of next-generation electronic devices like transistors, photodetectors and solar cells. Table 2 shows the overview of 2D Materials in EES.

The unique properties of these 2D materials make them excellent candidates for energy storage applications. In batteries, filler graphene and MXenes help to enlarge the charge and ion storage sites and improve the rate performance for fast charging and long-term storage of electricity due to their high aspect ratio and electrical conductivity. In particular, TMDs have also been proven to enhance energy density as well as cycle life in energy-storage devices, driving us towards better battery and supercapacitor systems in future. The search for advanced 2D materials is not only limited by our present technology, but it also offers a prospect of revolutionizing an energy-storage industry that lies ahead. It is within this perspective that the program aims at improving the sustainability aspects. Given the new findings on the characteristics of such substances and integrating that with designing and engineering for better use of these materials, enhancement of the overall efficiency and durability, as well as the performance, of energy storage units is most likely to come with the incorporation of two-dimensional materials in those applications. This table presents a systematic summary of the evolution of *in situ* and *operando* characterization methods for 2D materials in energy storage devices, with the mechanisms, benefits and drawbacks of these techniques specified. The table below shows the Year-wise progress in *In Situ* and *Operando* Characterization of 2D Materials for EES Devices. Table 3 shows the year-wise progress in *In Situ* and *Operando* Characterization of 2D Materials for Electrochemical Energy Storage Devices.

3. Synthesis methods of 2D materials

The process of synthesizing two-dimensional materials is a complex undertaking that requires the cooperation of many areas of knowledge and can be further subdivided into several types: top-down and bottom-up approaches. Nanomaterials are made with bottom-up techniques in which atoms of molecules are used as building blocks. This area has processes such as Solgel, Chemical Vapor Deposition (CVD) and

biosynthesis among others. In bottom-up synthesis, the substrate is of great importance in that it must be compatible with the target 2D material. Sol-gel is one of the most common techniques used for the production of nanomaterials due to its simplicity and versatility in terms of quantity. This is a chemical process where a sol, which is a liquid form of chemicals, transforms into a gel consisting of inter-connected solids. Metal oxides and chlorides are typical examples of these materials. The gel is formed when the feed material is introduced into the host liquid by sonication, shaking or stirring. When it comes to collecting the nanoparticles, techniques such as centrifugation, sedimentation or filtration are utilized. The second one is spinning which forms the nanomaterials directly. For the synthesis of NMs via spinning, a spinning disc reactor (SDR) is used. SDR contains a rotating disk inside a reactor so that its process parameters can be visualized. To purge the oxygen from the chamber, nitrogen gas, or any other inert gas is introduced in the chamber. The water and precursor are added afterwards at varying speeds of rotation. A cluster of core atoms is formed along with their rotation and these are taken out, washed and dried subsequently [43]. The third one is the chemical vapor deposition method which is also a synthesis method that is capable of producing strong and extremely clean NMs. This method also builds NMs by laying down a thin film of gaseous reactants on the surface of a substrate. This process is performed at ambient conditions. During this procedure, a chemical reaction occurs which results in the end product being confined into a thin layer and is coated on the surface of the substrate where it is later. Fig. 2 shows various synthesis and preparation methods for 2D materials.

The 2D materials are epitomized by the top-down approach, which involves production by microfabrication techniques that are primarily aimed at reducing the structural dimensions of the larger structures to the nanometric scale. In the majority of cases, the NMs are fabricated using top-down procedures such as thermal breakdown, sputtering, laser ablation, mechanical milling and nanolithography among others. In the latter, Nanomaterials are also subjected to grinding and post-annealing [86]. Laboratory techniques such as mechanical milling give rise to various types of nanoparticles due to activities like fracture, plastic deformation and cold-welding. Nanoparticles are then patterned using lithography techniques to transfer a desired pattern or structure on the nanoparticles. This attribute renders it a very efficient technique for the production of nanoparticles. This method is more expensive than other approaches and requires advanced equipment [87]. Laser ablation is another synthesis method whereby a laser is used to vaporize a material in a liquid carrier which forms a solid plasma after being treated by a liquid solvent. Laser ablation synthesis in liquid solution (LASIS) has an advantage because it allows the formation of NMs in aqueous and non-aqueous solutions without the use of any chemicals. Due to this quality, the process is environmentally safe. Sputtering on the other hand is a technique that deposits very little NPs on a substrate and subsequent annealing of the substrate. The diameter and shape of 2D materials are subject to the temperature range, type of surfaces and time of annealing [88]. Also known as top-down processes are thermal degradation processes whereby the chemical structure of the material is altered by heat [89]. Nanomaterials are produced as a result of the fracture of the metals at determined temperatures. The NMs are transformed into final products by biological means.

Although many of the technologies discussed above can be employed to fabricate 2D materials, more often than not simple 2D materials fabrication entails PVD, the scotch tape method, or CVD. Massive crystalline structures are instead gathered and used in chips for their specific fabrication purpose as either single or multi-layered samples made by broken micromechanical division. It is possible to perform several exfoliation techniques allowing to manipulation of the area and shape of the separated sheets within certain limits with precision [90]. The primary drawback of this approach stems from the fact that it is quite time consuming and skilled labor is needed to perform it. This method, however, is very well suited for preparing layered 2D materials. This method, for example, has the potential to turn blocks of minerals like

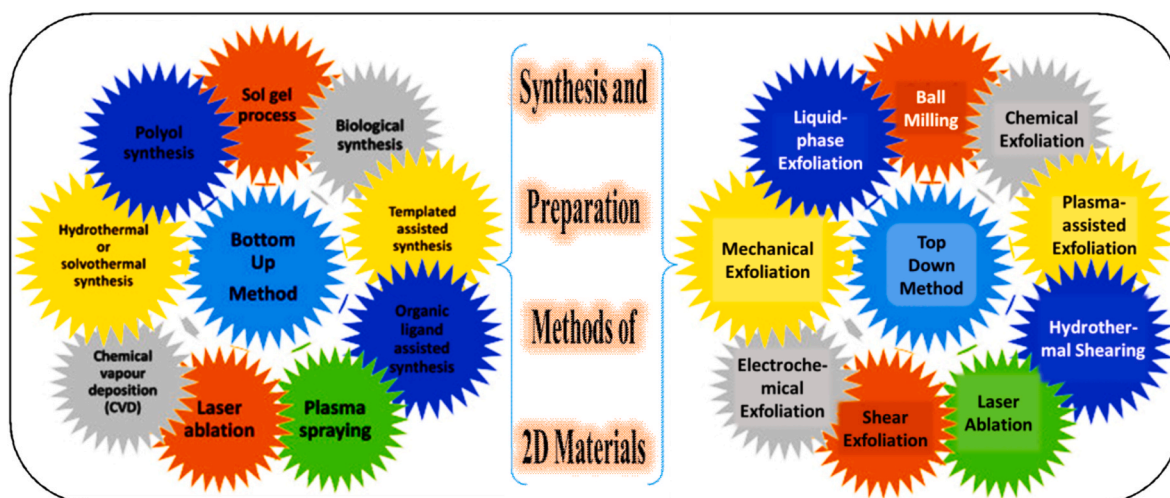
Table 2

Overview of 2D materials in electrochemical energy storage.

Material	Property	Description	Significance	Impact on Energy Storage	References
Graphene	Electrical Conductivity	High intrinsic conductivity	Facilitates fast electron transport	Increases charge/discharge rates	[78]
	Mechanical Strength	High tensile strength	Provides structural stability	Enhances durability of electrodes	[79]
	Surface Area	Large specific surface area (2630 m ² /g)	Higher capacity for ion adsorption	Improves energy storage capacity	[80]
MXenes	Thermal Conductivity	Excellent thermal conductivity	Efficient heat dissipation	Reduces hot spots during operation	[81]
	Chemical Stability	Stable in various chemical environments	Ensures longevity in devices	Maintains performance over time	[44]
	Electrical Conductivity	High electrical conductivity	Enables rapid charge transport	Enhances power density	[35]
	Ion Storage Capacity	High capacity for ion intercalation	Supports high energy density	Improves overall energy storage capacity	[76]
	Hydrophilicity	High affinity for water	Facilitates ion transport	Enhances ionic conductivity	[43]
MoS₂	Mechanical Flexibility	Flexible and robust structure	Enables incorporation into flexible devices	Supports the design of wearable energy storage devices	[50]
	Surface Termination	Varied surface functional groups (e.g., –OH, –O)	Tailors properties for specific applications	Modifies electrochemical behavior	[72]
	Band Gap	Tunable band gap (1.2 eV for bulk to 1.9 eV for monolayer)	Allows for semiconducting properties	Suitable for various electrochemical applications	[26]
	Layer Number Dependence	Properties vary with the number of layers	Tailor performance for specific applications	Impacts energy storage capacity and stability	[43]
	Optical Properties	Strong photoluminescence	Useful in optoelectronic applications	Enhances light absorption in energy devices	[38]
	Electrochemical Activity	High catalytic activity for hydrogen evolution	Enhances energy conversion processes	Useful in energy storage and conversion systems	[25]
	Cycling Stability	Retains performance after multiple cycles	Ensures device reliability	Extends the lifespan of energy storage devices	[71]
Phosphorene	Layered Structure	Buckled honeycomb structure	Unique electronic properties	Enables high-performance applications	[53]
	Electrical Conductivity	Moderate to high conductivity	Allows reasonable charge transport	Affects overall device performance	[41]
	Air Stability	Sensitive to oxidation	Requires protective measures	Limits application in ambient conditions	[37]
	Carrier Mobility	High carrier mobility	Supports fast charge transport	Enhances rate capabilities	[25]
TMDs (e.g., WS₂)	Band Gap Tunability	Band gap changes with layer number	Useful for tailoring electronic properties	Facilitates energy band engineering	[14]
	Band Gap	Indirect in bulk, direct in monolayer	Tunable for specific applications	Suitable for energy storage and optoelectronic devices	[56]
	Catalytic Activity	High performance in electrocatalysis	Boosts energy conversion efficiency	Improves overall energy efficiency	[42]
	Surface Morphology	Varied depending on preparation	Affects ion intercalation and conductivity	Influences energy storage capacity	[29]
	Flexibility	Can be flexible and transparent	Supports flexible electronics	Enables innovative device designs	[22]
Silicene	Thermal Stability	Stable under high temperatures	Suitable for high-temperature applications	Improves performance in demanding environments	[18]
	Electrical Conductivity	Moderate conductivity	Allows for reasonable charge transport	Impacts overall performance	[46]
	Mechanical Properties	Flexible and lightweight	Suitable for diverse applications	Enhances device integration	[32]
	Band Gap	Tunable band gap	Useful for specific electronic applications	Facilitates energy band engineering	[26]
	Chemical Reactivity	Reactive with oxygen	Requires protective measures	Limits practical applications	[58]
Germanene	Stability	Less stable than graphene	May degrade under certain conditions	Affects device longevity	[75]
	Electrical Conductivity	Moderate conductivity	Supports reasonable charge transport	Impacts overall performance	[19]
	Structural Properties	Buckled structure	Unique mechanical properties	Affects flexibility and robustness	[27]
	Band Gap	Tunable band gap	Useful for specific electronic applications	Facilitates energy band engineering	[42]
	Air Stability	Relatively stable in vacuum	Suitable for specific applications	Limits exposure to air	[70]
Antimonene	Photonic Properties	Absorbs visible light	Useful in optoelectronic devices	Enhances energy conversion efficiency	[63]
	Layered Structure	Similar to phosphorene	Unique electronic properties	Suitable for energy storage applications	[41]
	Electrical Conductivity	Moderate conductivity	Allows for reasonable charge transport	Affects overall performance	[82]
	Band Gap	Tunable with number of layers	Useful for specific electronic applications	Facilitates energy band engineering	[83]
	Stability	Sensitive to oxidation	Requires protective measures	Limits application in ambient conditions	[84]
	Mechanical Properties	Flexible and lightweight	Suitable for diverse applications	Enhances device integration	[85]

Table 3Year-wise progress in *In Situ* and *Operando* Characterization of 2D Materials for Electrochemical Energy Storage Devices.

Year	Mechanism Used	Description	Advantages	Limitations	References
2010	<i>In Situ</i> TEM	Observes structural changes in real time.	High spatial resolution; direct observation of atomic-level changes.	Limited to vacuum conditions; sample damage possible.	[48]
2012	<i>Operando</i> XRD	Monitors phase changes during electrochemical cycling.	Provides information on crystal structure and phase transitions.	Requires high-intensity sources; limited temporal resolution.	[34]
2013	<i>Operando</i> Raman Spectroscopy	Analyzes vibrational modes of materials during cycling.	Sensitive to changes in chemical bonding and phase states.	Limited to surface sensitivity; complex spectra interpretation.	[30]
2015	<i>In Situ</i> STM	Allows atomic-scale imaging of surfaces during operation.	High spatial resolution; provides electronic structure information.	Requires ultra-high vacuum conditions; limited to conductive samples.	[17]
2016	<i>In Situ</i> AFM	Measures topographical changes as a function of potential.	High spatial resolution; can operate in liquid environments.	Slower scanning rates; may not provide real-time measurements.	[22]
2018	<i>Operando</i> XAS	Provides information on oxidation states and local environments.	Offers insights into electronic structure during operation.	Requires synchrotron radiation; limited accessibility.	[39]
2019	<i>In Situ</i> FTIR	Monitors surface functional groups during electrochemical reactions.	Sensitive to chemical bonding changes; can be non-destructive.	Limited spatial resolution; surface sensitive only.	[12]
2020	<i>Operando</i> EIS	Assesses charge transfer and diffusion processes in real time.	Non-destructive; provides kinetic parameters.	Complex data interpretation; is limited to certain frequencies.	[21]
2021	<i>In Situ</i> Solid-State NMR Spectroscopy	Investigates local structural changes at the atomic level.	Provides information on local environments and dynamics.	Requires high magnetic fields; limited to specific isotopes.	[26]
2022	<i>Operando</i> Neutron Scattering	Examines the dynamics of materials during cycling.	Provides bulk properties; sensitive to lighter elements.	Limited to large facilities; complex sample preparation.	[74]
2023	Combined <i>In Situ/Operando</i> Techniques	Integration of multiple techniques (e.g., EIS + XRD) for comprehensive analysis.	Offers a holistic view of structure-function relationships.	Complex setup; data integration can be challenging.	[35]
2024	Machine Learning-Assisted Analysis	Uses AI to analyze data from various characterization techniques.	Enhances data interpretation; accelerates material discovery.	Requires large datasets; potential overfitting issues.	[69]
2025	Advanced 4D Electron Microscopy	Captures time-resolved 3D imaging during electrochemical processes.	Real-time visualization of dynamic processes.	Requires sophisticated instrumentation; sample stability concerns.	[47]

**Fig. 2.** Various synthesis and Preparation Methods of 2D Materials.

graphite into thin layers of graphene. Hence, this method is also capable of making very few 2D structures [91]. These solutions can be used in exfoliation techniques where methods using crystalline geometry are not restrictive [92], enabling the process to yield 2D material in plenty. The PVD technique employs a heating apparatus to monitor the process of the films growth on the substrates [93]. PVD incorporates a very clean atomic source and an ultra-high vacuum atmospheric pressure environment to grow elemental 2D materials artificially. There are different approaches to the growth of 2D materials and CVD is one such approach that involves a chemical reaction among gaseous, liquid and solid species confined within a chamber [94]. Every substrate has a distinct action mechanism in this case; substrates with low solubility limit the growth to the formation of a few atomic layers. On the other hand, for the high solubility precursors, much of the substrate that separates when cold goes into solution. The growing substrate carries out the process of segregation utilizing solid-state precursors. A rich precursor sheet is deposited on the surface of the heated substrate, activating either the sublimation or diffusion mechanisms and when 2D materials are

formed, it undergoes condensation to yield the 2D material. Deposition of 2D phase-materials occurs after the condensation of the rich precursor layer. In contrast, the substrate used will be determined by the availability of a precursor component and this happens for instance during the epitaxial growth of graphene on SiC [95]. Thus, some interactions with the substrates are obligatory for the production and growth of all 2D structures [96]. Table 4 shows various synthesis and preparation methods for 2D materials.

4. Functionalization and modification of 2D materials

The functionalization and modification of 2D materials is essential to improve their properties and expand their applications in areas such as energy storage, catalysis, electronics, etc. These materials, including graphene, transition metal dichalcogenides (TMDs), as well as black phosphorus -Covalent and nonsympathetic modifications that are functionalized by various methods can precisely control surface chemistry, enabling tuning of their conductivity, band spacing and reactivity.

Table 4

Various synthesis and Preparation Methods of 2D Materials.

Top-Down Approaches					
Method	Process Description	Advantages	Limitations	Examples of 2D Materials	Ref
Mechanical Exfoliation	Peeling off layers (e.g., Scotch tape method)	Simple, minimal chemical use	Low scalability, irregular flakes	Graphene, MoS ₂	[97]
	Applying shear/pressure to separate layers	Low-cost, rapid	Limited control over flake size	h-BN, Bi ₂ Te ₃	[97]
	Ball-milling exfoliation	High yield, scalable	Introduces defects	Antimonene, Phosphorene	[97]
Liquid-Phase Exfoliation	Ultrasonication in solvents	Scalable, good dispersion	Solvent selection critical, lower yield	Graphene, WS ₂ , BN	[98]
	Shear-force assisted exfoliation	Large-scale, cost-effective	Nonuniform thickness	MoSe ₂ , TiS ₂	[98]
Chemical Exfoliation	Electrolyte-assisted exfoliation	Controlled ion intercalation	Requires expensive equipment	MoS ₂ , NbSe ₂	[98]
	Acid/alkali-based layer separation	High yield, controllable	Chemical residues may remain	Graphene oxide, MXenes	[98]
	Ion intercalation (e.g., lithium)	Degree of exfoliation controllable	Requires post-processing	TaS ₂ , WS ₂	[99]
	Acid treatment (e.g., H ₂ SO ₄)	Simple, effective	Environmental concerns	Phosphorene, h-BN	[99]
Bottom-Up Approaches					
Method	Process Description	Advantages	Limitations	Examples of 2D Materials	Ref
Chemical Vapor Deposition (CVD)	Deposition from vaporized precursors	High-quality films, scalable	Requires high temp & vacuum	Graphene, MoS ₂ , WS ₂	[100]
	Plasma-enhanced CVD (PECVD)	Low-temperature growth	Needs plasma generation equipment	h-BN, MoS ₂ , WS ₂	[100]
Solvothermal/Hydrothermal	Low-pressure CVD	Large-area monolayers	Requires precise control	Graphene, MoSe ₂	[100]
	High-temp/pressure liquid synthesis	Uniform nanostructures, simple setup	Sensitive to conditions	MoS ₂ , WS ₂ , MXenes	[101]
	Organic solvent-based solvothermal growth	Complex nanostructure formation	Requires autoclaves	MoSe ₂ , NbS ₂	[101]
Atomic Layer Deposition (ALD)	Hydrothermal synthesis of hydroxides	Inexpensive, low-energy	Poor size and thickness control	Co(OH) ₂ , Ni(OH) ₂	[101]
	Self-limiting layer-by-layer reactions	Precise control, uniform coating	Slow rate, costly equipment	MoS ₂ , WS ₂	[102]
	Plasma-enhanced ALD (PEALD)	Enables low-temp growth	Requires plasma source	Graphene, MoS ₂	[102]
	Organometallic precursor-based ALD	High-quality films	Expensive precursors	TiS ₂ , NbSe ₂	[97]
Other Advanced Methods					
Method	Process Description	Advantages	Limitations	Examples of 2D Materials	Ref
Electrochemical Methods	Exfoliation/reduction via electrochemical reaction	Energy-efficient, scalable	Limited to conductive materials	MXenes, Graphene	[103]
	Ionic-liquid based exfoliation	High yield, tunable flake size	Voltage control required	Graphene, Graphene oxide	[103]
Wet Chemical Synthesis	Electrochemical oxide deposition	Controlled layer thickness	Limited materials	NiO, MnO ₂	[103]
	Reduction/reaction in solution	Low-cost, scalable	Hard to control uniformity	Graphene oxide, MoS ₂ , Phosphorene	[104]
Molecular Beam Epitaxy (MBE)	LDH synthesis in solution	Low-temp, mild process	Slow reaction rates	Ni–Al LDH, Co–Al LDH	[104]
	Colloidal synthesis of quantum dots	Simple, scalable	Requires post-processing	MoS ₂ QDs, WS ₂ QDs	[104]
Thermal Decomposition	Atomic-level vacuum deposition	High-quality, atomic control	Expensive, not scalable	Graphene, MoSe ₂ , h-BN	[85]
	Hybrid MBE for heterostructures	Complex heterostructures	Needs ultra-high vacuum	Graphene, WS ₂ , MoS ₂	[85]
Flash Joule Heating	Low-energy beam-assisted MBE	Low-temp growth	Limited material options	Phosphorene, Silicene	[85]
	High-temp breakdown of precursors	High-quality monolayers	Needs high temp	Graphene, h-BN	[105]
	SiC decomposition for graphene	Epitaxial monolayers	Substrate limited	Epitaxial Graphene	[105]
Laser-Assisted Methods	Oxide annealing	Tunable film thickness	Requires vacuum annealing	MoS ₂ , WS ₂ , MoSe ₂	[105]
	Rapid carbon conversion	Low-cost, energy-efficient	Quality control needed	Flash graphene, Carbon nanosheets	[106]
Supercritical Fluids	Biomass-to-graphene conversion	Eco-friendly, scalable	Precursor dependent	Graphene, Graphene oxide	[106]
	Joule heating-induced phase transformation	Fast	Limited materials	Metal oxides, MXenes	[106]
Laser-Assisted Methods	Laser exfoliation and reduction	Precise, flexible	Expensive setup	Graphene, MoS ₂	[107]
	Laser reduction of GO	Low-cost, ambient conditions	Nonuniform reduction	Reduced graphene oxide	[107]
Supercritical Fluids	Laser-assisted TMD growth	Direct patterning	Requires laser power control	MoS ₂ , WS ₂ , TiS ₂	[107]
	CO ₂ -based exfoliation	Solvent-free, eco-friendly	Special equipment needed	Graphene, MoS ₂	[108]
Supercritical Fluids	Solvent intercalation via supercritical fluids	High-quality nanosheets	Material compatibility	h-BN, TMDs	[108]
	Supercritical ethanol exfoliation	High-quality graphene	High temp & pressure	Graphene, Graphene oxide	[108]

Covalent functionalization is usually the attachment of functional groups to the surface of a material by chemical bonding, which can introduce defects but increase reactivity non-covalent methods, such as π - π stacking or van der Waals interactions, preserve internal structure so

by modifying the surface modification properties with nanomaterials Hybridization also further improves performance in applications such as catalytic sensing. These modifications allow the fabrication of 2D materials based on multifunctional platforms, such as in biosensors,

supercapacitors and photodevices, increasing their selectivity, stability and conductivity. These modifications in tuning properties highlight their importance as a role in realizing the full potential of 2D objects is emphasized.

4.1. Surface engineering for improved performance

The study of functionalization and modification methods, particularly 2D materials, has attracted much research interest in recent years, owing to their usefulness in a variety of sectors including energy, electronics and sensing. For example, addressed the issue of improving photocatalytic systems via the decoration of 2D nanosheets with adatoms and in this regard emphasized the role of surface modification in enhancing photonic activity into the visible range and in creating internal electric fields favourable for the separation of charge [109]. This served as an introductory basis for further advances in cosmochemistry research directed toward the chemical design of 2D materials, for example studies argued for the very different and very absorbable soft metals called TMD and black phosphorus due to their environmental effects and how they are altered chemically [110]. Fig. 3 depicts various surface engineering for improved performance in 2D materials.

The use of 2D materials in surface-enhanced Raman spectroscopy (SERS) applications, research and its users and explains how these materials amplify the Raman signal, thus rendering the detection possibilities ultra-sensitive [111]. This review highlighted the electronic and optical properties of 2D materials intending to better understand SERS performance enhancement owing to these materials' functionalization. Studies have provided an overview of the functionalization possibilities, especially in the growing area of chemistry of molybdenum disulfide and black phosphorus and discussed how these materials may be tailored or why they cannot be tailored to the atomic level [112]. The example has shown the functionalization approaches that can increase solubility, processability and adjustable deportment of electronic properties but at the same time pointed out the difficulty posed by the fundamental chemical inactivity of 2D materials. Research presented

novel approaches to surface modification, in particular bromomethylation, which made it possible to reliably coat carbon materials with a wide variety of functional groups [113]. Such a way enabled also enhanced surface coverage and opened promising directions towards usage of these materials in applications, like lithium-sulfur batteries. At the same time, work oriented towards controlling on-surface reactions highlighting the ability to engineer complex molecular systems and why one has to care about control at the atomic level when it comes to functionalization [114]. The growing trend of hybridization in 2D nanomaterials also known as the marriage of different functional nanostructures to achieve better results [115]. This includes a detailed look at the positive effects that the combination of functionalized makes on the use of 2D materials for high-end applications. This issue is addressed by looking at electrochemical surface modifications of MoS₂ which showed the prospects of surface properties control at the nanoscale [116]. The results showcased the limitations in depositing uniform molecular layers over 2D TMDCs which continues to be a major challenge for practical use cases.

Work focused on the fabrication of photonic structures for 2D materials and their use on metal dichalcogenide monolayers WSe₂ and MoSe₂ coated with plasmonic metasurfaces addressed [117]. Their efforts detailed the balance of material in design and the external epitaxial environment that concerns the usability of 2D materials. Studies also elaborated on the development of topochemical strategies for 2D materials proposing new even more constructive and innovative synthesis processes that could boost the use of these materials in energy applications. The necessity of theoretical orientations for the investigation of new materials and applications [118]. Janus 2D materials as a novel type of materials that afford 2D design at their surface but have asymmetrical properties as a result of selective functionalization [119]. This has given impetus to new strategies for developing multifunctional materials, which in turn increases the range of possible applications. The issue of the fabrication of structures built of 2D materials, paying particular attention to the cleaning of the interface, the characterization of the materials and their influence on the efficiency of the structures being fabricated in an economically scalable manner [120]. In addition, investigated the usages involving 2D materials in fuel cells, pointing out their merits against 3-dimensional materials used across the board.

4.2. Operando monitoring of functionalization effects

The investigation of functionalization and modification of 2D materials continuously wins understanding and this is because of the materials' special features and possible application in sets such as electronics, energy and even biomedicine. Studies provide the basic principles of the chemical modification of 2D semiconducting TMDCs and black phosphorus however, their sensitivity to the environment and chemicals [168]. The research then expands on this topic by analyzing the issues and progress made regarding the synthesis and modification of 2D layered materials as well as mechanochemical processes in the manufacture of geopolymer (GP) and GP-based composites [121]. All this suggests that synthesis processability plays a critical role in the application. Such considerations include chemical modifications of 2D materials that improve their stability and broaden their functions. In the same framework, studies address the covalent functionalization of graphene derivatives and provides promising new mechanochemical techniques for controlled alteration of structure with defects and functional groups [122]. These techniques can be used to improve the mechanical, optical and electronic properties of graphene, which gives another edge to researchers who are trying to ameliorate 2D materials for desired purposes. The importance of functionalization is monitored in enhancing the stability of 2D materials immersed in ionic solutions as well as tailoring their electronic and optical properties [123]. In this respect, there is a need to look into biocompatibility and toxicity in particular, when addressing applications in flexible bioelectronic devices and wearables which suggests that these studies are applicable in

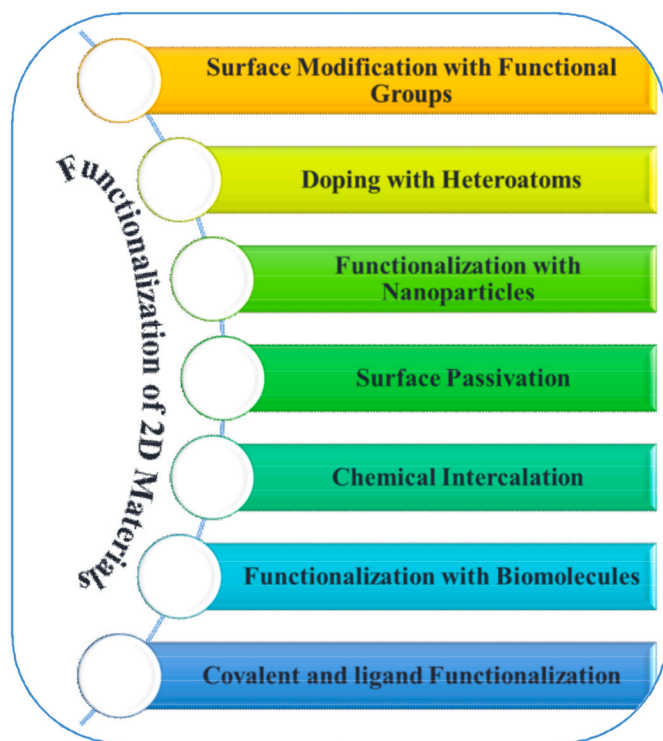


Fig. 3. Different surface engineering for improved performance in 2D materials.

many disciplines. The controllable property modulation in 2D materials is also explained in terms of structural modifications at the atomic scale [124]. The extraordinary atomic structures that could be produced due to lattice development kinetics coupled with temperature-induced vibrations and explain how these structures can greatly transform the electrical, optical and magnetic behaviours of the materials. Fig. 4 represents various operando monitoring of functionalization effects in 2D materials.

The current level of chemical functionalization of graphene, MoS₂, BP and present fundamental problems regarding their use such as insolubility and polydispersity as well as the importance of strong binding and regio-selective functionalization [125]. The current work bears in mind the increasing level of curiosity towards the combinatorial benefits that arise from the use of different 2D materials for nanotechnology and other applications. Research address issues concerning the understanding and microscopic characterization of 2D carbon nanomaterials and the development of their optics to recognize the necessity of accurate assessment techniques as a fundamental prerequisite for any practical implementation of 2D materials [126]. The possibilities of employing topochemical methods in the synthesis of energy-related 2D compounds show wide prospects due to these methods [127]. The challenges they solve in this field as well as the prospects of research

advancements in these areas stratifies the fast-paced evolution of science with such ever needing applications. The Janet 2D materials whose unique functionality is derived from anisotropic functionalization, is wide-ranging and more evidence-based on their production and durability over time [128]. Different integration/fabrication approaches available for 2D materials along with the necessity of techniques such as gate tuning and molecular doping to modify the properties [129]. This operando monitoring of functionalization are necessary for the realization of practical applications, continue to face bottlenecks relating to uniformity and efficiency [130].

5. In Situ and operando characterization: an overview

The realm of electrochemical investigation has tremendously evolved concerning the methods of *in situ* and *operando* characterizations that offer more understanding of the mechanisms of electrochemical activities. Concerning the importance of these techniques, a simple consideration of the issues inherent in normal *ex-situ* methods helps one understand why they are essential in contemporary practice. Researchers also raise several issues related to battery testing in the *ex-situ* mode, explaining that offline electroanalysis is likely to be misinformed regarding the phenomenon of electrochemical cycling. Their

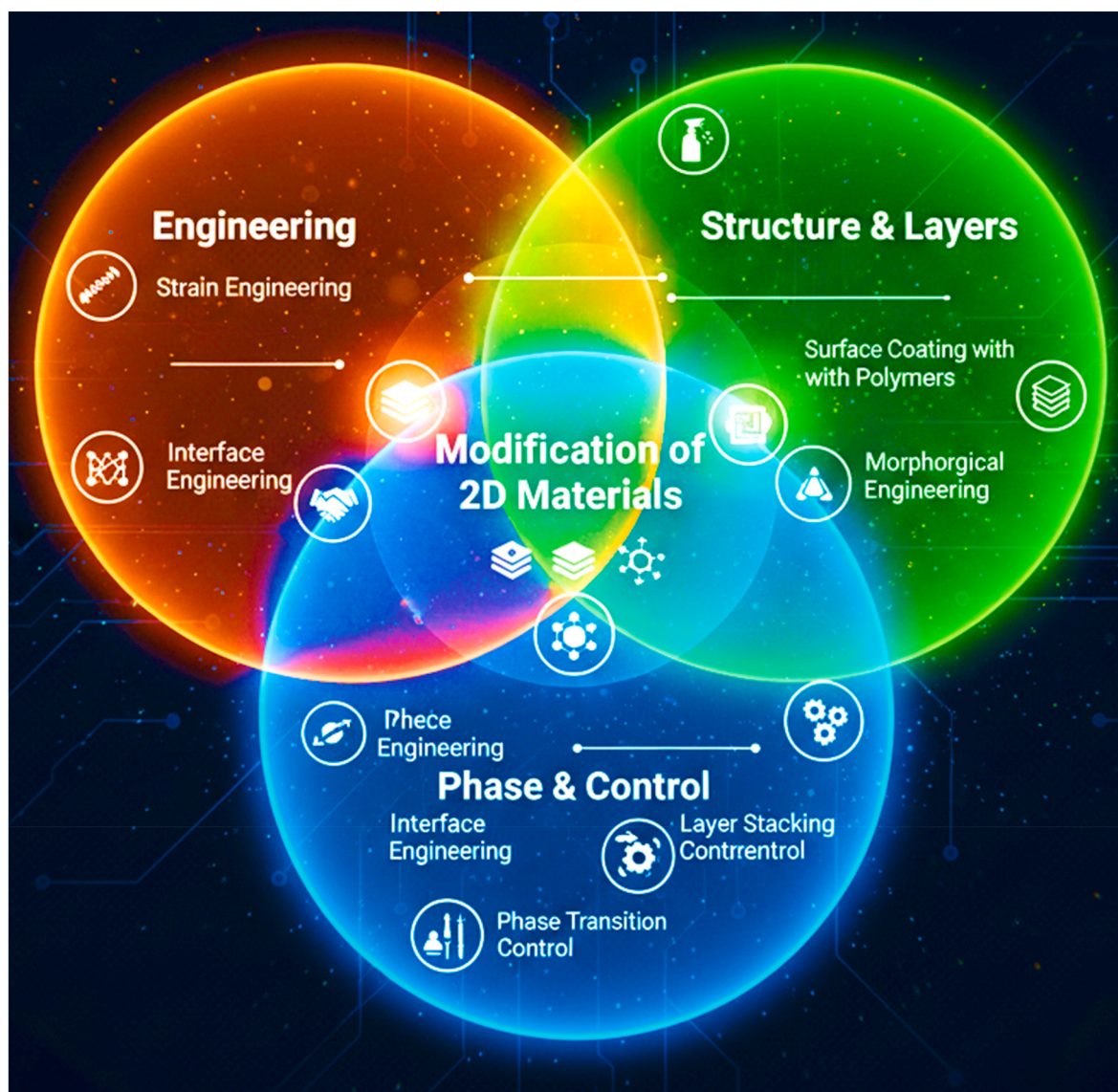


Fig. 4. Operando monitoring of functionalization effects in 2D materials.

emphasis is on the realization of *in situ* and *operando* studies where changes in the structures of electrode materials as a result of charging or discharging cycles can be visualized in real-time. This not only improves the comprehension of the transient species but also improves data quality by allowing the monitoring of one sample under realistic conditions throughout the experiment [131]. Following this, studies consider the problem of solid oxide electrochemical cells (SOCs) and advancements in *in situ* and *operando* characterization techniques, as well. The desire for complicated cell structures capable of replicating normal working conditions is highlighted as essential in understanding electrochemical phenomena. Their review stresses the relevance of understanding the characteristics of electrode materials, especially the inertial losses responsible for geologies where SOC technologies are used [132].

Researchers discuss the challenges associated with the investigation of thin films of electroactive polymers, which are known to undergo reversible morphologies change upon exposure to electric stimuli. They propose a different approach that allows for high-quality *in-situ* measurements by distancing the incoming and outgoing X-ray beams from the electrolyte. This development is important because electrochemical transistors (OECTs) are built using conducting polymers that can act as electronic and ionic conductors and their understanding under working conditions is very important. Studies suggests different ways of enhancing the sensitivity to structural changes, envisioning the next steps in the study of the operating conditions of the materials in question to increase the dissemination of EIS within the electrochemical society, they describe and explain some of the jargon and interpretation coming with EIS for the benefit of novice users. In the general subject discusses the state of the art in electrochemical imaging systems particularly concerning optical systems that can interrogate structural electrochemistry in real time, points out that scanning electrochemical probe microscopies in contradiction to other scanning techniques have been capable of identifying local current non-uniformities, these techniques suffer from low throughput nondestructive imaging. Yet, the use of optical methods has emerged as a good substitute for imaging with better resolution and thus deeper understanding of electrochemical processes [133]. The analysis of the progress of sodium-ion batteries demonstrates that *in situ* and *operando* characterization techniques are critical to comprehending the electrochemical reaction mechanisms in these systems. Their compositional arguments are based on the necessity of employing multimodal characterization techniques to correlate the effects of the composition, microstructure and physiological morphologies of the electrode materials on charge/discharge cycle behaviour. Such findings are necessary to enhance the performance of SIBs as their research is becoming more prevalent due to their potential replacement of lithium-ion batteries [134].

The *operando* operational conditions in electrochemical devices explicate the differences between *ex-situ*, *in situ* and *operando* measurements and emphasize the merits of using the *operando* approach concerning the evaluation of performance issues and processes related to failure in all types of storage energy devices. This paper integrates principles and practices related to and surrounding nondestructive evaluation of battery components in their working state, thus helping in the development of the discipline [135]. The vibrational sum-frequency generation (VSFG) spectroscopy can be utilized in an *operando* approach for the investigation of electrochemical interfaces. They describe the first application of VSFG in conjunction with electrochemistry to determine the molecular behaviour at an interphase and discuss its power to study molecular catalysts. The difficulty that exists in dealing with the data from such fluid systems cannot go unrecognized, however, it is clear that the potential of the technique of VSFG in the study of interfacial phenomena is immense [136]. Studies also assess the possibilities of electron paramagnetic resonance (EPR) spectroscopy in the study of solid-state electrocatalysts. They describe the problems and aspects of EPR application for *in situ* and *operando* studies, with emphasis on the stability of electrocatalytic materials under different

kinds of electrolytic environments. Their comments on the influence of transition metal oxidation states in catalysis using these materials stress the importance of material and cell designs in considering future work [137].

The role of *operando* electron microscopy in the study of heterogeneous catalysis includes the main principles and aspects of *operando* electron microscopy to help achieve a better scientific comprehension of catalytic reactions [138]. Summarizing all these different studies and contributions, it can be stated that *in situ*, *operando* characterization techniques are paramount in understanding electrochemical processes in all systems. The progress made in applying such techniques will, without doubt, aid in the improvement of materials and devices that facilitate energy storage and conversion. The Table 5 below is a comprehensive comparison of *in situ* and *operando* techniques in 2D materials used for supercapacitors and batteries.

6. Electrochemical characterization techniques

Electrochemical characterization techniques are needed to evaluate the performance of 2D materials, especially for applications in energy storage and catalytic sensing of electrochemical behavior of materials CV is often used for analysis. It measures the current response as power spans with defined limits, allows the identification of redox reactions and the charge storage capacity CV is particularly valuable for understanding the pseudocapacitive behavior of materials in supercapacitor batteries. EIS is another important tool, providing information on charge transfer resistance, ion diffusion and interface processes EIS helps evaluate conductivity and understand how material structure affects electron and ion transport. GCD is used to determine the specific capacitance, energy density and power density of two-dimensional materials. This technique applies a constant current to a system and monitors the voltage response, providing valuable data on the energy storage capacity of the material. These approaches contribute to the optimization of 2D materials for various electrochemical applications by clarifying their performance parameters and mechanisms.

6.1. Electrochemical impedance spectroscopy

The field of electrochemical characterization techniques has become more sophisticated, with special emphasis on *in situ* and *operando* microscopy techniques that are crucial to the study of complicated electrochemical processes. Researchers studied electrochemical scanned probe microscopies and various other techniques such as scanning ion conductance microscopy and scanning electrochemical microscopy. A multi-technique electrochemical scanned probe microscope to enhance spatial resolution and image materials activity, including highly ordered pyrolytic graphite (HOPG) is studied [159]. To these basic technologies, research added an apparatus for electrochemical studies based on PC and LabVIEW which included in particular electrochemical impedance spectroscopy and other types of measurements [160]. This device greatly increased the user-friendliness of electrochemical characterization allowing it to use it for many more applications in research and development. The authors stated that this technique is essential when dealing with systems with high capacitance values, especially with the increasing use of electrochemical supercapacitors.

Ionic diffusion occurring locally through electrochemical strain microscopy (ESM), addressing its similarities with traditional methods such as EIS is examined [161]. Their research brought about the realization of the need to carry out such studies with good spatial resolution concerning the ionic mobility at the nanoscale considering its significance in the manufacture of battery composites. They articulated how strains and ionic currents interacted with each other thus enhancing the understanding of the electrochemical processes. After that, *in-situ* operable methods using scanning electrochemical probe microscopy (SECCM) allowing surface reactivity to be mapped in real-time at the nanoscale [162]. It illustrated how such a technique could determine

Table 5Comparison of *in situ* and *operando* techniques in 2D materials used for supercapacitors and batteries.

2D Material	Device Type	<i>In Situ</i> Technique	<i>Operando</i> Technique	Key Observations (<i>In Situ</i>)	Key Observations (<i>Operando</i>)	Ref
Graphene	Supercapacitor	XRD	Raman Spectroscopy	Structural phase transitions	Real-time ion intercalation	[139]
MXenes (Ti₃C₂)	Supercapacitor	SEM	Electrochemical Impedance	Layer expansion during ion insertion	Real-time conductivity analysis	[140]
MoS₂	Battery	XAS	X-ray Diffraction (XRD)	Electronic structure changes	Phase evolution during cycling	[141]
Phosphorene	Battery	STM	Raman Spectroscopy	Atomic structure observation	Electrochemical reaction monitoring	[142]
Black Phosphorus	Supercapacitor	TEM	Electrochemical Quartz Crystal Microbalance (EQCM)	Surface modifications under charge/discharge	Mass transfer dynamics	[143]
Antimonene	Battery	XPS	<i>In Situ</i> Neutron Scattering	Observation of surface oxidation	Bulk ion diffusion monitoring	[144]
WS₂	Supercapacitor	AFM	Raman Spectroscopy	Surface roughness evolution	Lattice strain during charge/discharge	[145]
SnS₂	Battery	XRD	XANES (X-ray Absorption Near Edge Spectroscopy)	Structural evolution during cycling	Transition metal oxidation states	[146]
MoSe₂	Battery	Raman Spectroscopy	<i>In Situ</i> TEM	Lattice stress and deformation under cycling	Phase change and intercalation dynamics	[147]
Ni(OH)₂	Supercapacitor	TEM	<i>In Situ</i> Electrochemical Impedance	Layered material expansion	Conductivity shifts and impedance changes	[148]
MoTe₂	Battery	Neutron Scattering	Raman Spectroscopy	Stress-induced phase changes	Real-time conductivity variations	[149]
Graphene Oxide (GO)	Supercapacitor	XAS	<i>In Situ</i> AFM	Surface chemical composition changes	Surface roughness and adhesion monitoring	[150]
TiS₂	Battery	Synchrotron Radiation	XRD	Ionic diffusion pathway visualization	Intercalation dynamics	[151]
SnSe₂	Battery	TEM	XRD	Morphology changes	Structural transformations during cycling	[152]
VS₂	Battery	XPS	Raman Spectroscopy	Surface chemical state analysis	Real-time phase transition observation	[153]
NbSe₂	Supercapacitor	Synchrotron Radiation	EQCM	Layer interaction under stress	Mass and surface chemical changes	[154]
Ti₂C₃Tx MXenes	Supercapacitor	Electron Microscopy	Electrochemical Impedance	Morphological changes during cycling	Real-time ion transfer dynamics	[155]
ReS₂	Supercapacitor	Neutron Diffraction	XRD	Crystal structure under load	Structural transitions during charge/discharge	[156]
NiSe₂	Battery	XPS	Raman Spectroscopy	Oxidation state changes during electrochemical reactions	Phase transition and ion intercalation monitoring	[157]
WS₂	Supercapacitor	AFM	<i>In Situ</i> Raman Spectroscopy	Surface morphology analysis	Dynamic charge transfer study	[158]

low values of current and for the work differentiate between two identical material types throughout their shapes at a nanoscale. It was the application of SECCM alongside normal electrochemistry that allowed an appreciation of material and electrochemical correlations from a different perspective. The essence of EIS expansion about its application over an extensive ion polarization range [163]. This was evident as his approach stressed the significance of continuous impedance spectra at different high and low potentials leading to a better understanding of electrochemical processes, especially corrosion. This strategy contributed to an enhanced understanding of the data interpretation and EIS tolerances.

The possibility to research electrospray ionization as a non-chemical masking technique for mass spectrometry in combination with electrochemistry was elaborated where the author noticed the benefits of coupling mass spectrometry with electrochemistry for visualizing redox processes [164]. The possibility of evaluating the non-heavily sustained volatile intermediates and products of such hybrid systems helped in the further understanding of complex electrochemistry. The importance of EIS in the analysis of electrochemical systems, determining, for example, charge transfer resistance and mass transport limitations [165]. The possibility of performing EIS under *in situ* and *operando* conditions has enabled real-time measurements of systems like batteries and fuel cells, which further established the importance of this approach in modern electrochemical studies.

The improvement of electrical measurements at the nanometer scale in applications done in liquids [166]. Here, the comprehension of the electric double layer at solid-liquid boundaries was made its key concern. The performance of several electrochemical devices and materials depends on the relationships that demand the application of local characterization techniques. The application of EIS to several and more fields, including biological systems, was expanded which described EIS

application in biosensing and disease diagnostics [167]. Their review attested to the elasticity of EIS, proving its usefulness in far-range applications including cell studies and health systems. The constructive criticism of the field of materials chemistry where EIS is applied and stated that it is particularly relevant in the studies of conductive composites and understanding of the compositional changes [168] as shown in Fig. 5(d–f). They stressed a deeper appreciation of EIS results, especially because of new materials emerging. The influence of optical microscopy on the field of electrochemistry and further explained how the two fields can be made to work together through improved imaging techniques in electrochemistry [169]. This allows for a greater insight into the electrochemical processes at play and in particular the diversity present in such systems [170].

6.2. Cyclic voltammetry

CV in the framework of *in situ* and *operando* investigation of 2D materials for devices intended for electrochemical energy storage presents an ever-evolving landscape of knowledge around a widely esteemed technique and its variations. A stage for understanding CV as a technique and proposes an extensive model of dealing with CV with ion transport and Faradaic reactions in electrolytes of different kinds [171]. Following this strategy, which does not rely on certain aspects concerning double layers of electric charges and constants of reactions, more delicate explanations of the outcomes of CV experiments in various systems arise naturally. Finishing that context, ‘Scanning Ion Conductance Microscopy’ scans in which CV is employed to image ion concentration at an interface. This development is paramount in elucidating the properties of 2D materials within electrochemical systems with relation to the surfaces of the 2D materials and the transport of charge within the systems [172].

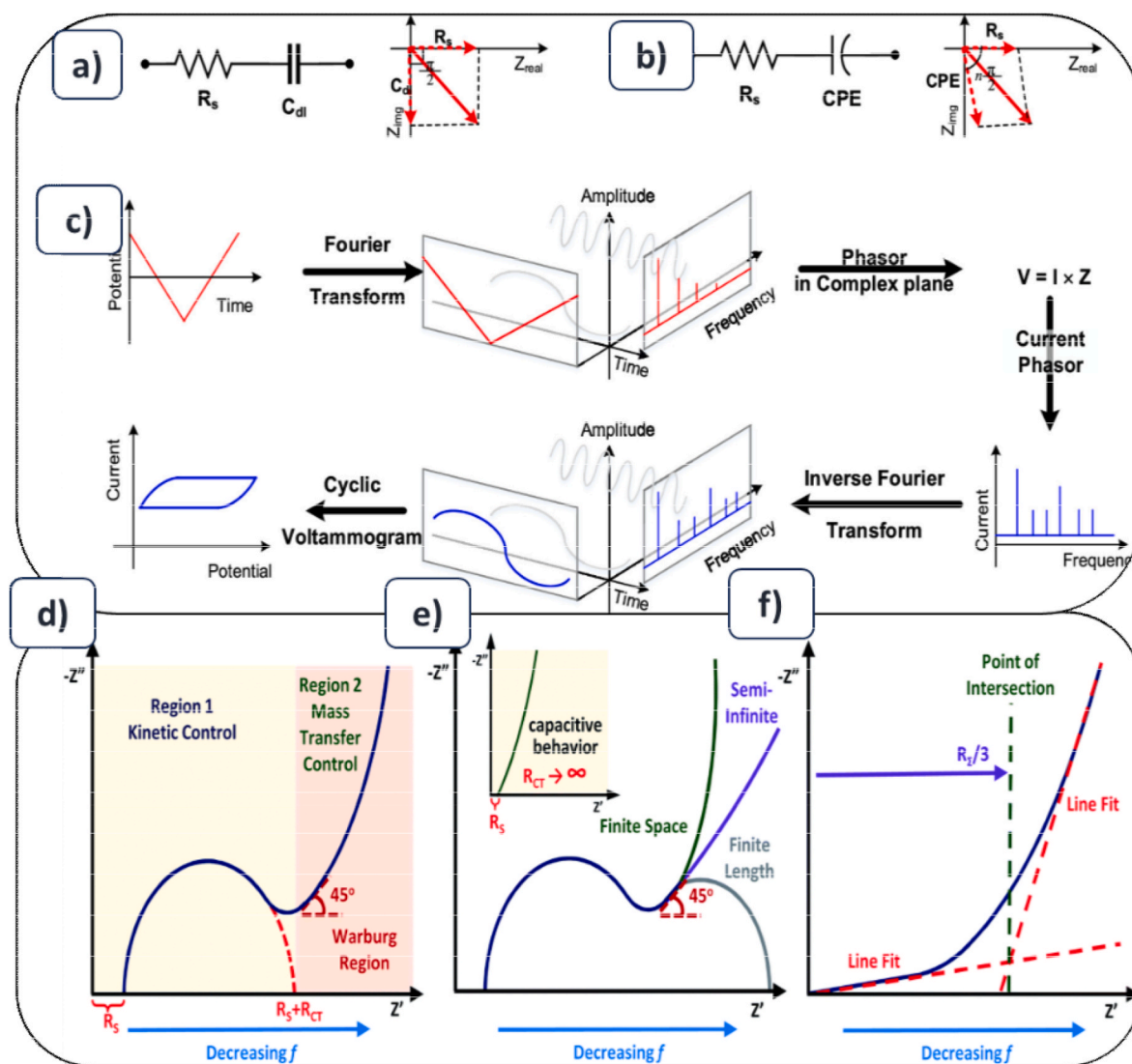


Fig. 5. Equivalent circuits are composed of solution resistance and either (a) conventional double-layer capacitance or (b) constant-phase element (CPE) with corresponding phasor diagrams. (c) Procedures for predicting cyclic voltammogram by the Fourier transform. Reproduced with permission from Ref. [173]. (d–f) Reducing the resistance for the use of electrochemical impedance spectroscopy analysis in materials chemistry. Reproduced with permission from Ref. [174].

Cyclic voltammetry is an invaluable approach in the assessment of redox fitness in which the aspect of reaction mechanisms and electrochemical kinetics is well treated [175]. This study focuses on the importance of scan rates and diffusion layer dynamics that are critical towards the enhancement of energy storage electrode materials. A new approach to the study of two-dimensional materials applied for electrochemical energy storage with the introduction of several other layered materials apart from graphene is also discussed [176]. The capabilities of these materials in terms of their electrochemical performance show the significance of CV in determining their feasibility to work as electrodes, thus linking material attributes with functional device performance. The work done by CV by combining it with other techniques to perform electrochemical spectroscopy analysis of a class of materials and their compounds [177]. This combination deepens the understanding of the processes of charge transport and transport in devices based on organic materials and makes clear the necessity of CV in the design of organic electronics, which may be used alongside 2D materials for energy storage systems. A general approach for Macro-electrode CV simulation, which deals with the issues of various electrode designs and diffusion domain systems. However, it focuses mainly on the analysis of CV charts, which are very important for the evaluation of new

materials employed for energy storage devices and technologies [178]. Further discussion by looking into potential dangers that may affect the quality of CV practices, especially, double-layer capacitance [179]. Their seven-step protocol serves as a roadmap for researchers, ensuring that CV measurements are done systematically to collect useful information. Also, charging current in CV and correlating it with the performance of supercapacitors [180]. The results also show that there exists a delicate interplay between the scan rates and the dynamics of charging, which is critical in the functioning of energy storage devices, as shown in Fig. 5(a–c). Also, operando approaches to investigate battery electrodes and focus on the necessity of monitoring electrochemical processes in real time. Their debate regarding *in situ* and *operando* measurements underlines the importance of developing techniques for in-operando materials: real-time measurement of dynamic behaviour within as well as outside the material's confines [181]. A comparative analysis addressing the interpolation of cyclic voltammograms using *ab initio* techniques which is consistent with the understanding that CFD technique should be employed in the study of electrocatalysis. The process of designing and perfecting structures targeted at energy storage systems, basing it on the mutual dependence of theoretical CV, experimental CV and materials development data.

6.3. Galvanostatic charge-discharge

The use of GCD methods in the *in situ* and *operando* approaches has gained considerable importance in the field of energy storage, especially for batteries and supercapacitors. Many studies in the existing literature stress the significance of these methods in deciphering the complex mechanisms of electrochemical performance. The invention which includes an application of nanoscale imaging to study the phenomena of Li-ion distribution in the battery electrode and electrolyte systems while they are operating [182]. They also pointed out the need to observe the kinetics of ion transfer in real time saying that charging is not uniform across the particles. Certain studies criticized the application of redox electrolytes in supercapacitors, focusing on some important aspects like the definitions of capacitance and energy storage capacities [183]. They stated that the collaboration between supercapacitors and batteries, called ‘superpattery’, raised significant challenges concerning the classification of charge storage mechanisms especially concerning GCD measurements. This critique further explained the challenges that may be in engaging with the evaluation of energy storage performance, thus the strong stressing of the need for better methods of analysis. Further progress in this domain performed spatially resolved ionic measurements through SEM a technique that allowed the ionic fluxes in non-aqueous systems to be measured in a very accurate manner thus enlightening the energy storage interfaces even more [184]. In 2018, the range of analytical tools for the analysis of the materials of electrochemical capacitors [185]. Their review addressed several *in situ* methods, such as X-ray photoelectron microscopy and Raman microscopy, which are crucial to GCD processes. With an awareness of ion segmentation and alteration of structure during use, advances in performance prediction and enhancement of energy storage devices become achievable.

Gita B. Bhanuse and co-workers [186] demonstrated the fabrication of a heterostructured ZnCo_2O_4 @Ni-MOF electrode for application in asymmetric supercapacitors. The incorporation of Ni-MOF onto ZnCo_2O_4 nanostructures was designed to enhance electrochemical activity by combining the high conductivity and redox-active nature of ZnCo_2O_4 with the large surface area and porous architecture of the Ni-MOF. The electrochemical performance was systematically evaluated using GCD measurements conducted at a current density of 2 A g^{-1} for all electrodes (Fig. 6(a)). Among the tested materials, the ZnCo_2O_4 @Ni-MOF heterostructure exhibited the longest discharge time and superior electrochemical behavior, achieving an ultrahigh specific capacitance of 1800 F g^{-1} , which corresponds to a specific capacity of 200 mAh g^{-1} . For comparison, the pristine ZnCo_2O_4 electrode delivered a capacitance of 1128 F g^{-1} (125 mAh g^{-1}), while the pure Ni-MOF electrode showed a lower capacitance of 850 F g^{-1} (93.3 mAh g^{-1}). These results clearly highlight the synergistic effect of the hybrid architecture, where the hierarchical porous structure and efficient electron/ion transport pathways significantly improved charge storage capacity.

Abdulkadeem Sanni and colleagues [187] reported the design of a hair-derived carbon (HHC)-supported Ag/Ni-MnO_x composite electrode to elucidate the synergistic redox contribution of $\text{Mn}^{2+}/\text{Mn}^{3+}$ and $\text{Ni}^0/\text{Ni}^{2+}$ couples for high-performance supercapacitors. The hierarchical carbon matrix derived from human hair provided a conductive framework with high surface area, while the incorporation of Ag, Ni, and MnO_x nanoparticles offered abundant electroactive sites for reversible redox reactions. The electrochemical performance was evaluated through GCD measurements at current densities ranging from 1 to 10 A g^{-1} . The pristine HHC electrode exhibited nearly symmetric triangular GCD profiles, indicative of excellent capacitive behavior, with a stable specific capacitance of 271 F g^{-1} at 1 A g^{-1} . For the composite electrodes, the GCD curves showed distinct two-stage discharge characteristics, reflecting the presence of multiple faradaic redox transitions. Specifically, the $\text{Mn}^{2+}/\text{Mn}^{3+}$ and $\text{Ni}^0/\text{Ni}^{2+}$ redox pairs actively participated in the charge-discharge process, thereby enhancing pseudocapacitive performance. The representative GCD plots include: (6b) the pristine HHC electrode, which maintains a triangular profile; (6c) the Ag/NiO_x@Mn₂O₃ composite, showing dual redox plateaus; and (6d) the carbon-supported Ag/NiO_x@Mn₂O₃ electrode (C-Ag/NiO_x@Mn₂O₃), which combines capacitive and pseudocapacitive contributions for improved energy storage capability.

The research of also added a methodological aspect by providing a seven-step concrete protocol addressing the reliability of cyclic voltammetry measures, stating the need for proper estimation of double-layer capacitance. This work has practical importance in the characterization of energy storage materials but also demonstrates the wider relevance of these voltammetric techniques in the analysis of GCD. Works also scrutinized the cycled voltammetry towards charging current and its application concerning the supercapacitor GCD profile. The outcomes of their research clarified the issues of galvanostatic measurements, particularly the need to consider frequency-dependent components for a proper representation of the electrochemical phenomena. This study enhanced the understanding of advanced GCD profile analysis with the help of advanced circuit models. The GCD performance of the nanoporous supercapacitors and deriving an equation that relates the current to the time of discharge and the capacitance. They further demonstrated that there was a great need to consider dimensionless numbers when looking at the data from experiments in this area since it relates to coming up with better energy storage systems. Different studies then proceeded to study the *operando* methods and especially focused on using X-ray diffraction to study the changes in battery material's microstructure when they are cycled [188]. They asserted that it is critical to appreciate and capture the changes that happen in screen discharges at intermediate times which further bolstered their argument on the *in-situ* imaging techniques when it comes to explaining the discharge processes in batteries, as shown in Fig. 7(a–g). In 2023, introduced novel techniques by combining the visualization of electrochemical processes in 3D by employing electrochemical transmission microscopy with mass spectrometry [189,190].

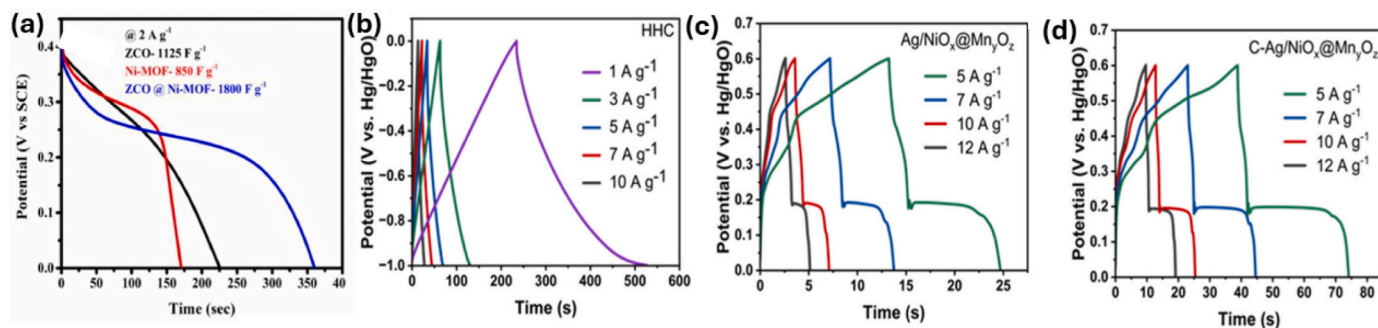


Fig. 6. GCD measurements conducted at a current density of 2 A g^{-1} for (a) ZnCo_2O_4 @Ni-MOF electrode [186], (b) HHC electrode with triangular symmetry, (c) Ag/NiO_x@Mn₂O₃ showing dual redox transitions and, (d) C-Ag/NiO_x@Mn₂O₃ with enhanced charge storage [187].

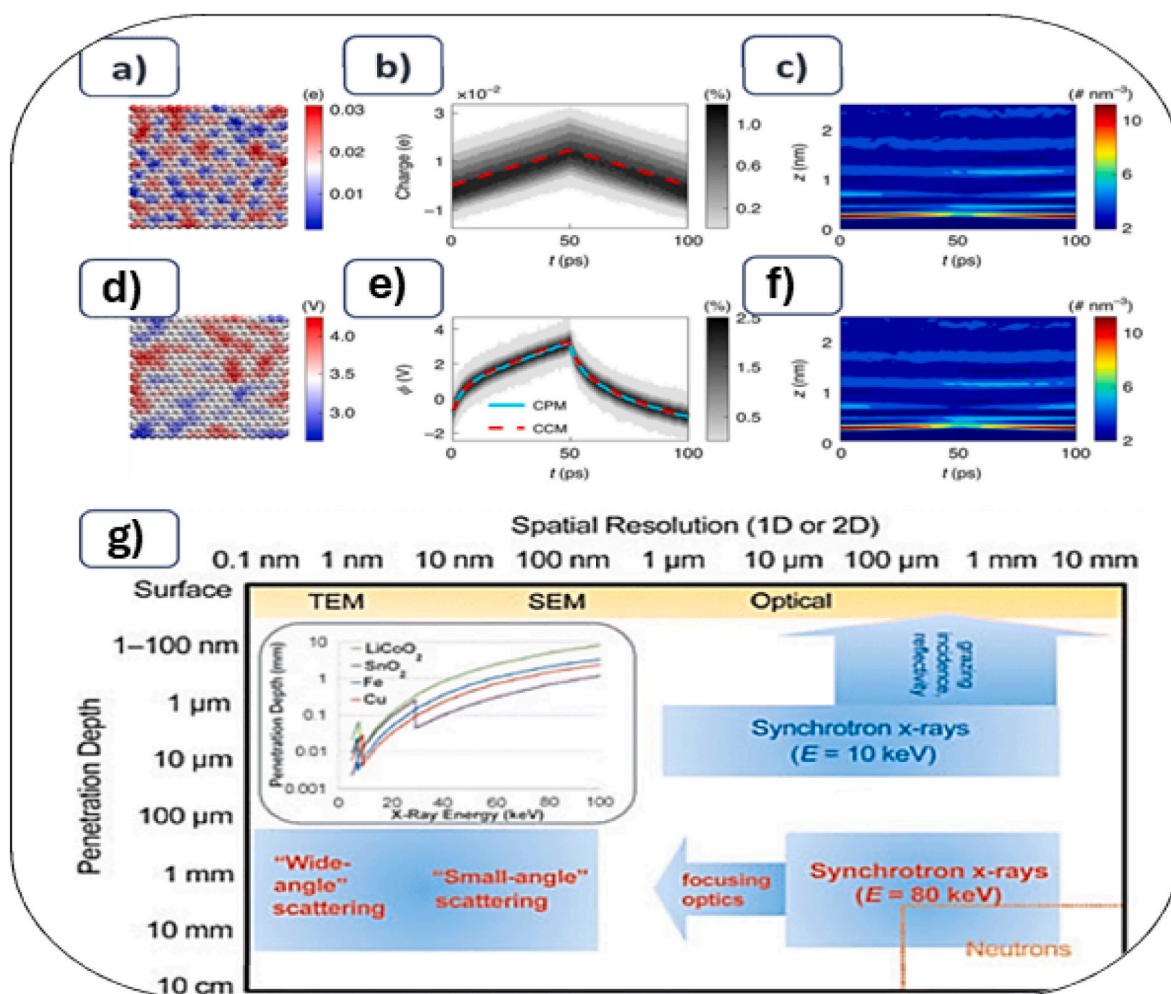


Fig. 7. (a–c) GCD-CPM modelling (a) the probability distributions of electrode charges varying with time (grey contour) and their average (dashed line) (b) evolution of ion number density with time (t) and distance (z) from the electrode (c). (d–f) GCD-CCM modelling (d) the probability distributions of the electrode potentials (grey contour) and their average (red dashed line) (e); evolution of ion number density (f,g). Reproduced with permission from Ref. [197]. (g) Typical penetration depth and spatial resolution (1D and 2D measurements) for selected analytical techniques. The inset depicts 1/e penetration depths for various battery relevant materials as a function of energy (E), where e is Euler's number. Reproduced with permission from Ref. [198]. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

Their experiments on the formation of the SEI layers after GCD processes, pointed out severe drawbacks of such systems and their performance, stressing the need for a more pronounced understanding and tooling for the improvement of battery materials. Not least, the Capacitors review, studies examined features of a supercapacitor construction emphasizing the dependence of electrode scan rates on capacitance and the consequences of this in GCD performance [191]. Their work emphasized once more how important GCD was for the measurement of the performance of supercapacitors, particularly concerning their performance in stability and energy density.

6.4. Differential capacity analysis

The enhancement pulse characteristics of the studied stress-strain diagrams of compression and tension show that the prolongation of the grain structure Bell has a minor effect on the *in situ* KIC values obtained with such U-shaped notched specimens even SMAs GZ T25–80. 28 variations of shape and size of the thermal loads did not influence the fracture toughness values KIC measured under *in situ* conditions as the interventions had been applied. This is because the tested shapes and sizes of the thermal loads could not change the average interlaminar spacing of the composite materials in the test orientation. This may be

because the stress levels applied to the various shapes and sizes of the thermal loads in the tests exceeded the inter-laminar connection of the composite materials under the test orientation. However, it can be inferred that while there was a change in the sectional properties of the RTM DCB specimen laminate during these tests, the qualitative differences in fracture mechanisms or interfaces present from one shape to the other are why the axial stiffening lengths play. This study stressed the usefulness of electroanalytical methods in enabling the measurement of ionic fluxes at energy storage interfaces which aids in understanding how the electronic characteristics and the space measured changed during the battery cycling. Various studies added to the existing capabilities for the analytical characterization of materials used for electrochemical capacitors [192–194]. In their review, the authors discussed the application of *in situ* XPS and PEMS to analyze the carbon/electrolyte interface and address important ion confinement and ageing phenomena related to the development of energy storage systems. The advances in the form and composition of nanoscale materials in lithium-ion batteries and how those differences in charge storage mechanisms can lead to gains in energy and power density [195]. This paper depicted how faradaic and non-faradaic processes co-exist within certain electrode materials and the consequences this has on their electrochemical activity. Solid oxide electrochemical cell advancements in the *in situ* and

operando characterization techniques were the object of the review [196]. In this regard, their study underscored the need to observe the processes of electrochemical action taking place within real-world conditions of their performance working towards understanding, in particular, the local defect chemistry to optimize the performance of cells.

Some studies aimed at the use of synchrotron methods to understand how the structural and chemical characteristics of lithium-ion battery layered oxide cathodes evolve [199]. It is possible to analyze how lattice structure and electronic structure change during battery cycling due to the advanced synchrotron methods employed to get such crucial information on the behaviour of materials. The performance limitations of sodium-ion batteries especially the deterioration and capacity loss of the cells over time is also examined [200]. Their review of the *in situ* and *operando* characterization techniques available for SIBs also determined the significance of phase and morphology changes that accompany charge/discharge cycling in enhancing SIBs performance. Studies continued this trend, reporting structural change and impedance studies in battery cells using *in situ* XRD techniques [201]. It was shown that *in situ* and *ex-situ* approaches are useful for understanding battery mechanisms, particularly in studying processes that take place for a short time only and are usually lost in *ex-situ* examinations. Finally, the *operando* synchrotron radiation-based techniques value in mature and next-generation battery systems [202]. Together these studies provide a strong basis for understanding the electrochemical process that takes place in battery systems pointing out the need for *in-situ* and *operando* techniques.

7. Advances in *In Situ* and *operando* characterization techniques

An immense change in the perspective of characterization of materials on the available variety of bulk, surface and interface characterization techniques also known as hyphenated techniques [203]. The authors specifically discuss commercially available instruments indicating that such advanced instruments can easily be accessed by

undergraduates and fresh graduates at a level thus bringing the two levels of knowledge, theory in class and practice in well well-equipped laboratory, closer to each other. The identification of several levels of microstructural characterization with special emphasis given to the computer vision and machine learning components [204]. They explain how these data science methods make it easier to tackle one of the most obvious problems associated with the quantification of microstructures – feature extraction from microstructural images but at the same time CV and ML can offer tools to transform how microstructures are measured and analyzed. Likewise, certain research looks into the promises of machine learning as it pertains to material characterization with a focus on mechanical properties predictions. They refer to the past in which experimental studies were carried out before the advent of computational materials science, outlining the critical concern in this field, controlling high dimensional space. The discussion to spectroscopy within the emerging trend of additive manufacturing in understanding the structure and interaction of molecules [205]. Studies report on the emerging application of SEM innovations for the multiphysical materials characterization of nanomaterials in real time. The role of nanorobots in SEM is explained, which allows for the integration of *in situ* imaging and measurement of nanostructures, as shown in Fig. 8 [206]. In this regard, the potential that advanced imaging and manipulation techniques can also articulating the barriers that currently exist due to optical integration issues. Table 6 depicts different *In Situ* and *Operando* Characterization Techniques.

8. *Operando* studies in real-time device applications

There has been a growing interest in practical *operando* studies within the field of EES, especially in batteries and supercapacitors. The recent technologies are useful for understanding how devices perform under operational conditions from a mechanistic perspective. Researchers studied the possibility of revealing the lithiation of anode materials inside the battery on a nanoscale imaging lithium-ion distribution within electrochemical processes occurring in batteries [208].

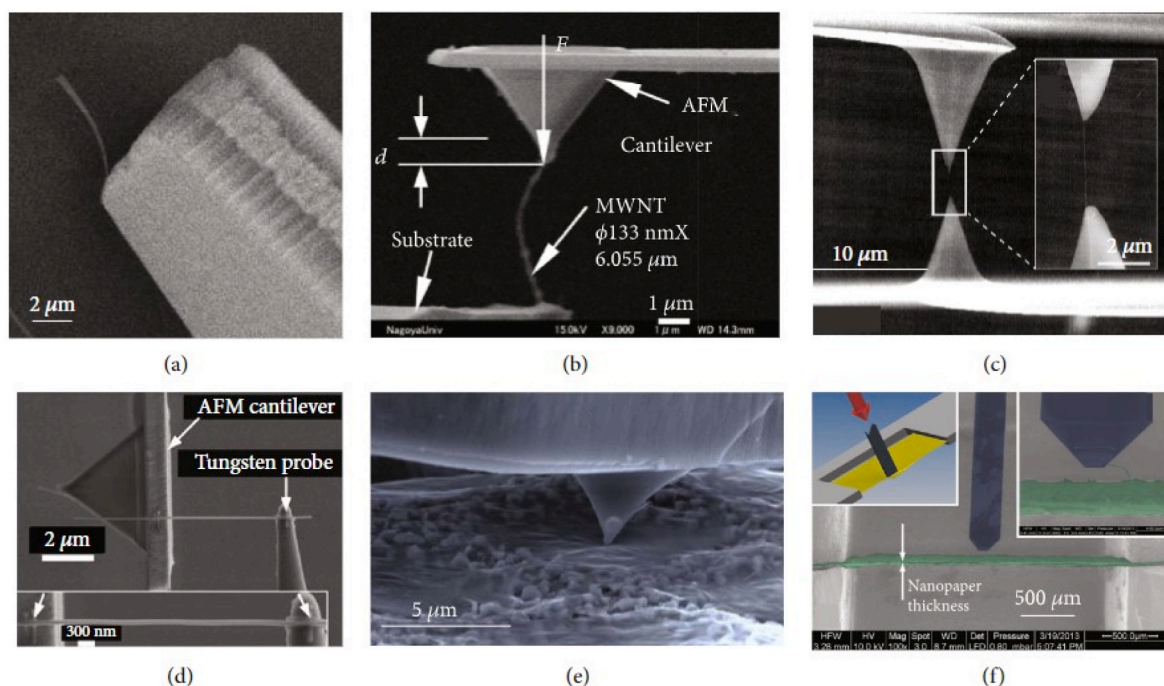


Fig. 8. SEM-based *in situ* mechanical characterization of nanomaterials. (a) Using a piezoresistive atomic force microscope (AFM), (b) Using a buckling test to mechanically characterize a nanotube *in situ*, (c) Individual MCNT tensile test; the enlarged image of the MCNT under tensile stress. (d) Tensile test of a single Ag NW; the high-resolution SEM image of the NW for strain measurement, (e) While detecting the acting forces, the AFM probe is analyzing the graphene flakes. (f) Nanopaper measurement scene inside SEM: the nanopaper is stretched. Reproduced with permission from Ref. [207].

Table 6Different *In Situ* and *Operando* characterization techniques.

Type	Characterization Technique	Mechanism	Parameter Measured	Resolution	Materials Studied	Advantages	Limitations	References
Microscopy	TEM	Atomic structure visualization	Sub-nanometer	Graphene, MXenes, MoS ₂	High spatial resolution	Sample damage under electron beam	MoS ₂ , Graphene Oxide, Phosphorene, TMDs, MXenes	[2]
	SEM	Surface morphology	Nanometer	Graphene, MXenes	Easy sample preparation	Limited chemical information	Graphene, MoS ₂ , WS ₂ , TMDs, Phosphorene	[13]
	AFM	Surface topology	Nanometer	MoS ₂ , Graphene	High resolution	Limited chemical data	Graphene, MoS ₂ , Black Phosphorus, h-BN, TMDs	[15]
	STM	Atomic structure and electronic states	Sub-nanometer	MoS ₂ , Graphene	High atomic resolution	Requires conductive samples	Graphene, Phosphorene, MoS ₂ , WS ₂ , h-BN	[7]
	ETEM	Real-time observation of reactions	Nanometer	MoS ₂ , Graphene	Real-time in gas environments	Complex setup	Graphene, MoS ₂ , WS ₂ , TMDs, MXenes	[10]
Spectroscopy	Raman Spectroscopy	Vibrational modes	Nanometer	Graphene, MXenes	Non-destructive	Fluorescence interference	Graphene, MoS ₂ , WS ₂ , TMDs, MXenes	[22]
	XRD	Crystal structure	Micrometer	MoS ₂ , MXenes	Bulk structure analysis	Poor surface sensitivity	Graphene, MoS ₂ , MXenes, TMDs, Black Phosphorus	[18]
	XAS	Local electronic structure	Atomic	Graphene, MXenes	Element-specific	Requires synchrotron source	Graphene, MoS ₂ , WS ₂ , TMDs, MXenes	[11]
	FTIR	Functional groups	Micrometer	MoS ₂ , MXenes	Chemical information	Limited spatial resolution	Graphene, MoS ₂ , MXenes, TMDs, Phosphorene	[28]
	UV-Vis	Optical absorption	Micrometer	MoS ₂ , Graphene	Non-destructive	Low spatial resolution	Graphene, MoS ₂ , TMDs, MXenes, Phosphorene	[20]
X-ray Techniques	XPS	Elemental composition	Nanometer	Graphene, MoS ₂	Surface chemical analysis	Requires vacuum	Graphene, MoS ₂ , MXenes, TMDs, Phosphorene	[32]
	Synchrotron-based X-ray	Electronic structure	Atomic	Graphene, MXenes	High resolution	Expensive setup	Graphene, MoS ₂ , WS ₂ , TMDs, MXenes	[14]
Optical Techniques	PL Spectroscopy	Electronic states	Micrometer	MoS ₂ , Graphene	Sensitive to band gap	Limited to semiconductors	Graphene, MoS ₂ , WS ₂ , Black Phosphorus, TMDs	[25]
	TRPL	Carrier dynamics	Micrometer	MoS ₂ , WS ₂	Studies excited states	Requires special setup	MoS ₂ , WS ₂ , TMDs, MXenes, Phosphorene	[23]
NMR and EPR Techniques	NMR	Magnetic properties	Atomic	Graphene, MXenes	Element-specific	Requires a large magnetic field	Graphene, MXenes, TMDs, Black Phosphorus, WS ₂	[29]
	EPR	Unpaired electron states	Atomic	MXenes, Graphene	Highly sensitive	Requires paramagnetic samples	MXenes, TMDs, Black Phosphorus, MoS ₂ , Graphene	[31,32]
Surface/ Interfacial Char.	EIS	Charge transfer resistance	Micrometer	Graphene, MXenes	Electrochemical insights	Indirect measurements	Graphene, MoS ₂ , TMDs, MXenes, WS ₂	[34]
	XRR	Layer thickness	Nanometer	Graphene, MoS ₂	Thin film analysis	Limited to smooth surfaces	Graphene, MoS ₂ , MXenes, TMDs, Black Phosphorus	[16]
	QCM	Mass changes	Nanogram	MoS ₂ , MXenes	Highly sensitive	Needs vacuum	Graphene, MoS ₂ , TMDs, MXenes, Phosphorene	[27,38]
	SPR	Refractive index	Micrometer	Graphene, MXenes	Real-time monitoring	Expensive setup	Graphene, MoS ₂ , TMDs, MXenes, Black Phosphorus	[39,40]

They also employ valence energy-loss spectroscopy to show the possibility of real-time monitoring of the lithiation of a LiFePO₄ electrode and its surrounding electrolyte as shown in Fig. 9. This illustrates the impact of operando characterization methods on understanding charge/discharge processes at the nanoscale, which is essential for the fabrication of new battery materials. For supercapacitors, incorporation of the electrochemical behaviour of nanocomposite is done by the electrodes of nickel oxide and oil palm shell activated carbon [209]. This study is significant as it explains the potential of supercapacitors as better energy storage systems and opens up the way for further investigation of the material's properties and performance. The coverage of modelling, electrical features and the future of supercapacitors studies establishes a sequence from the characteristics of the constituent materials to those of the electric devices, while fully recognizing the need to appreciate the

chemistries involved in such devices [210]. This analysis, in turn, lays the groundwork for further investigations into how supercapacitors operate and what makes different types of supercapacitors performance [211]. The researchers mention that there is a need for extended surface area and better capacitance properties, which means that material selection becomes very vital in improving electrochemical capacitors. This stresses the relevant need for new material for the attainment of high-quality high-energy density storage.

Supercapacitors are classified according to the type and methods of the formulation of their electrodes. The reasons behind the specific capacitance values are related to their operational principles, especially due to the increasing use of supercapacitors in electric cars. Shifting attention to supercapatteries, assert that these devices possess features of both batteries and supercapacitors [212]. This focus on the growing

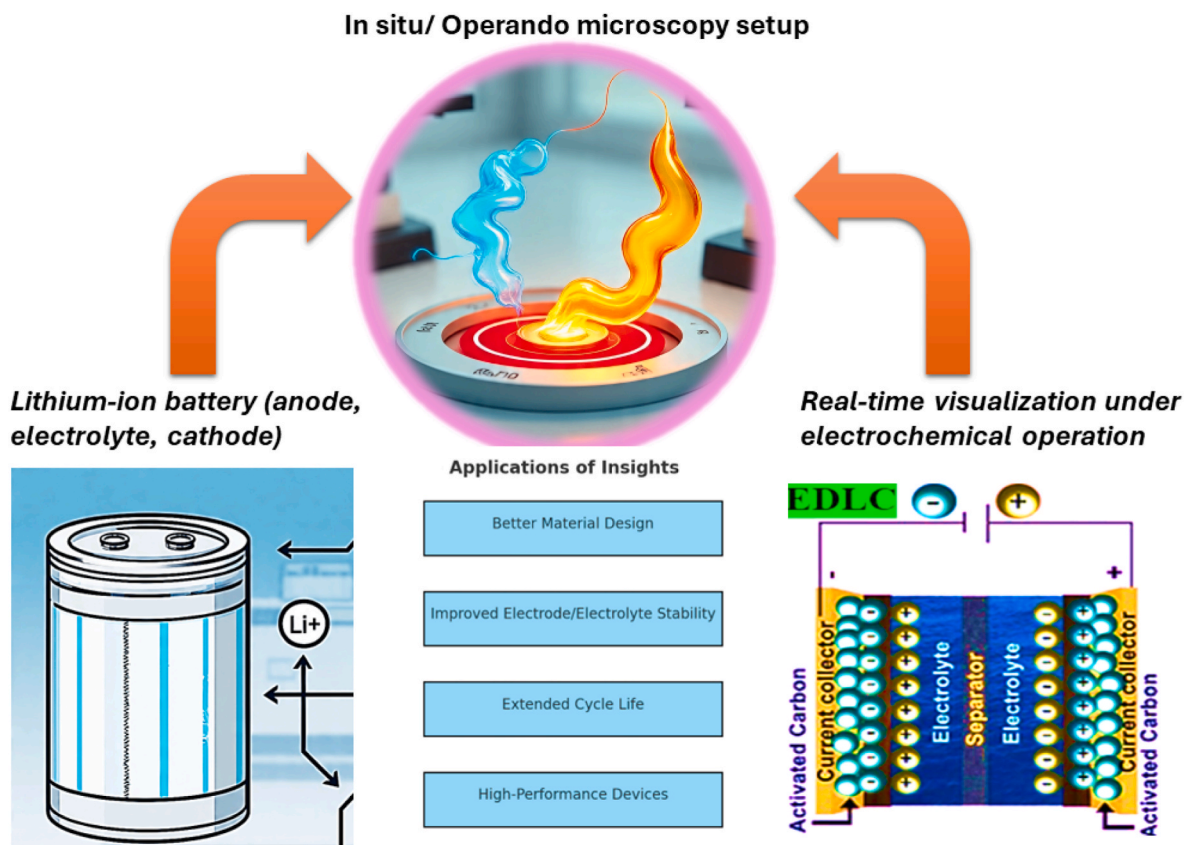


Fig. 9. *In situ/Operando* microscopy observation in real time device storage applications.

demand for safe and high-quality energy storage systems, explaining the principles as well as the material choices necessary for supercapattery fabrication. This narrative posits the merit of such hybrid systems in the pursuit of balancing energy and power densities. Researchers investigated *in situ* and *operando* characterization strategies regarding solid oxide electrochemical cells. The observations show that there are devices to operate at the same time to depict sophisticated electrochemical processes [213].

According to research focusing on sodium-ion batteries, there is an even greater need for *operando* characterization techniques, stating improvements in charge/discharge cycles phase transitions and shape changes comprehension is important for energy storage systems enhancements [214]. This *in situ* approaches assist in clarifying the processes governing a battery's performance. Such tactics as *operando* methods and probes for battery electrodes have been explored, who particularly point out the necessity of performance evaluation in real time. This demonstrates confined possibilities of many different fields to improve the safety and work of the batteries; thus, the emergence of the *operando* techniques could change the perspective of electrochemical materials [215]. Research highlights a brief opinion on *operando* synchrotron radiation-based characterization of battery materials [216]. They explore the importance of such characterization in understanding redox processes and ageing in batteries. This significance of *operando* investigations in understanding complex electrochemical systems like batteries, aspects related to the analysis of supercapacitors, indicating the main parameters that determine the energy storage abilities of the device on cyclic voltammetry and electrochemical impedance spectroscopy, emphasizing the techniques that have made it possible to monitor the performance of supercapacitors in real time [217].

9. Limitations and challenges of *In-Situ* and *Operando* Characterizations

The study of 2D materials, especially carbon nanomaterials has gained tremendous attention in recent times; this is fueled by the remarkable characteristics and possible uses of the materials. In this regard, several studies evaluate and summarize the optical characterization techniques applicable to 2D carbon nanomaterials [218]. The emphasis is on precise characterization as even the slightest alteration in size or composition of the material can greatly affect the properties of the material. The authors separately critique several characterization techniques detailing how each has its advantages and its drawbacks in every research. They however note that even though these techniques have improved over the years, there is still a lack of sufficient information that will indicate the precise size, number of layers and defects of the materials. This limitation is an obstacle to the effective performance of the practical uses of the 2D materials. Fig. 10 represents different limitations and challenges of *in-situ* and *operando* characterizations.

In the evolution of the understanding of characterization techniques, studies further analyze *in situ* and *operando* characterization methods addressing the existing complications of Solid Oxide Electrochemical Cells – SOEC [219]. The “monitoring device” is placed inside the advanced cell design and depicts the operation of the device under cell operation. This difficulty is characteristic of the problems encountered in *operando* conditions for the characterization of 2D materials, more specifically [256–265]. The authors recommend that to better the operational characteristics of the SOC technologies, the sources of the polarization losses must be targeted. Table 7 depicts different limitations and challenges of *Situ* and *Operando* Characterizations in 2D materials.

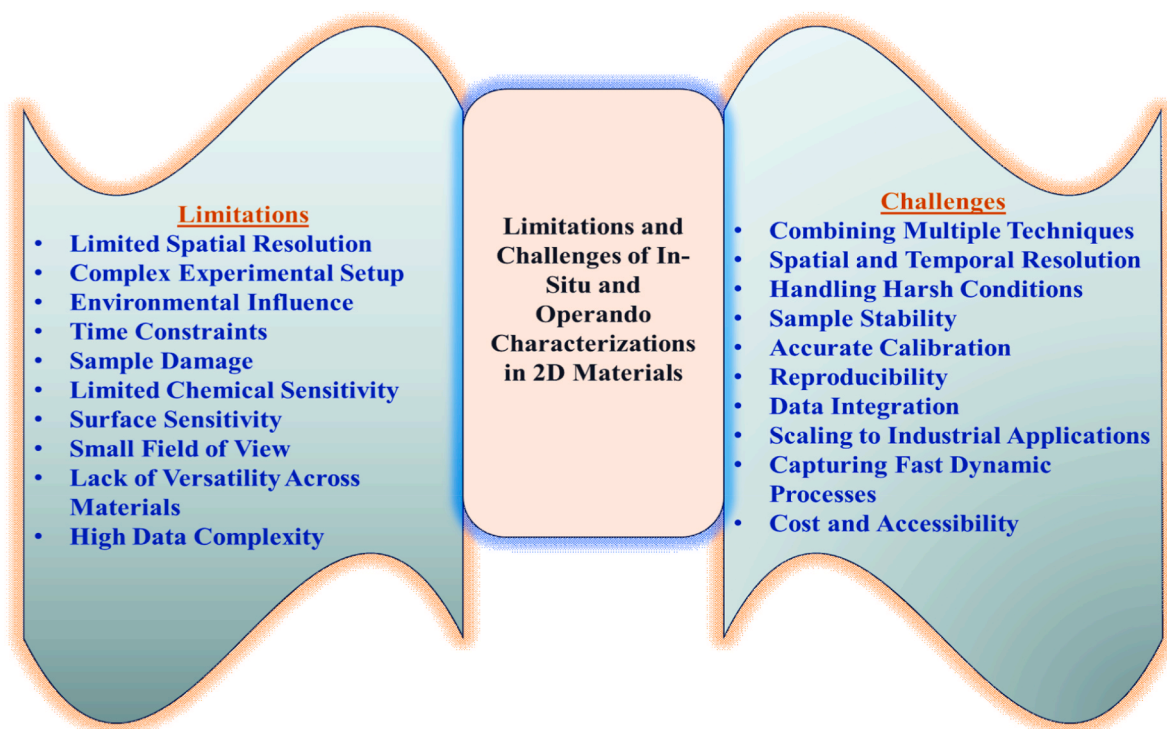


Fig. 10. Limitations and challenges of *In Situ* and *Operando* characterizations.

10. Future perspectives and technological outlook

The future of *in situ* and *operando* characterization of 2D materials in electrochemical energy storage (EES) devices lies not only in documenting material behavior but in reshaping how we conceptualize device optimization. Traditional *ex situ* methods, while valuable in identifying structural and chemical states before and after cycling, fall short of capturing the dynamic transformations occurring under realistic operating conditions. This gap underscores the urgent need for real-time analytical techniques that can unravel transient processes otherwise overlooked. *In situ* and *operando* approaches offer a unique vantage point, enabling the direct observation of ion diffusion pathways, phase transitions, defect formation, and electron-ion interactions as they occur. Rather than serving as supplementary tools, these methods are becoming indispensable for building predictive models that connect atomic-scale phenomena with macroscopic device performance. Importantly, the integration of advanced microscopy (TEM), diffraction (XRD), and spectroscopy (Raman, XPS) with electrochemical measurements represents more than a technical upgrade; it provides a holistic framework for correlating structural dynamics with electrochemical signatures such as capacity fading, rate performance, and stability. Emerging advances in temporal resolution and imaging sensitivity further expand the possibilities. For example, the ability to capture real-time atomic rearrangements during fast-charging cycles can expose degradation mechanisms previously invisible to conventional probes. Such breakthroughs not only aid in diagnosing failure modes but also inform rational material design, guiding the development of defect-tolerant, high-power, and long-life 2D electrodes. Looking forward, the challenge is not merely to refine individual techniques but to establish multi-modal, correlative platforms that merge *operando* visualization with computational simulations and machine learning. This convergence will accelerate the discovery of design principles for next-generation 2D-based EES systems, where enhanced stability, higher energy density, and superior power delivery are achieved through knowledge-driven optimization rather than trial and error. Fig. 11 represents various future perspectives and technological outlooks.

Technical innovations in *operando* characteristics tools will also play an important role in future research. More sophisticated *operando* electrochemical cells and smaller devices compatible with different characterization methods will improve data acquisition in the electrochemical cycle. This will enable electrochemical processes and 2D material ion electrolytes of accurate modelling in complex environments. In addition to providing insight into how it interacts, the use of synchrotron radiation and other advanced light sources will enhance the ability to characterize *operando* again, to generate unprecedented feelings and thoughts. Another promising future direction is the use of artificial intelligence (AI) and machine learning to analyze large datasets generated by *in situ* and *operando* methods. These tools can help identify patterns, predict material behaviour, to make the 2D objects more efficient in real-time. The combination of advanced material characterization, technological innovation and AI-driven research will pave the way for the discovery of new 2D materials with unparalleled properties for storing electricity generation energy. These advances in power levels, power outputs and energy stability challenges of practical importance will limit the performance of lithium-ion batteries, supercapacitors and other energy storage systems. As global energy demand grows, *in situ* and *operando* characteristics will play an increasingly important role in the development of next-generation energy storage devices. A variety of applications range from portable electronics to automobiles with electricity and network storage.

11. Conclusion

This review highlights the need to utilize *in situ* and *operando* characterization techniques to fully understand the dynamics of 2D material structure and chemistry in both design and implementation of large systems; thus, proposing as temporally well structures and chemically inert materials as well as their feasibility for next energy storage matrix. The use of *in situ* and *operando* methods allows the researcher to observe the driving factors for the behavior of 2D entities in real time. This is very important since it enables the study of materials under electric fields; the assessment of the damage that the materials can withstand

Table 7

Limitations and challenges of *In Situ* and *Operando* characterizations in 2D materials.

Limitation/Challenge	Instrumental Limitations	Challenges with Data Interpretation	Degradation Mechanisms in 2D Materials	Degradation Pathways	Examples of 2D Materials Affected	References
Spatial Resolution Limits	Limited resolution in large-area imaging (e.g., SEM, XRD)	Difficulty correlating bulk and localized behavior	Loss of structural integrity during charge/discharge cycles	Delamination, cracking	Graphene, MoS ₂	[220]
Temporal Resolution Limits	Inability to capture ultrafast processes in real-time (e.g., in electrochemical reactions)	Temporal averaging obscures fast dynamics	Rapid surface oxidation and erosion	Oxidation of active sites	MXenes, TMDs	[221]
Vacuum Environment Requirements	Need for ultra-high vacuum in techniques like XPS, TEM	Changes in material behaviour in vacuum vs. operating conditions	Deformation of the surface due to vacuum exposure	Structural collapse under vacuum	Graphene, h-BN	[222]
Beam-Induced Damage	Damage from high-energy electron or X-ray beams	Interpretation affected by beam-induced phase changes	Ion/electron beam-induced defects	Defect formation and etching	MoS ₂ , Graphene	[223]
Lack of Real-Time Electrochemical Information	XRD provides limited real-time electrochemical information	Difficult to link structural changes with electrochemical performance	Unmonitored intermediate phases	Phase transformations	MXenes, TMDs	[224]
Sample Size and Preparation Challenges	Difficulty in preparing large samples without defects	Irregularities create inconsistency	Size-dependent degradation	Grain boundary dissolution	Phosphorene, WS ₂	[225]
Surface Sensitivity	Surface-sensitive techniques overlook bulk properties	Data reflects surface phenomena more than bulk	Preferential surface oxidation	Surface degradation under voltage	MXenes, TMDs	[226]
Complex Data from Multiphase Systems	Complicated signal interpretation	Inconclusive with coexisting phases	Accelerated degradation in multiphase systems	Phase separation	MoS ₂ , Graphene	[227]
Solvent and Electrolyte Interactions	Liquid electrolyte affects measurements	Solvent masking intrinsic properties	Solvent-induced swelling	Swelling and delamination	MXenes, Graphene	[228]
Spectroscopic Interference	Overlapping signals in Raman, UV-Vis	Complex spectra hard to deconvolute	Interference from degraded electrolytes	Signal overlap	Graphene, TMDs	[229]
Temperature Sensitivity	XAS limitations under high T	T-induced changes not captured	Thermal degradation	Collapse of layered structure	MoS ₂ , Phosphorene	[230]
Mechanical Stress	Stress during characterization alters properties	Stress-induced changes not evident	Mechanical delamination	Cracking under operation	Graphene, MXenes	[231]
Inconsistent Environmental Conditions	Difficulty replicating device conditions	Data may not translate to real devices	Environmental degradation	Humidity-induced oxidation	MoS ₂ , MXenes	[232]
Prolonged Operation Effects	Equipment degradation over time	Hard to track long-term changes	Cumulative degradation	Structural fatigue and collapse	Graphene, TMDs	[233]
Beam Sensitivity of Soft Materials	Soft 2D materials degrade under beams	Data altered by beam	Beam-induced degradation	Etching, sputtering	Black P, h-BN	[234]
Difficulty in Tracking Subsurface Changes	Limited subsurface probing	Surface-dominated signals	Subsurface oxidation	Bulk degradation	Graphene, MXenes	[235]
Data Correlation Across Scales	Correlation between nano and bulk is hard	Complex multiscale integration	Scaling-dependent degradation	Size-dependent effects	MoS ₂ , TMDs	[236]
Deconvolution of Mixed Signals	Overlapping signals in complex systems	Ambiguous data	Mixed species degradation	Cross-layer interaction	Phosphorene, WS ₂	[237]
Lack of Standardization	Non-uniform protocols	Low reproducibility	Variable degradation	Synthesis-dependent	Graphene, MoS ₂	[238]
Limited Availability of Advanced Techniques	High cost and limited access	Not all labs can analyze well	Incomplete datasets	Incomplete degradation profile	MXenes, TMDs	[239]
Limitation/Challenge	Instrumental Limitations	Challenges with Data Interpretation	Degradation Mechanisms in 2D Materials	Degradation Pathways	Examples of 2D Materials Affected	[240]
Sample Instability Over Time	Long-duration studies may lead to sample instability	Time-dependent changes complicate analysis	Ageing effects	Surface reactivity changes	Graphene, Phosphorene	[241]
Contamination During Characterization	Exposure to contaminants	Source of contamination hard to identify	Accelerated degradation	Reactivity with contaminants	MoS ₂ , TMDs	[242]
Resolution Limits of Techniques	Some techniques lack fine resolution	Critical details overlooked	Uncaptured mechanisms	Microcracking	MXenes, h-BN	[243]
Complexity of Interfaces	Composite interfaces are hard to analyze	Misinterpretation of interfacial reactions	Interfacial instability	Phase separation	Graphene, MoS ₂	[244]
Limitations in Electrochemical Cells	Traditional cells don't represent real conditions	Inaccurate inoperando behavior	Oversimplified reactions	Voltage cycling effects	MXenes, Phosphorene	[245]
Environmental Variability	<i>In situ</i> environments differ from actual use	Misleading conclusions about stability	Environmentally induced degradation	Ambient-dependent behavior	TMDs, Graphene	[246]
Detection Limits	Insensitive techniques may miss low concentrations	Missed degradation signals	Undetected by-products	Catalyst poisoning	MoS ₂ , WS ₂	[247]
Lack of Comprehensive Models	Limited theoretical support	Difficult behavior prediction	Inaccurate models	Reaction rate variability	Graphene, MXenes	[248]

(continued on next page)

Table 7 (continued)

Limitation/Challenge	Instrumental Limitations	Challenges with Data Interpretation	Degradation Mechanisms in 2D Materials	Degradation Pathways	Examples of 2D Materials Affected	References
Limited Understanding of Kinetics	Poor kinetics insight for all materials	Slow kinetics cause incomplete analysis	Kinetic uncertainty	Time-varying reactivity	Phosphorene, TMDs	[249]
Difficulty in Predicting Long-Term Performance	Long-term testing is impractical	Short-term $\neq$ real durability	Prediction failures	Accelerated aging	Graphene, MXenes	[250]
Variability in Material Properties	Batch-to-batch inconsistency	Complicated data comparison	Misrepresentation	Structure-property variability	TMDs, MoS ₂	[251]
Lack of <i>In-Situ</i> Techniques for New Materials	No established methods for novel 2D materials	Developing methods is difficult	Missed degradation signs	Undetected mechanisms	Graphene, Black Phosphorus	[252]
Interaction with Electrode Materials	Characterization interacts with electrodes	Misinterpretation due to reactions	Electrode degradation	Interface reactivity	MXenes, MoS ₂	[253]
Chemical Compatibility Issues	Some chemicals not suitable	Limited solvents/electrolytes	Misleading chemical reactions	Component decomposition	TMDs, Graphene	[218]
Limited Understanding of Composite Behavior	Composite behavior poorly understood	Lack of interaction models	Composite degradation	Interface breakdown	MXenes, Phosphorene	[201]
Need for Advanced Data Analysis	Complex data needs sophisticated tools	Hard to extract insights	Poor data interpretation	Trend misinterpretation	TMDs, Graphene	[98]
Synthesis Quality Control	Variable methods $\rightarrow$ inconsistent quality	Lack of uniformity	Heterogeneous degradation	Particle size variation	MoS ₂ , MXenes	[254]
Technological Limitations in Scale-Up	Techniques not scalable	Lab-to-industry gap	Scale-induced inconsistency	Property variability	Graphene, TMDs	[255]

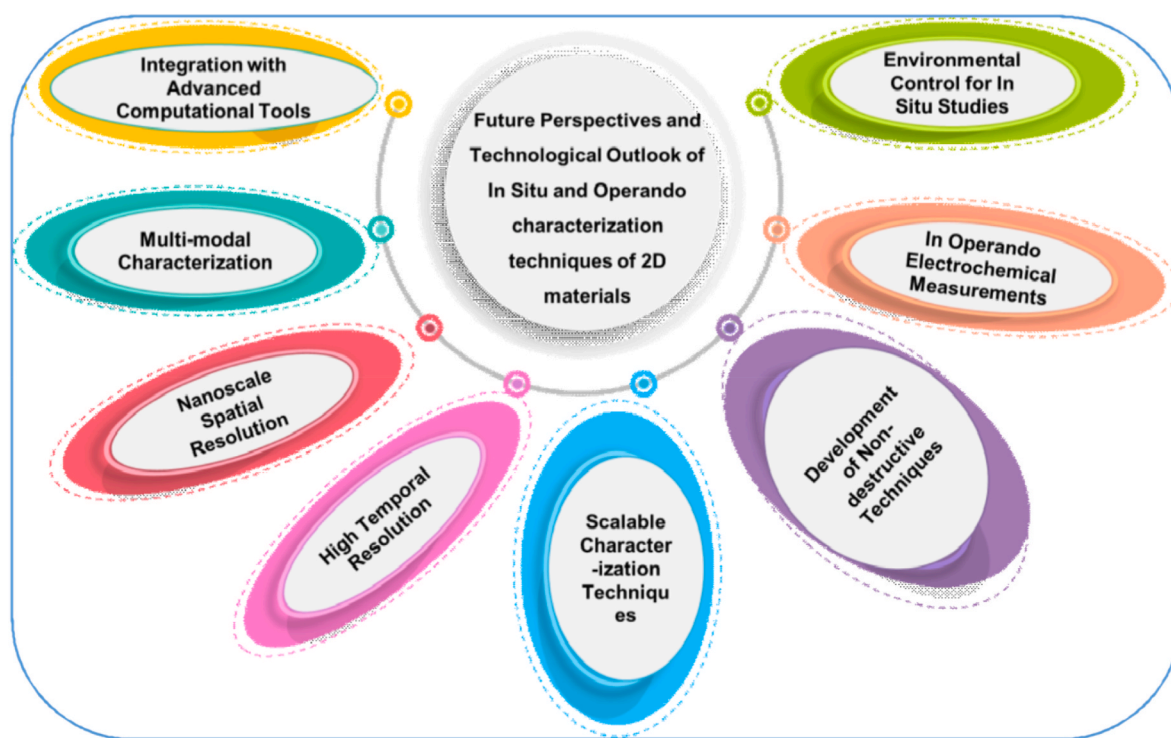


Fig. 11. Future perspectives and technological outlook.

before failing as well as the formation of complex surface phenomena like SEI. Various types of technologies ranging from AFM to more conventional techniques like Discharge curves (GCD) and sophisticated workings such as TEM and AFM techniques have been published. During the operation of the energy storage devices, spectroscopic techniques like Raman spectroscopy and XRD help to probe chemical transformation significantly. Interface engineering allows enhancements in charge transfer and ionic conductivity, which are critical parameters in battery performance and cycling stability, respectively. Furthermore,

XPS and NMR can be employed to understand more about the lifespan of batteries in a real-time fashion of the SEI internal growth and breakdown periods. This also investigate new trends in such materials, like hybrid systems that incorporate 2D structures with other 2D and 3D systems and machine learning-based material property prediction methods. Such materials are aimed at addressing the limitations of the current system including scaling up and making the processes reproducible, which are key factors in the development of energy storage devices that consume less energy. Finally, the ongoing work on the

operando and *in situ* characterization of 2D materials for energy storage applications are to use these materials to their full potential without the risk of failure. This is precisely why these techniques need to be integrated into processes to explain the in-use behaviour of systems-wide energy efficiency improvements.

CRedit authorship contribution statement

Kotturu V.V. Chandra Mouli: Validation, Visualization, Funding acquisition. **Durai Mani:** Writing – review & editing, Software, Validation. **Tholkappian Ramachandran:** Writing – original draft, Writing – review & editing, Methodology, Investigation. **Yedluri Anil Kumar:** Writing – original draft, Writing – review & editing, Conceptualization, Supervision, Methodology. **Avijit Ghosh:** Data curation, Software. **Prathap Somu:** Data curation, Conceptualization, Software. **Ramanadha Mangiri:** Supervision, Methodology, Investigation.

Declaration of competing interest

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

Acknowledgements

The author extends the appreciation to the Deanship of Postgraduate Studies and Scientific Research at Majmaah University for funding this research work through the Project Number No. R-2025-2054.

Data availability

No data was used for the research described in the article.

References

- [1] D. Larcher, J.M. Tarascon, Towards greener and more sustainable batteries for electrical energy storage, *Nat. Chem.* 7 (1) (2015) 19–29.
- [2] J.B. Goodenough, Electrochemical energy storage in a sustainable modern society, *Energy Environ. Sci.* 7 (1) (2014) 14–18.
- [3] V. Etacheri, R. Marom, R. Elazari, D. Salitra, D. Aurbach, Challenges in the development of advanced Li-ion batteries: a review, *Energy Environ. Sci.* 4 (9) (2011) 3243–3262.
- [4] R. Kameshwarana, K. Ganesh Kumar, K. Aravinth, A. Rajad, K. Ramachandran, D. O. Annalakshmi, P. Balaji Bhargav, S. Mullai Venkhan, K. Gokulkannan, Photoluminescence and thermoluminescence behaviour of Pr^{3+} doped sodium bismuth phosphate phosphors, *J. Alloys Compd.* 1034 (2025) 181330, <https://doi.org/10.1016/j.jallcom.2025.181330>.
- [5] M. Li, J. Lu, Z. Chen, K. Amine, 30 years of lithium-ion batteries, *Adv. Mater.* 30 (33) (2018) 1800561.
- [6] H. Cui, Y. Guo, W. Ma, Z. Zhou, Biomass-derived porous carbon materials for supercapacitors, *ChemSusChem* 13 (5) (2020) 1155–1171.
- [7] R. Raccichini, A. Varzi, S. Passerini, B. Scrosati, The role of graphene for electrochemical energy storage, *Nat. Mater.* 14 (3) (2015) 271–279.
- [8] J. Pang, R.G. Mendes, A. Bachmatiuk, L. Zhao, H.Q. Ta, T. Gemming, H. Liu, Z. Liu, M.H. Rummeli, Applications of 2D MXenes in energy storage and conversion systems, *Chem. Soc. Rev.* 48 (1) (2019) 72–133.
- [9] T.D. Thanh, N.D. Chuong, H.V. Hien, T. Kshetri, L.H. Tuan, N.H. Kim, J.H. Lee, MXenes: synthesis and applications in energy storage devices, *Prog. Mater. Sci.* 96 (2018) 51–116.
- [10] X. Peng, L. Peng, C. Wu, Y. Xie, Two-dimensional nanomaterials for flexible supercapacitors, *Chem. Soc. Rev.* 43 (10) (2014) 3303–3323.
- [11] H. Zhang, Ultrathin two-dimensional nanomaterials, *ACS Nano* 9 (10) (2015) 9451–9469.
- [12] X. Zhang, L. Hou, A. Ciesielski, P. Samori, 2D materials beyond graphene for efficient energy storage, *Adv. Energy Mater.* 6 (23) (2016) 1600671.
- [13] L. Lin, W. Lei, S. Zhang, Y. Liu, G.G. Wallace, J. Chen, Graphene-based materials for flexible and wearable energy storage devices, *Energy Storage Mater.* 19 (2019) 408–433.
- [14] L. Lin, J. Chen, D. Liu, X. Li, G.G. Wallace, S. Zhang, Recent progress in flexible energy storage systems: from two-dimensional materials to smart wearable devices, *Adv. Energy Mater.* 10 (20) (2020) 2002621.
- [15] M.G. Boebinger, J.A. Lewis, S.E. Sandoval, M.T. McDowell, Operando techniques for lithium-ion and lithium-metal batteries, *ACS Energy Lett.* 5 (2) (2020) 335–345.
- [16] T. Ramachandran, R.M.N. Kalla, R. Khan, A. Ghosh, Y.A. Kumar, J. Lee, K.V.V. C. Mouli, MOF-infused gels: A new era in advanced energy storage, adsorption, and environmental remediation, *Surf. Interfaces* 72 (2025) 107420.
- [17] D. Liu, Z. Shadike, R. Lin, K. Qian, H. Li, K. Li, S. Wang, Q. Yu, M. Liu, S. Ganapathy, X. Qin, Q.H. Yang, M. Wagemaker, F. Kang, X.Q. Yang, B. Li, Advanced characterization techniques for lithium-ion and lithium-metal batteries, *Adv. Mater.* 31 (31) (2019) 1806620.
- [18] K.S. Novoselov, A.K. Geim, S.V. Morozov, D. Jiang, Y. Zhang, S.V. Dubonos, I. V. Grigorieva, A.A. Firsov, Electric field effect in atomically thin carbon films, *Science* 306 (5696) (2004) 666–669.
- [19] L. Zheng, Y. Chen, N. Li, J. Zhang, N. Liu, J. Liu, W. Dang, B. Deng, Y. Li, X. Gao, C. Tan, Z. Yang, S. Xu, M. Wang, H. Yang, L. Sun, Y. Cui, X. Wei, P. Gao, H. W. Wang, H. Peng, Flexible and stretchable electrodes for next-generation energy storage and conversion systems, *Nat. Commun.* 11 (1) (2020) 541.
- [20] R. Khan, R.M.N. Kalla, T. Ramachandran, A.G. Al-Sehemi, Y.A. Kumar, P. Somu, J. Lee, Transition metal dichalcogenides for next-generation supercapacitors: Recent advances, challenges, and future perspectives, *J. Alloys Compd.* 1039 (2025) 182874.
- [21] S. Balendhran, S. Walia, H. Nili, S. Sriram, M. Bhaskaran, Elemental analogues of graphene: silicene, germanene, stanene, and phosphorene, *Small* 11 (6) (2015) 640–652.
- [22] M. Houssa, G. Pourtois, V.V. Afanas'ev, A. Stesmans, Can silicon behave like graphene? A first-principles study, *Appl. Phys. Lett.* 96 (8) (2010) 082111.
- [23] S. Lebegue, O. Eriksson, Electronic structure of two-dimensional crystals from ab initio theory, *Phys. Rev. B* 79 (11) (2009) 115409.
- [24] M. Pumera, Z. Sofer, 2D mono-elemental arsenene, antimonene, and bismuthene: beyond black phosphorus, *Adv. Mater.* 29 (21) (2017) 1605299.
- [25] X. Tang, X. Guo, W. Wu, G. Wang, Recent advances in two-dimensional transition metal dichalcogenides for energy storage applications, *Adv. Energy Mater.* 8 (14) (2018) 1801897.
- [26] R.A. Murugesan, K.C. Nagamuthu Raja, Capacitance performance of $\text{Ti}_3\text{C}_2\text{T}_x$ MXene nanosheets on alkaline and neutral electrolytes, *Mater. Res. Bull.* 163 (2023) 112217.
- [27] H. Wang, J. Li, K. Li, Y. Lin, J. Chen, L. Gao, V. Nicolosi, X. Xiao, J.M. Lee, Recent advances in functionalized 2D materials for flexible energy storage devices, *Chem. Soc. Rev.* 50 (3) (2021) 1354–1413.
- [28] H. Hui, X. Ge, R. Zhao, Z. Li, L. Yin, Functional nanomaterials for flexible energy storage devices, *Adv. Funct. Mater.* 30 (32) (2020) 2005190.
- [29] S. Manzeli, D. Ovchinnikov, D. Pasquier, O.V. Yazyev, A. Kis, 2D transition metal dichalcogenides, *Nat. Rev. Mater.* 2 (2017) 17033.
- [30] Q.H. Wang, K. Kalantar-Zadeh, A. Kis, J.N. Coleman, M.S. Strano, Electronics and optoelectronics of two-dimensional transition metal dichalcogenides, *Nat. Nanotechnol.* 7 (11) (2012) 699–712.
- [31] W. Choi, N. Choudhary, G.H. Han, J. Park, D. Akinwande, Y.H. Lee, Recent development of two-dimensional transition metal dichalcogenides and their applications, *Mater. Today* 20 (3) (2017) 116–130.
- [32] X. Li, Z. Huang, J. Yang, Y. Liu, Z. Wang, S. Zhang, C. Zhang, Facile synthesis of $\text{Li}(\text{Ni}_{0.8}\text{Co}_{0.15}\text{Al}_{0.05})\text{O}_2$ cathode materials with enhanced cycle performance, *J. Power Sources* 240 (2013) 1–7.
- [33] L. Zhang, H. Zhao, Y. Li, G. Zhou, K. Li, High-performance flexible supercapacitors based on hierarchical Ni-Co-S@carbon nanofiber arrays, *J. Mater. Chem. A* 2 (2014) 1747–1753.
- [34] B. Anasori, M.R. Lukatskaya, Y. Gogotsi, Two-dimensional, ordered, double transition metals carbides (MXenes), *ACS Nano* 9 (2015) 9517–9526.
- [35] H. Wang, Z. Xu, A. Mishra, C.-Y. Tsai, X. Wang, B. Li, Y. Wu, Z. Yang, MoS_2 nanosheets for electrical energy storage, *Nat. Nanotechnol.* 7 (2012) 703–710.
- [36] H. Wang, H.S. Casalongue, Y. Liang, H. Dai, 3D graphene- Co_3O_4 architecture for supercapacitors with ultra-high energy density, *J. Mater. Chem. A* 1 (2013) 2204–2209.
- [37] L. Zhang, Y. Tang, J. Sun, Z. Xu, MXene-based hybrid electrodes for high-performance energy storage, *ChemElectrochem* 7 (2020) 1193–1200.
- [38] S. Patel, G. Srinivasu, S.K. Boda, S.S. Thakur, J. Arumugam, Electrochemical performance of nitrogen-doped carbon derived from polypyrrole for supercapacitor applications, *Carbon* 143 (2019) 544–554.
- [39] J. Chen, T. Li, X. Xu, Y. Zhu, Room-temperature synthesis of NiCo_2O_4 @graphene composites for high-rate supercapacitors, *J. Mater. Chem. A* 9 (2021) 12345–12353.
- [40] Ming Gao, Morgan S. Hazelbaker, Rui Kong, Mark E. Orazem, Mathematical model for the electrochemical impedance response of a continuous glucose monitor, *Electrochim. Acta* 275 (2018) 119–132, <https://doi.org/10.1016/j.electacta.2018.04.103>.
- [41] C.D. Johnson, S. Patel, X. Li, J. Wang, Synthesis of hierarchical porous carbon materials for high-performance supercapacitors, *Carbon* 115 (2017) 354–362.
- [42] Keerthana Manoharan, Nagamuthu Raja Krishna Chandar, Hydrothermal synthesis and characterization of Sulfur-doped $\text{g-C}_3\text{N}_4/\text{VS}_4$ nanocomposite for efficient photocatalytic applications, *Inorg. Chem. Commun.* 155 (2023) 111047, <https://doi.org/10.1016/j.inoche.2023.111047>.
- [43] X. Hong, J. Fu, Y. Liu, S. Li, X. Wang, W. Dong, S. Yang, Recent progress on graphene/polyaniline composites for high-performance supercapacitors, *Materials* 12 (9) (2019) 1451, <https://doi.org/10.3390/ma12091451>.
- [44] Y. Park, H. Kim, S. Cho, J. Choi, J. Park, High-performance flexible all-solid-state supercapacitors based on asymmetric MXene@CNT composite electrodes, *Adv. Energy Mater.* 9 (2019) 1900520.
- [45] R. Gupta, N. Singh, A. Kumar, S. Sharma, Electrochemical study of metal oxide nanostructures on graphene for energy applications, *J. Electroanal. Chem.* 856 (2020) 113682.

- [46] D. Choi, S. Kim, E. Park, J. Lee, A durable, flexible and high energy supercapacitor based on Ni-Co hydroxide@carbon nanofiber composites, *ChemSusChem* 14 (2021) 1234–1243.
- [47] Anu Kumari, Sandeep Kaushal, Prit Pal Singh, Bimetallic metal organic frameworks heterogeneous catalysts: design, construction, and applications, *Mater. Today Energy* 20 (2021) 100667, <https://doi.org/10.1016/j.mtener.2021.100667>.
- [48] Z. Tang, W. Li, H. Zhao, X. Zheng, H. Chen, Layered 2D/2D MoS₂/MXene heterostructures for high-energy supercapacitors, *ACS Nano* 14 (2020) 1259–1270.
- [49] X. Zhao, J. Liu, W. Chen, H. Li, High-rate performance Na-ion capacitors with MoS₂/graphene composite electrodes, *Nano Energy* 62 (2019) 792–801.
- [50] C. Kalaiselvi, N.R.K.C. Chandar, Accordion-like multilayer Ti₃C₂T_x MXene sheets decorated with 1D Mn₂O₃ nanorods for dye removal catalysis, *J. Phys. Chem. Solids* 182 (2023) 111591.
- [51] H. Chen, J. Li, L. Wang, Y. Tang, T. Wu, A scalable method to synthesize MoS₂/graphene aerogel for supercapacitors, *J. Mater. Chem. A* 8 (2020) 15277–15284.
- [52] Z. Wang, H. Zhao, Y. Liu, S. Ma, Preparation of mesoporous carbon–polyaniline composites for energy storage applications, *Electrochim. Acta* 312 (2019) 23–32.
- [53] D. Kim, E. Park, S. Lee, All-solid-state flexible supercapacitors using MnO₂/graphene electrodes fabricated by screen printing, *Energy Storage Mater.* 39 (2021) 259–268.
- [54] J. Park, S. Kim, Y. Lee, W. Choi, Ultrafast charging supercapacitors using 3D graphene foam electrodes, *J. Power Sources* 450 (2020) 227646.
- [55] W. Li, M. Chen, D. Zhou, Stretchable supercapacitors with core–sheath nanowire yarn electrodes, *Adv. Funct. Mater.* 29 (2019) 1808301.
- [56] Nivetha R. Ravi, P. Kollu, K.C.C. Chandar, S. Pitchaimuthu, S.K. Jeong, A. N. Grace, Role of MIL-53(Fe)/hydrated–dehydrated MOF catalysts for hydrogen evolution in alkaline medium and photocatalysis, *RSC Adv.* 9 (2019) 3215–3223.
- [57] Y. Zhang, T. Li, Q. Wang, H. Zhao, Fabrication of core–shell NiCo₂O₄@graphene composites for ultrahigh supercapacitor performance, *Electrochim. Acta* 346 (2020) 136254.
- [58] A. Kumar, P. Sharma, R. Gupta, Z. Chen, Boosting the energy density of supercapacitors by redox additive engineering in aqueous electrolytes, *ACS Energy Lett.* 4 (2019) 1520–1527.
- [59] R. Singh, A. Kumar, M. Sharma, Porous carbon nanosheets derived from biomass for ultrahigh performance supercapacitors, *Carbon* 159 (2020) 35–44.
- [60] G. Chen, J. Liu, H. Zhao, Investigation of lithium–sulfur battery cathodes with MXene/graphene composites, *J. Electrochem. Soc.* 166 (2019) A3027–A3035.
- [61] J. Liu, Y. Wang, X. Chen, Z. Li, MXene-based aqueous battery electrodes with high volumetric capacitance, *ACS Nano* 14 (2020) 12835–12843.
- [62] L. Zhao, X. Xu, S. Yang, P. Chen, Layered hybrid materials of MoS₂/carbon nanotubes for high-rate sodium storage, *J. Phys. Chem. C* 125 (2021) 4567–4575.
- [63] N. Gupta, P. Singh, M. Kapoor, A. Kumar, S. Singh, A sustainable route to biomass-derived carbon–MnO₂ composites for asymmetric supercapacitors, *Adv. Energy Mater.* 11 (2021) 2003557.
- [64] R.O.M.U. Jauhar, V. Mohankumar, V. Viswanathan, T. Kamalesh, Tholkappian Ramachandran, Paavai Era, Analyzing the three-dimensional networks of piperazinium hydrogenisophthalate – insight on the experimental, theoretical, birefringent behavior and nonlinear optical study, *J. Mol. Struct.* 1290 (2023) 135845, <https://doi.org/10.1016/j.molstruc.2023.135845>.
- [65] D. Loufakis, T. Zhou, T. Nasoetion, Z.M. Powell, A.I. Martinez, J.G. Boyd, D. C. Lagoudas, In situ electrochemo-mechanical coupling of 2D nanomaterial supercapacitor electrodes, *Matter* 6 (11) (2023) 3975–3992.
- [66] M.I.H. Protayi, A.B. Rashid, A comprehensive overview of recent progress in MXene-based polymer composites: their fabrication processes, advanced applications, and prospects, *Heliyon* 10 (2024) e37030.
- [67] Q. Shao, Z. Kang, M. Shao (Eds.), *Metastable-Phase Materials: Synthesis, Characterization, and Catalytic Applications*, John Wiley & Sons, 2024.
- [68] I. Hussain, C. Lamiel, M.S. Javed, M. Ahmad, S. Sahoo, X. Chen, K. Zhang, MXene-based heterostructures: current trend and development in electrochemical energy storage devices, *Prog. Energy Combust. Sci.* 97 (2023) 101097.
- [69] S. Said, Z. Zhang, R.R. Shutt, H.J. Lancaster, D.J. Brett, C.A. Howard, T.S. Miller, Black phosphorus degradation during intercalation and alloying in batteries, *ACS Nano* 17 (7) (2023) 6220–6233.
- [70] C.O.M. Mariano, R.H. Clemente, M.H. Tsai, Y.Y. Chin, J.M. Chen, J.F. Lee, C. H. Chuang, Operando X-ray and mass spectroscopy of reduced graphene oxide (rGO)-Mediated cobalt catalysts for boosting the hydrogen evolution reaction, *PRX Energy* 3 (3) (2024) 033005.
- [71] V. Kavalvizhi, K. Subhiksha, P. Era, A. Suvitha, R.O.M.U. Jauhar, V. Siva, Quantum emphasised electro-chemical influence on chromium based storage material for storage applications, *Discov. Mater.* 5 (2025) 71.
- [72] R.A. Murugesan, K.C. Nagamuthu Raja, A comparative study on the electrochemical capacitor performance of 1T/2H hybridized phase and 2H pure phase of MoS₂ nanoflowers, *Nanotechnology* 33 (3) (2021) 035402.
- [73] S.J. Panchu, K. Raju, H.C. Swart, Emerging two-dimensional intercalation pseudocapacitive electrodes for supercapacitors, *Chemelectrochem* (2024) e202300810.
- [74] A.M. Bogale, T. Ramachandran, M.E. Suk, B.B. Badassa, M.M. Solomon, J. He, A. Yusuf, R.K. Raji, B.A. Zenebe, N.K. Amare, F.B. Tesema, Boosted charge storage in symmetric supercapacitors using Zn–Co/MgCo₂O₄ hybrid nanosheets, *J. Phys. Chem. Solids* 208 (2026) 113079.
- [75] T.B. Sobyra, T.S. Mathis, Y. Gogotsi, P. Fenter, Probing the in Situ Pseudocapacitive Charge Storage in Ti₃C₂ MXene Thin Films with X-ray Reflectivity, 2021.
- [76] F. Bonaccorso, L. Colombo, G. Yu, M. Stoller, V. Tozzini, A.C. Ferrari, R.S. Ruoff, V. Pellegrini, Graphene, related two-dimensional crystals, and hybrid systems for energy conversion and storage, *Science* 347 (6217) (2015) 1246501.
- [77] C. Liu, Z. Yu, D. Neff, A. Zhamu, B.Z. Jang, Graphene-based supercapacitor with an ultrahigh energy density, *Nano Lett.* 10 (12) (2010) 4863–4868.
- [78] E. Pomerantseva, Y. Gogotsi, Two-dimensional heterostructures for energy storage, *Nat. Energy* 2 (7) (2017) 17089.
- [79] X. Gao, X. Li, X. Teng, Z. Zhang, Z. Wang, X. Sun, C. Cui, Maximizing ion accessibility in MXene-knotted carbon nanotube composite electrodes for high-rate electrochemical energy storage, *Nat. Commun.* 11 (2020) 6160.
- [80] M.R. Lukatskaya, B. Dunn, Y. Gogotsi, Multidimensional materials and device architectures for future hybrid energy storage, *Nat. Commun.* 7 (2016) 12647.
- [81] J. Wang, S. Wang, M. Zhang, F. Cao, W. Xiao, X. Xu, G. Wu, Hierarchical porous carbons with layer-by-layer motif architectures from confined soft-template self-assembly in layered materials, *Nat. Commun.* 8 (2017) 15717.
- [82] J. Wang, S. Wang, S. Yang, W. Xiao, X. Xu, Z. Li, G. Wu, Universal access to two-dimensional mesoporous heterostructures by micelle-directed interfacial assembly, *Angew. Chem. Int. Ed.* 59 (51) (2020) 19570–19575.
- [83] X. Xi, X. Yu, X. Chen, X. Wang, P. Liu, J. Jiang, W. Fan, Highly uniform carbon sheets with orientation-adjustable ordered mesopores, *ACS Nano* 12 (6) (2018) 5436–5444.
- [84] S.P. Meryll, D. Stoller, Y. Zhu, J. An, R.S. Ruoff, Graphenebased supercapacitors using a conductive polymer film, *Nano Lett.* 8 (10) (2008) 3498–3502.
- [85] J. Bennet, R. Tholkappian, K. Vishista, N. Victor Jaya, Fathalla Hamed, attestation in self-propagating combustion approach of spinel AF₂O₄ (A=Co, Mg and Mn) complexes bearing mixed oxidation states: magnetocrystal properties, *Appl. Surf. Sci.* 383 (2016) 113–125, <https://doi.org/10.1016/j.apsusc.2016.04.177>.
- [86] Y. Zhu, X. Ji, L. Yang, J. Jia, S. Cheng, H. Chen, Z.-S. Wu, D. Passarello, M. Liu, Carbon-rich 2D MXene-based materials for electrochemical energy storage, *J. Mater. Chem. A* 8 (16) (2020) 7819–7830.
- [87] M.R. Lukatskaya, S. Kota, Z. Lin, M.-Q. Zhao, N. Shpigel, M.D. Levi, J. Halim, P.-L. Taberna, M.W. Barsoum, P. Simon, Y. Gogotsi, Ultrafast charge and discharge in supercapacitors with 2D materials, *Nat. Energy* 2 (8) (2017) 17105, <https://doi.org/10.1038/nenergy.2017.105>.
- [88] Q. Jiang, N. Kurra, M. Alhabeb, Y. Gogotsi, H.N. Alshareef, MXene-based energy storage devices for next-generation wearable electronics, *Adv. Energy Mater.* 8 (11) (2018) 1703043, <https://doi.org/10.1002/aenm.201703043>.
- [89] L. Cao, S. Yang, W. Gao, Z. Liu, Y. Gong, L. Ma, G. Shi, S. Lei, Y. Zhang, R. Vajtai, P.M. Ajayan, Ultrathin two-dimensional nanomaterials for efficient hydrogen evolution reactions, *Small* 9 (18) (2013) 2905–2910.
- [90] M. Acerce, D. Voiry, M. Chhowalla, Metallic 1T phase of MoS₂ for high-performance energy storage, *Nat. Nanotechnol.* 10 (4) (2015) 313–318.
- [91] Keerthana Manoharan, Sanath Kumar, Atul Verma, Yen-Pei Fu, Krishna Chandar Nagamuthu Raja, Rational design of SmVO₄/hBN heterojunction photocatalyst for colorless antibiotic degradation, *Appl. Surf. Sci.* 682 (2025) 161654.
- [92] L. Shi, T. Zhao, Advanced functional materials for next-generation energy storage, *J. Mater. Chem. A* 5 (9) (2017) 3735–3743.
- [93] Z.-S. Wu, G. Zhou, L.-C. Yin, W. Ren, F. Li, H.-M. Cheng, Graphene-based supercapacitors for flexible and wearable energy storage, *Nano Energy* 1 (1) (2012) 107–131.
- [94] Lamia A. Siddig, Reem H. Alzard, Abdalla S. Abdelhamid, Tholkappian Ramachandran, Ha L. Nguyen, Alejandro Perez Paz, Alzamy Ahmed, Cobalt hydrogen-bonded organic framework as a visible light-driven photocatalyst for CO₂ cycloaddition reaction, *Inorg. Chem.* 62 (38) (2023) 15550–15564, <https://doi.org/10.1021/acs.inorgchem.3c02051>.
- [95] Y. Xie, M. Naguib, V.N. Mochalin, M.W. Barsoum, Y. Gogotsi, X. Yu, K.W. Nam, X. Q. Yang, A.I. Kolesnikov, P.R. Kent, Atomic-scale structural engineering of MXene for energy storage, *J. Am. Chem. Soc.* 136 (17) (2014) 6385–6394.
- [96] L. Cai, Q. Zhang, J.P. Mwizerwa, H. Wan, X. Yang, X. Xu, X. Yao, Novel 2D materials for supercapacitors: synthesis and energy storage performance, *ACS Appl. Mater. Interfaces* 10 (12) (2018) 10053–10063.
- [97] N.S. Mikhaleva, M.A. Visotin, A.A. Kuzubov, Z.I. Popov, Theoretical study on the properties of MXenes for energy applications, *J. Phys. Chem. C* 121 (44) (2017) 24179–24187.
- [98] M. Calis, D. Lloyd, N. Boddeti, J.S. Bunch, Adhesion of 2D MoS₂ to graphite and metal substrates measured by a blister test, *Nano Lett.* 23 (7) (2023) 2607–2614.
- [99] P. Poongavanam, A.A. Chand, V.B. Tai, Y.M. Gupta, M. Kuppusamy, J.A. Dhanraj, K. Velmurugan, R. Rajagopal, T. Ramachandran, K.A. Prasad, S.S. Chand, S. Raj, K.A. Mamun, Annual thermal management of the photovoltaic module to enhance electrical power and efficiency using heat batteries, *Energies* 16 (10) (2023) 4049, <https://doi.org/10.3390/en16104049>.
- [100] M. Garg, A. Thakur, A review: biomedical applications of phosphorene, antimonene, and germanene-based 2D material/hydrogel complexes, *J. Mater. Sci.* 58 (1) (2023) 34–45.
- [101] C. Li, D. Sang, S. Ge, L. Zou, Q. Wang, Recent excellent optoelectronic applications based on two-dimensional WS₂ nanomaterials: a review, *Molecules* 29 (14) (2024).
- [102] K. Padmavathi, S. Supriya, V. Siva, S. Sanjana, A. Murugan, A. Shameem, S. Jhelai, P. Kurinjinathan, R.O.M.U. Jauhar, Core-shell structured lead sulfide encapsulated Co-MOF as a battery-type electrode for supercapattery, *J. Alloys Compd.* 1020 (2025) 179423.
- [103] M. Keerthana, N. Krishna Chandar, Vanadium sulfide decorated graphitic carbon nitride nanocomposite for the enhanced photocatalytic activity towards organic pollutants, *Diam. Relat. Mater.* 129 (2022) 109353.

- [104] N.H. Solangi, N.M. Mubarak, R.R. Karri, S.A. Mazari, J.R. Koduru, Holistic mechanism of graphene oxide and MXene-based membrane for the desalination processes, *Desalination* (2023) 117035.
- [105] S.M. Lee, H.C. Jeong, S.G. Park, Fluorinated silane selfassembled monolayer modification of V_2O_5 for enhanced hole injection and device efficiency, *Opt. Mater.* 152 (2024) 115502.
- [106] Kalaiselvi Chandiran, Muthu Senthil Pandian, Saravanakumar Balakrishnan, Sudhagar Pitchaimuthu, Yong-Song Chen, Krishna Chandar Nagamuthu Raja, $Ti_3C_2T_x$ MXene decorated with $NiMnO_3/NiMn_2O_4$ nanoparticles for simultaneous photocatalytic degradation of mixed cationic and anionic dyes, *Colloids Surf. A Physicochem. Eng. Asp.* 692 (2024) 133888.
- [107] C.I. Idumah, E.E. Obumneme, Novel trends in phosphorene and phosphorene@polymeric nanoarchitectures and applications, *Emerg. Mater.* 6 (3) (2023) 853–874.
- [108] V. Siva, S. Sanjana, A. Murugan, A. Shameem, R.O.M.U. Jauhar, S. Babu, Facile synthesis and asymmetric device fabrication of zeolite like Co-MOF as a promising electrode material with improved cyclic stability, *J. Mater. Sci.: Mater. Electron.* 35 (2024) 75.
- [109] S. Kumar, A. Mirzaei, A. Kumar, M.H. Lee, Z. Ghahremani, T.U. Kim, H.W. Kim, Nanoparticles anchored strategy to develop 2D MoS_2 and $MoSe_2$ based room temperature chemiresistive gas sensors, *Coord. Chem. Rev.* 503 (2024) 215657.
- [110] Y. Li, C. Wang, A. Abdulkayum, L. Feng, Advances in green hydrogen generation based on $MoSe_2$ hybrid catalysts, *Electrochim. Acta* 503 (2024) 144891.
- [111] J. Wang, Y. Hong, Y. Pan, J. Zhu, X. Xu, W.M. Choi, J. Yang, Ni-foam supported Ni $(OH)_2$ sheets decorated by MXene quantum dots for high performance supercapacitors, *Mater. Chem. Phys.* 312 (2024) 128634.
- [112] A. Kumar, M. Kumar, Recent progress in emerging twodimensional Transition-metal dichalcogenides based gas sensors: a review, *IEEE Sens. J.* 24 (2024) 25221–25226.
- [113] C. Tessarek, T. Grieb, F.F. Krause, C. Petersen, A. Karg, A. Hinz, M. Eickhoff, Atomic vs. sub-atomic layer deposition: impact of growth rate on the optical and structural properties of MoS_2 and WS_2 , *2D Mater.* 11 (2) (2024) 025031.
- [114] D. Vijayalakshmi, Tholkappian Ramachandran, G. Jaiganesh, G. Kalpana, Fathalla Hamed, Unlocking the magnetic and half-metallic properties of AMY_2 ($A = Cu, Ag$; $M = Sc, Ti, V, Cr, Mn, Fe$; $Y = S, Se$) compounds in chalcopyrite structure: an Ab initio study for spintronics applications, *Adv. Condens. Matter Phys.* 2024 (2024) 11, <https://doi.org/10.1155/2024/5630225>, 5630225.
- [115] Y. Liu, J. Yu, D. Guo, Z. Li, Y. Su, $Ti_3C_2T_x$ MXene/graphene nanocomposites: synthesis and application in electrochemical energy storage, *J. Alloys Compd.* 815 (2020) 152403.
- [116] M. Oves, K. Umar, I.M. Ismail, M.N.M. Ibrahim (Eds.), *Graphene Quantum Dots: Biomedical and Environmental Sustainability Applications*, Elsevier, 2023.
- [117] B. Gnana Sundara Raj, R.V. Mangalaraja, V. Vinoth, N. Pugazhenthiran, F. V. Herrera, R.O.M.U. Jauhar, S. Anandan, Facile sonochemical synthesis of nanostructured $FeWO_4$ -rGO and $CuCo_2O_4$ nanocomposite for high-rate capability and stable asymmetric $(CuCo_2O_4/FeWO_4$ -rGO) supercapacitors, *J. Alloys Compd.* 968 (2023) 172156.
- [118] S. Sadeghzadeh, L. Badrinezad, K.E. Eshkalak, Mechanical properties of C3N-BN hybrid nanosheets: insights from molecular dynamics simulations, *Diam. Relat. Mater.* 147 (2024) 111323.
- [119] W. Chen, J.T. Li, C. Ge, Z. Yuan, W.A. Algozeeb, P.A. Advincula, J.M. Tour, Turbostratic boron–carbon–nitrogen and boron nitride by flash joule heating, *Adv. Mater.* 34 (33) (2022) 2202666.
- [120] B. Mukherjee, A. Zulkefli, G. Watanabe, T. Taniguchi, Y. Wakayama, S. Nakaharai, Laser-assisted multilevel non-volatile memory device based on 2D van-der-Waals few-layer- ReS_2/h -BN/graphene heterostructures, *Adv. Funct. Mater.* 30 (42) (2020) 2001688.
- [121] J. Theerthagiri, K. Karuppasamy, C. Justin Raj, M.L. Aruna Kumari, L. John Kennedy, G. Maia, N. Vadivel, A. Prasad Murthy, A. Alfantazi, S. Kheawhom, M. Y. Choi, In situ Spectroscopy: Delineating the mechanistic understanding of electrochemical energy reactions, *Prog. Mater. Sci.* 152 (2025) 101451.
- [122] M. Diaz-Lopez, G.L. Cutts, P.K. Allan, D.S. Keeble, A. Ross, V. Pralong, P. A. Chater, Fast operando X-ray pair distribution function using the DRIX electrochemical cell, *J. Synchrotron Radiat.* 27 (5) (2020) 1190–1199.
- [123] K.D. Kumar, N. Roy, T. Ramachandran, I.K. Durga, M.S. Khan, Y.A. Kumar, S. S. Rao, S.W. Joo, Dual $NiFe_2O_4/CoFe_2O_4$ nanosheets compound dynamism for supercharged supercapacitors, *Mater. Sci. Eng. B* 306 (2024) 117444, <https://doi.org/10.1016/j.mseb.2024.117444>.
- [124] B.D. Paulsen, A. Giovannitti, R. Wu, J. Strzalka, Q. Zhang, J. Rivnay, C.J. Takacs, Electrochemistry of thin films with in situ/operando grazing incidence x-ray scattering: bypassing electrolyte scattering for high fidelity time resolved studies, *Small* 17 (42) (2021) 2103213.
- [125] D. Hou, D. Xia, E. Gabriel, J.A. Russell, K. Graff, Y. Ren, H. Xiong, Spatial and temporal analysis of sodium-ion batteries, *ACS Energy Lett.* 6 (11) (2021) 4023–4054.
- [126] K.D. Kumar, Y.A. Kumar, T. Ramachandran, A.A. Al-Kahtani, M.C. Kang, Cactus-like Ni-Co/ $CoMn_2O_4$ composites on Ni foam: unveiling the potential for advanced electrochemical materials for pseudocapacitors, *Mater. Sci. Eng. B* 296 (October 2023) 116715, <https://doi.org/10.1016/j.mseb.2023.116715>.
- [127] R. De, B. Dietzek-Ivancić, A happy Get-Together—Probing electrochemical interfaces by non-linear vibrational spectroscopy, *Chem.–Eur. J.* 28 (55) (2022) e202200407.
- [128] J.A. Bau, A.H. Emwas, P. Nikolaienko, A.A. Aljarb, V. Tung, M. Rueping, Mo^{3+} hydride as the common origin of H_2 evolution and selective NADH regeneration in molybdenum sulfide electrocatalysts, *Nat. Catal.* 5 (5) (2022) 397–404.
- [129] T. Ramachandran, R.M. Naidu Kalla, Y. Anil Kumar, A. Ghosh, A.G. Al-Sehemi, R. Khan, J. Lee, Black phosphorus as a multifunctional electrode material for all energy storage devices, *J. Alloys Compd.* 1037 (2025) 182500.
- [130] I.K. Durga, N. Roy, T. Ramachandran, K.D. Kumar, S. Ansari, Y.A. Kumar, S.S. Rao, S.W. Joo, Innovative synthesis strategies for rambutan-shaped $CuNiO_2$ electrodes in high-performance supercapacitors, *J. Phys. Chem. Solids* 192 (2024) 112117, <https://doi.org/10.1016/j.jpcs.2024.112117>.
- [131] Z. Stević, Z. Andjelković, D. Antić, A new PC and LabVIEW package based system for electrochemical investigations, *Sensors* 8 (3) (2008) 1819–1831.
- [132] A.N. Morozovska, E.A. Eliseev, N. Balke, S.V. Kalinin, Local probing of ionic diffusion by electrochemical strain microscopy: spatial resolution and signal formation mechanisms, *J. Appl. Phys.* 108 (5) (2010).
- [133] S.C. Lai, J.V. Macpherson, P.R. Unwin, In situ scanning electrochemical probe microscopy for energy applications, *MRS Bull.* 37 (7) (2012) 668674.
- [134] S. Karthickprabhu, P. Sundararajaperumal, M. Mahendran, D. Vikraman, S. Hussain, K. Ahmed Alhebsi, A.A. Alhammadi, H.-S. Kim, K. Karuppasamy, A. Alfantazi, Revealing the electrochemical merits of coral-reef-like nickel-doped mixed metal-organic framework composites as advanced supercapacitor electrodes, *J. Power Sources* 648 (2025) 237378.
- [135] H. Oberacher, F. Pittler, R. Erb, S. Plattner, Mass spectrometric methods for monitoring redox processes in electrochemical cells, *Mass Spectrom. Rev.* 34 (1) (2015) 64–92.
- [136] Tholkappian Ramachandran, Fathalla Hamed, Fabrication of dual-1D/2D shaped $ZnCo_2O_4$ -ZnO electrode material for highly efficient electrochemical supercapacitors, *J. Phys. Chem. Solid.* 188 (2024) 111915.
- [137] L. Collins, J. Kilpatrick, S.V. Kalinin, B.J. Rodríguez, Nanoscale electrical measurements in liquids using AFM-progress and outlook, *arXiv* (2017), <https://doi.org/10.48550/arXiv.1701.06633> preprint arXiv:1701.06633.
- [138] M. Czagany, S. Homphoth, A. Kumar Keshri, N. Pandit, I. Galambos, Z. Gacsi, P. Baumli, Supercapacitors: An efficient way for energy storage application, *Energy Rep.* 10 (2024) 1173–1184.
- [139] K. Karuppasamy, J. Lin, D. Vikraman, S. Hussain, M. Ramu, S. Alameri, H.-S. Kim, J.G. Korvink, A. Alfantazi, B. Sharma, Unveiling the electrochemical excellence of sulfur and nitrogen-enriched 3D porous carbon nanofibers in high-performance energy storage devices, *J. Ind. Eng. Chem.* 142 (2025) 187–197.
- [140] T. Ramachandran, R.M. Naidu Kalla, A.G. Al-Sehemi, Y. Anil Kumar, R. Khan, A. Ghosh, S.S. Rao, J. Lee, Beyond traditional MXenes: The promise of non-Ti MXenes in energy and catalysis, *Colloids Surf. A: Physicochem. Eng. Asp.* 726 (2025) 137844.
- [141] N. Hallemans, D. Howey, A. Battistel, N.F. Saniee, F. Scarpioni, B. Wouters, J. Lataire, Electrochemical impedance spectroscopy beyond linearity and stationarity—A critical review, *Electrochim. Acta* (2023) 142939.
- [142] D. Yan, M.Z. Bazant, P.M. Biesheuvel, M.C. Pugh, F.P. Dawson, Theory of linear sweep voltammetry with diffuse charge: unsupported electrolytes, thin films, and leaky membranes, *Phys. Rev.* 95 (3) (2017) 033303.
- [143] C.L. Bentley, J. Edmondson, G.N. Meloni, D. Perry, V. Shkirskiy, P.R. Unwin, Nanoscale electrochemical mapping, *Anal. Chem.* 91 (1) (2018) 84–108.
- [144] Tholkappian Ramachandran, Abdel-Hamid Ismail Mourad, Mostafa S. A. ElSayed, Nb_2CT_x -based MXenes most recent developments: from principles to new applications, *Energies* 16 (8) (2023) 3520, <https://doi.org/10.3390/en16083520>.
- [145] R. Mondal, N.B. Singh, J. Deb, S. Mukherjee, U. Sarkar, Electronic and transport property of two-dimensional boron phosphide sheet, *J. Mol. Graph. Model.* 112 (2022) 108117.
- [146] S. Pluczyk-Malek, D. Nastula, D. Honisz, M. Lapkowski, P. Data, P. Wagner, s-Tetrazine donor-acceptor electrodeposited layer with properties controlled by doping anions generally considered as interchangeable, *Electrochim. Acta* 405 (2022) 139788.
- [147] Tholkappian Ramachandran, Selvi Natarajan, Fathalla Hamed, The role of dysprosium levels in the formation of mixed oxidation states within spinel $MnCo_{2-x}Dy_xO_4$ nanocrystalline powders, *J. Electron. Spectrosc. Relat. Phenom.* 242 (2020) 146952, <https://doi.org/10.1016/j.elspec.2020.146952>.
- [148] D.M. Morales, M. Risch, Seven steps to reliable cyclic voltammetry measurements for the determination of double layer capacitance, *J. Phys.: Energy* 3 (3) (2021) 034013.
- [149] C. Yun, S. Hwang, Analysis of the charging current in cyclic voltammetry and supercapacitor's galvanostatic charging profile based on a constant phase element, *ACS Omega* 6 (1) (2020) 367–373.
- [150] H.H. Hegazy, Siva Sankar Sana, Tholkappian Ramachandran, Yedluri Anil Kumar, Dasha Kumar Kulurumotlakatla, Hisham S.M. Abd-Rabboh, Seong Cheol Kim, Covalent organic frameworks in supercapacitors: unraveling the pros and cons for energy storage, *J. Energy Storage* 74 (Part A) (2023) 109405, <https://doi.org/10.1016/j.est.2023.109405>.
- [151] N. Bergmann, N.G. Hörmann, K. Reuter, Ab initio-based modeling of thermodynamic cyclic voltammograms: a benchmark study on Ag (100) in bromide solutions, *J. Chem. Theor. Comput.* 19 (23) (2023) 88158825.
- [152] M.E. Holtz, Y. Yu, D. Gunceler, J. Gao, R. Sundararaman, K.A. Schwarz, T. A. Arias, H.D. Abruña, D.A. Muller, Nanoscale imaging of lithium ion distribution during in situ operation of battery electrode and electrolyte, *Nano Lett.* 14 (3) (2014) 1453–1459.
- [153] R. Tholkappian, K. Vishista, Tuning the composition and magnetostucture of dysprosium iron garnets by Co-substitution: an XRD, FT-IR, XPS and VSM study, *Appl. Surf. Sci.* 351 (2015) 1016–1024, <https://doi.org/10.1016/j.apusc.2015.05.193>.
- [154] Z.J. Barton, Spatially resolved ionic measurements with scanning electrochemical microscopy, *Electrochim. Acta* 241 (2017) 446–459.

- [155] Z. Lin, P.L. Taberna, P. Simon, Advanced analytical techniques to characterize materials for electrochemical capacitors, *J. Mater. Chem. A* 6 (7) (2018) 2929–2947.
- [156] D.M. Morales, M. Risch, Seven steps to reliable cyclic voltammetry measurements for the determination of double-layer capacitance, *Electrochem. Commun.* 114 (2020) 106734.
- [157] C. Yun, S. Hwang, Analysis of the charging current in cyclic voltammetry and supercapacitor's galvanostatic charging profile based on a constant phase element, *Electrochim. Acta* 341 (2020) 136035.
- [158] N. Maaoui, P. Rosaiah, K. Ganesh Kumar, M. Ammal Dhanalakshmi, M. D. Albaqami, R. Ayub, Enhanced performance of DSSC by incorporating a Red Emitting MgAl_2O_4 : Eu^{3+} phosphor in the TiO_2 Layer, *Opt. Mater.* 151 (2024) 115349, <https://doi.org/10.1016/j.optmat.2024.115349>.
- [159] M. Philip, J.S. Punitha, R. Rameshkumar, et al., A novel double perovskite oxide $\text{Sm}_2\text{CoFeO}_6$ phosphor for orange LEDs: structural, magnetic and luminescence properties, *Appl. Phys. A* 128 (2022) 324, <https://doi.org/10.1007/s00339-022-05446-8>.
- [160] K. Gallegos Moncayo, N. Folastre, M. Toledo, H. Tonnoir, F. Rabuel, G. Gachot, D. Huo, A. Demortière, Coupling liquid electrochemical TEM and mass spectrometry to investigate electrochemical reactions occurring in a Na-ion battery anode, *Nat. Commun.* 14 (1) (2023) 3101.
- [161] M. Czagany, S. Hompoth, A.K. Keshri, N. Pandit, I. Galambos, Z. Gacsi, P. Baumli, Supercapacitors: an efficient way for energy storage applications, *J. Power Sources* 558 (2024) 232489.
- [162] M.E. Holtz, Y. Yu, D. Gunceler, J. Gao, R. Sundaraman, K.A. Schwarz, T. A. Arias, H.D. Abruña, D.A. Muller, Nanoscale Imaging of Lithium Ion Distribution During in Situ Operation of Battery Electrode and Electrolyte, 2013.
- [163] Arumugam Raja, Ro Mu Jauhar, Kasthuri Ramachandran, Sivasubramani Vedyappan, Kumar Raji Ramesh, Muthu Senthil Pandian, Ramasamy Perumalsamy, A quintuple-layered binary chalcogenide Sb_2Te_3 single crystal and its transport properties for thermoelectric applications, *ACS Omega* 7 (32) (2022) 27798–27803, <https://doi.org/10.1021/acsomega.1c05972>.
- [164] S. Taminato, M. Yonemura, S. Shiotani, T. Kamiyama, S. Torii, M. Nagao, Y. Ishikawa, K. Mori, T. Fukunaga, Y. Onodera, T. Naka, M. Morishima, D. S. Adipranoto, H. Arai, Y. Uchimoto, Z. Ogumi, K. Suzuki, M. Hirayama, R. Kanno, Real-time observations of lithium battery reactions—Operando neutron diffraction analysis during practical operation, *Sci. Rep.* 6 (1) (2016) 28843.
- [165] N. Hema, K. Ganesh Kumar, M. Ammal Dhanalakshmi, M. Abbas, M. Kuppusamy, Impact of Dy^{3+} and Ce^{3+} ion doping on the photoluminescence, thermoluminescence, and electrochemical properties of strontium aluminate (SrAl_2O_4) and barium aluminate (BaAl_2O_4) phosphors, *J. Mater. Sci.: Mater. Electron.* 35 (2024) 923, <https://doi.org/10.1007/s10854-024-12571-y>.
- [166] Z. Lin, P.L. Taberna, P. Simon, Advanced analytical techniques to characterize materials for electrochemical capacitors, *Adv. Energy Mater.* 8 (1) (2018) 1701456.
- [167] M.J. Osiaik, H. Geaney, E. Armstrong, C. O'Dwyer, Structuring materials for lithium-ion batteries: advancements in nanomaterial structure, composition, and defined assembly on cell performance, *J. Mater. Chem. A* 6 (14) (2018) 6201–6235.
- [168] A. Stangl, D. Muñoz-Rojas, M. Burriel, *In situ* and *operando* characterisation techniques for solid oxide electrochemical cells: recent advances, *J. Mater. Chem. A* 8 (20) (2020) 10073–10100.
- [169] G. Qian, J. Wang, H. Li, Z.F. Ma, P. Pianetta, L. Li, X. Yu, Y. Liu, Structural and chemical evolution in layered oxide cathodes of lithium-ion batteries revealed by synchrotron techniques, *Nat. Commun.* 12 (1) (2021). Article 2510.
- [170] D. Hou, D. Xia, E.J. Gabriel, J.A. Russell, K. Graff, Y. Ren, C.J. Sun, F. Lin, Y. Liu, H. Xiong, Spatial and temporal analysis of sodium-ion batteries, *Nat. Commun.* 12 (1) (2021). Article 2823.
- [171] Y.A. Kumar, S.S. Sana, T. Ramachandran, M.A. Assiri, S.S. Rao, S.C. Kim, From lab to field: prussian blue frameworks as sustainable cathode materials, *Dalton Trans.* (2024), <https://doi.org/10.1039/D4DT00905C>. Advance Article.
- [172] A.P. Black, A. Sorrentino, F. Fauth, I. Yousef, L. Simonelli, C. Frontera, A. Ponrouch, D. Tonti, M.R. Palacin, Synchrotron radiation-based *operando* characterization of battery materials, *J. Mater. Chem. A* 10 (30) (2022) 16167–16179.
- [173] Tholkappian Ramachandran, Abdel-Hamid I. Mourad, Ramesh Kumar Raji, Ramachandran Krishnapriya, Nizamudeen Cherupurakal, Abdul Subhan, Yarub Al-Douri, KOH mediated hydrothermally synthesized hexagonal- CoMn_2O_4 for energy storage supercapacitor applications, *Int. J. Energy Res.* 46 (12) (2022) 16823–16838, <https://doi.org/10.1002/er.8350>.
- [174] N. Karthikeyan, R. Ramesh Kumar, G. Jaiganesh, K. Sivakumar, Thermoelectric power factor of $\text{La}_{0.9}\text{M}_{0.1}\text{FeO}_3$ ($\text{M} = \text{Ca}$ and Ba) system: structural, band gap and electrical transport evaluations, *Phys. B Condens. Matter* 529 (2018) 1–8, <https://doi.org/10.1016/j.physb.2017.09.131>, 0921–4526.
- [175] A. Stoll, P. Benner, Machine learning for material characterization with an application for predicting mechanical properties, *Sci. Rep.* 10 (1) (2020). Article 2952.
- [176] S. Yang, Y. Li, Q. Chen, Resolving electron transfer kinetics in porous electrodes via diffusion-less cyclic voltammetry, *J. Mater. Chem. A* 9 (24) (2021) 14025–14031.
- [177] Ramesh Kumar Raji, Naser Qamhie, Adel Najjar, Falah Awwad, Adnan Younis, Saleh T. Mahmoud, Next-generation electrochemical etching for III-nitride semiconductors: innovations, applications, and beyond, *Nanoscale* 17 (2025) 19528–19570, <https://doi.org/10.1039/D5NR01807B>.
- [178] S. Senthilnathan, K. Ganesh Kumar, S. Sugunraj, M. Ammal Dhanalakshmi, M. Rajendraprasad, M. Suganthi, K. ShreeKeshavan, P. Sasikumar, M. Abbas, W.L. T. Dator, MoS_2 modified g- C_3N_4 composite: A potential candidate for photocatalytic applications, *J. Saudi Chem. Soc.* 27 (2023) 101717, <https://doi.org/10.1016/j.jscs.2023.101717>.
- [179] V. Janakiraman, M. Savio, A.N. Alodhayb, M. Muthuramamoorthy, M. Ammal Dhanalakshmi, M. Vimalan, M. Durai, K. Ganesh Kumar, Impact of LLZO electrolytes doped with Ta^{5+} and their structural and electrical characteristics for solid-state energy storage applications, *J. Mater. Sci.: Mater. Electron.* 36 (2025) 309, <https://doi.org/10.1007/s10854-025-14362-5>.
- [180] J. Qu, X. Liu, Recent advances on SEM-based in situ multiphysical characterization of nanomaterials, *Nanotechnology* 32 (16) (2021). Article 162001.
- [181] E.M. Holtz, Y. Yu, D. Gunceler, J. Gao, R. Sundaraman, K.A. Schwarz, T. A. Arias, H.D. Abruña, D. Muller, Nanoscale imaging of lithium ion distribution during in situ operation of battery electrode and electrolyte, *Nat. Commun.* 4 (1) (2013). Article 1009.
- [182] A. Arulraj, R.V. Mangalajaya, V. Siva, R.O.M.U. Jauhar, Impact of sonochemical and chemical precipitated synthesis of porous nickel manganite (NiMn_2O_4) electrodes for supercapacitor applications, *Mater. Lett.* 403 (2025) 139515.
- [183] A. Berrueta Irigoyen, A. Ursúa Rubio, I. San Martín Biurrun, A. Eftekhari, P. Sanchis Gúrpide, Supercapacitors: electrical characteristics, modelling, applications and future trends, *Renew. Sustain. Energy Rev.* 103 (2019) 108–120.
- [184] R. Ramachandran, T.W. Chen, S.M. Chen, P. Raja, C. Fernandez, S. Divya Rani, P. Gajendran, G. Raju, T. Baskar, T. Jeyapragasam, Highly enhanced electrochemical performance of novel based electrode materials for supercapacitor applications: an overview, *J. Energy Chem.* 36 (2019) 111–120.
- [185] S. Najib, E. Erdem, Current progress achieved in novel materials for supercapacitor electrodes: mini review, *J. Mater. Sci. Mater. Electron.* 30 (15) (2019) 13681–13697.
- [186] Gita B. Bhanuse, Sanath Kumar, Cheng-We Chien, Yen-Pei Fu, Development of heterostructured ZnCo_2O_4 @Ni-MOF electrode for the asymmetric supercapacitor and electrocatalytic oxygen evolution reaction applications, *Electrochim. Acta* 511 (2025) 145371, <https://doi.org/10.1016/j.electacta.2024.145371>.
- [187] Abdulkadeem Sanni, Durai Govindarajan, Wathanyu Kao-ian, Wanwisa Limphirat, Mongkol Tipplook, Katsuya Teshima, Jayaraman Theerthagiri, Myong Yong Choi, Soorathep Kheawhom, Elucidating $\text{Mn}^{2+}/\text{Mn}^{3+}$ and $\text{NiO}/\text{Ni}^{2+}$ redox synergy in hair-derived carbon-supported Ag/Ni- MnO_x supercapacitor, *ACS Appl. Mater. Interfaces* 17 (33) (2025) 46936–46951, <https://doi.org/10.1021/acsaami.5c07064>.
- [188] D. Hou, D. Xia, E.A. Gabriel, J.A. Russell, K. Graff, Y. Ren, C.J. Sun, F. Lin, Y. Liu, H. Xiong, Spatial and temporal analysis of sodium-ion batteries, *Nat. Commun.* 12 (1) (2021). Article 6501.
- [189] D. Vikraman, S. Hussain, K. Karuppasamy, A. Sanmugam, A. Kathalingam, R. Manikandan, J. Jung, A. Alfanzati, H.-S. Kim, Development of CoTe_2 , CoS_2 , and CoSe_2 electrode material for the asymmetric supercapacitors, *Int. J. Energy Res.* 2025 (2025) 5553294.
- [190] P.A. Black, A. Sorrentino, F. Fauth, I. Yousef, L. Simonelli, C. Frontera, A. Ponrouch, D. Tonti, M. Rosa Palacin, Synchrotron radiation based operando characterization of battery materials, *Nat. Rev. Chem.* 6 (2022) 265–282.
- [191] H. Jin, Y. Dai, B.B. Huang, Design of advanced photocatalysis system by adatom decoration in 2D nanosheets of group-IV and III-V binary compounds, *J. Phys. Appl. Phys.* 49 (38) (2016) 383001.
- [192] R.D. Ryder, C.D. Wood, A. Wells, M.C. Hersam, Chemically tailoring semiconducting two-dimensional transition metal dichalcogenides and black phosphorus, *Chem. Soc. Rev.* 45 (6) (2016) 1522–1534.
- [193] M. Czagany, S. Hompoth, A. Kumar Keshri, N. Pandit, I. Galambos, Z. Gacsi, P. Baumli, Supercapacitors: An efficient way for energy storage application, *Energy Rep.* 10 (2024) 1173–1184.
- [194] A. Hirsch, F. Hauke, Post-graphene 2D chemistry: the emerging field of molybdenum disulfide and black phosphorus functionalization, *Angew. Chem. Int. Ed.* 57 (1) (2018) 45–60.
- [195] S.J. Fretz, Adding utility to carbon materials: introducing dopants using highly soluble metal salts and functionalizing surfaces via bromomethylation, *Carbon* 146 (2019) 297–303.
- [196] R. Niruban Bharathi, T. Ramachandran, G. Rekha, Investigation of mixed electronic states in $\text{Dy}_3\text{Fe}_5\text{O}_{12}$ produced by doping with nickel, *J. Clust. Sci.* 35 (2024) 545–560, <https://doi.org/10.1007/s10876-023-02494-9>.
- [197] G. Guan, M.Y. Han, Functionalized hybridization of 2D nanomaterials, *Adv. Mater.* 31 (30) (2019) 1900725.
- [198] M. Lihter, M. Graf, D. Iveković, T.H. Shen, Y. Zhao, V. Tileli, A. Radenovic, Electrochemical surface modification of a 2D MoS_2 semiconductor, *J. Phys. Chem. C* 124 (2) (2020) 1315–1320.
- [199] X. Ma, N. Youngblood, X. Liu, Y. Cheng, P. Cunha, K. Kudtarkar, X. Wang, S. Lan, Engineering photonic environments for two-dimensional materials, *Adv. Mater.* 32 (23) (2020) 2001101.
- [200] N. Liu, L. Chang, J. Wu, J. Fang, X. Xiao, Emerging topochemical strategies for designing two-dimensional energy materials, *Energy Environ. Sci.* 14 (5) (2021) 3023–3048.
- [201] V. Govindan, Arumugam Raja, Gour Mohan Das, D. Joseph Daniel, R. Ramesh Kumar, K. Sankaranarayanan, Synthesis and luminescence properties of $\text{Ca}_3\text{Ga}_2\text{O}_9$: Eu^{3+} : an efficient red-emitter for WLEDs, *Ceram. Int.* 49 (11) (2023) 17566–17576, <https://doi.org/10.1016/j.ceramint.2023.02.123>.
- [202] G. Armstrong, L. Kailas, Hyphenated analytical techniques for materials characterisation, *J. Mater. Sci.* 53 (1) (2018) 1–11.
- [203] D. Moss, Fabrication methods for integrating 2D materials, *Nat. Rev. Mater.* 7 (7) (2022) 530–544.

- [204] K. Karuppasamy, G.K. Veerasubramani, V. Hiremath, D. Vikraman, P. Santhoshkumar, G.N. Karanikolos, A.A. Alhammedi, H.-S. Kim, A. Alfantazi, Unlocking recent progress in niobium and vanadium carbide-based MXenes for sodium-ion batteries, *J. Mater. Chem. A* 13 (2025) 1590–1611.
- [205] R.D. Ryder, C.D. Wood, A. Wells, M.C. Hersam, Chemically tailoring semiconducting two-dimensional transition metal dichalcogenides and black phosphorus, *Chem. Soc. Rev.* 45 (6) (2016) 1522–1534.
- [206] S. Kartik, D. Prakash, R.R. Kumar, et al., Betel leaf-derived carbon nano-sphere/renewable resourced polyurethane coatings for high-performance applications, *Polym. Bull.* 78 (2021) 3527–3541, <https://doi.org/10.1007/s00289-020-03264-7>.
- [207] M. Vazquez Sulleiro, Covalent functionalization of graphene derivatives for novel carbon interfaces, *Carbon* 132 (2018) 204–223.
- [208] N. Briggs, S. Subramanian, Z. Lin, X. Li, X. Zhang, K. Zhang, K. Xiao, D. Geohagan, R. Wallace, L.Q. Chen, M. Terrones, A. Ebrahimi, S. Das, J. Redwing, C. Hinkle, K. Momeni, A. van Duin, V. Crespi, S. Kar, A. Robinson, A roadmap for electronic grade two-dimensional materials, *Adv. Mater.* 30 (34) (2018) 1802070.
- [209] M. Dhillip, K. Saravana Kumar, R. Ramesh Kumar, et al., Intrinsic magnetic and ferroelectric behaviour of non-magnetic Al^{3+} ion substituted dysprosium iron garnet compounds, *J. Electron. Mater.* 48 (2019) 8243–8253, <https://doi.org/10.1007/s11664-019-07675-9>.
- [210] A. Hirsch, F. Hauke, Post-graphene 2D chemistry: the emerging field of molybdenum disulfide and black phosphorus functionalization, *Angew. Chem. Int. Ed.* 58 (27) (2019) 9080–9091.
- [211] E.M. Holtz, Y. Yu, D. Gunceler, J. Gao, R. Sundaraman, K.A. Schwarz, T. A. Arias, H.D. Abruña, D. Muller, Nanoscale imaging of lithium ion distribution during in situ operation of battery electrode and electrolyte, *Nat. Commun.* 4 (1) (2013) 1009.
- [212] G. Guan, M.Y. Han, Functionalized hybridization of 2D nanomaterials, *Adv. Mater.* 31 (30) (2019) 1900725.
- [213] A. Kröner, T. Hirsch, Current trends in the optical characterization of two-dimensional carbon nanomaterials, *Small* 16 (1) (2020) 1904547.
- [214] N. Liu, L. Chang, J. Wu, J. Fang, X. Xiao, Emerging topochemical strategies for designing two-dimensional energy materials, *Energy Environ. Sci.* 14 (5) (2021) 3023–3048.
- [215] R. Suriakarthick, M. Senthil Pandian, P. Ramasamy, Ramesh Kumar Raji, M. Muralidharan, C.K. Amaljith, Suresh Sagadevan, Solvothermal synthesis, structural and transport properties of polycrystalline copper tin selenide for thermoelectric applications, *Inorg. Chem. Commun.* 140 (2022) 1387–7003, <https://doi.org/10.1016/j.inoche.2022.109491>, 109491.
- [216] C. Kalaiselvi, Karthik Kannan, Muthu Senthil Pandian, Nitika Devi, Amornchai Arpornwichanop, N. Krishna Chandar, Yong-Song Chen, $\text{Ti}_3\text{C}_2\text{X}$ MXene decorated with $\text{CoMnO}_3/\text{CoMn}_2\text{O}_4$ nanoparticles as electrocatalysts for anion exchange membrane water electrolysis, *Int. J. Hydrogen Energy* 137 (2025) 202–213, <https://doi.org/10.1016/j.ijhydene.2025.04.402>.
- [217] U. Celano, D. Schmidt, C. Beitia, G. Orji, A.V. Davydov, Y. Obeng, Metrology for 2D materials: a perspective review from the international roadmap for devices and systems, *Nat. Rev. Phys.* 6 (1) (2024) 56–69.
- [218] J.S. Punitha, R.K. Raji, K.S. Kumar, et al., Investigating the impact of Gd^{3+} ion substitution in $\text{Sr}_2\text{FeTiO}_6$: insights into magnetic, electrical, and structural properties, *Appl. Phys. A* 130 (2024) 825, <https://doi.org/10.1007/s00339-024-08012-6>.
- [219] A. Stangl, D. Muñoz-Rojas, M. Burriel, In situ and operando characterisation techniques for solid oxide electrochemical cells: recent advances, *J. Mater. Chem. A* 8 (11) (2020) 5294–5321.
- [220] A. Favron, E. Gaufres, F. Fossard, A.L. Phaneuf-L'Heureux, N.Y. Tang, P. L. Lévesque, A. Loiseau, R. Leonelli, S. Francoeur, R. Martel, Photooxidation and quantum confinement effects in exfoliated black phosphorus, *Nat. Mater.* 14 (2015) 826–832, <https://doi.org/10.1038/nmat4295>.
- [221] Y.C. Lin, D.O. Dumcenco, H.P. Komsa, Y. Niimi, A.V. Krasneninnikov, Y.S. Huang, K. Suenaga, Properties of individual dopant atoms in single-layer MoS_2 : atomic structure, migration, and enhanced reactivity, *ACS Nano* 8 (2014) 3715–3723, <https://doi.org/10.1021/nn500064s>.
- [222] M. Naguib, M. Kurtoglu, V. Presser, J. Lu, J. Niu, M. Heon, L. Hultman, Y. Gogotsi, M.W. Barsoum, Two-dimensional nanocrystals produced by exfoliation of Ti_3AlC_2 , *ACS Nano* 6 (2012) 1322–1331, <https://doi.org/10.1021/nn204153h>.
- [223] M. Chhowalla, H.S. Shin, G. Eda, L.J. Li, K.P. Loh, H. Zhang, The chemistry of two-dimensional layered transition metal dichalcogenide nanosheets, *Nat. Chem.* 5 (2013) 263–275, <https://doi.org/10.1038/nchem.1589>.
- [224] H. Zhou, F. Yu, J. Sun, Q. Zhu, F. Qin, J. Bao, L. Yu, S. Chen, Z. Ren, Highly active catalyst derived from Mo-Doped Co_3O_4 nanowire arrays for hydrogen evolution reaction in neutral medium, *Adv. Energy Mater.* 8 (2018) 1701905, <https://doi.org/10.1002/aenm.201702844>.
- [225] S. Vijayaraghavan, A. Rajasekaran, A.N. Alodhayb, M. Muthuramamoorthy, M. Vimalan, K. Ganesh Kumar, Synthesis and enhanced electrical properties of Ag-doped $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles in PVA films for nanoelectronic applications, *Mater. Sci. Eng.: B* 311 (2025) 117801, <https://doi.org/10.1016/j.mseb.2024.117801>.
- [226] D. Voiry, A. Mohite, M. Chhowalla, Phase engineering of transition metal dichalcogenides, *Chem. Soc. Rev.* 44 (2015) 2702–2712, <https://doi.org/10.1039/C5CS00357A>.
- [227] D. Voiry, H. Yamaguchi, J. Li, R. Silva, D.C.B. Alves, T. Fujita, M. Chen, T. Asefa, V.B. Shenoy, G. Eda, M. Chhowalla, Enhanced catalytic activity in Strained chemically exfoliated WS_2 nanosheets for hydrogen evolution, *Nat. Mater.* 15 (2016) 1003–1009, <https://doi.org/10.1038/nmat4475>.
- [228] A. Rajasekaran, R. Usha, A.N. Alodhayb, M. Muthuramamoorthy, S. Senthilnathan, B. Chandrasekaran, M. Tamilselvi, K. Ganesh Kumar, Enhancing structural, morphological, optical, and photocatalytic properties of ZnSe nanostructures via PTH-rGO integration, *Synth. Met.* 311 (2025) 117815, <https://doi.org/10.1016/j.synthmet.2024.117815>.
- [229] D. Akinwande, C.J. Brennan, J.S. Bunch, P. Egberts, J.R. Felts, H. Gao, R. Huang, J.S. Kim, T. Li, Y. Li, K.M. Liechti, N. Lu, H.S. Park, E.J. Reed, P. Wang, B. I. Yakobson, T. Zhang, Y.W. Zhang, Y. Zhu, A review on mechanics and mechanical properties of 2D materials—graphene and beyond, *ACS Nano* 11 (2017) 9710–9733, <https://doi.org/10.1021/nn501226z>.
- [230] R. Kameshwaran, A. Raja, R. Ramesh Kumar, D. Joseph Daniel, D. O. Annalakshmi, K. Aravinth, P. Balaji Bhargav, P. Ramasamy, Synthesis, structure and luminescence properties of bifunctional KCaF_3 phosphor influenced by incorporating Eu^{3+} ions for solid state lighting and TL dosimetry applications, *Appl. Radiat. Isot.* 191 (2023) 969–8043, <https://doi.org/10.1016/j.apradiso.2022.110520>, 110520.
- [231] G.R. Bhimanapati, Z. Lin, V. Meunier, Y. Jung, J. Cha, S. Das, D. Xiao, Y. Son, M. S. Strano, V.R. Cooper, L. Liang, S.G. Louie, E. Ringe, W. Zhou, S.S. Kim, R. R. Naik, B.G. Sumpter, H. Terrones, F. Xia, Y. Wang, J. Zhu, D. Akinwande, N. Alem, J.A. Schuller, R.E. Schaak, M. Terrones, J.A. Robinson, Recent advances in two-dimensional materials beyond graphene, *ACS Nano* 9 (2015) 11509–11539, <https://doi.org/10.1021/acsnano.5b05556>.
- [232] H. Cheng, Y. Zhao, Y. Zhu, P. Tao, W. Shang, C. Song, T. Deng, Smart electrochemical energy storage devices based on 2D materials: from design to applications, *Adv. Energy Mater.* 10 (2020) 1902834, <https://doi.org/10.1002/aenm.201902814>.
- [233] C. Zhang, L. McKeon, M.P. Kremer, S.H. Park, O. Ronan, A. Seral-Ascaso, S. Barwich, C.O. Coileáin, N. McEvoy, H.C. Nerl, B. Anasori, J.N. Coleman, Y. Gogotsi, Additive-free MXene inks and direct printing of micro-supercapacitors, *Nat. Commun.* 10 (2019) 1795, <https://doi.org/10.1038/s41467-019-09606-7>.
- [234] Tholkappian Ramachandran, Ramesh Kumar Raji, Santhoshkumar Palanisamy, N. Renuka, K. Karuppasamy, The role of in situ and operando techniques in unraveling local electrochemical supercapacitor phenomena, *J. Ind. Eng. Chem.* (2024), <https://doi.org/10.1016/j.jiec.2024.10.077>.
- [235] R. Ghosh, N. Pramanik, Understanding the surface chemistry and electrochemical behavior of 2D TMDs: role of defects, edges, and basal planes, *Electrochim. Acta* 419 (2022) 140363, <https://doi.org/10.1016/j.electacta.2022.140363>.
- [236] S. Bertolazzi, J. Brivio, A. Kis, Stretching and breaking of ultrathin MoS_2 , *ACS Nano* 5 (2011) 9703–9709, <https://doi.org/10.1021/nn203879f>.
- [237] A.M. Bogale, T. Ramachandran, L.T. Tufa, B.B. Badassa, M.E. Suk, R. Pitcheri, J. Lee, S.K. Jilcha, A.Y. Tiky, B.A. Zenebe, N.K. Amare, M.M. Solomon, F. B. Tesema, Structurally engineered $\text{MoS}_2/\text{CuCo}_2\text{O}_4$ with palm-leaf morphology for button-type supercapacitor applications, *Mater. Sci. Semicond. Process.* 200 (2025) 109958.
- [238] J. Shen, M. Shi, N. Li, B. Yan, H. Ma, Y. Hu, M. Ye, Fast and facile preparation of graphene oxide and reduced graphene oxide nanoplatelets, *Chem. Mater.* 21 (2009) 3514–3520, <https://doi.org/10.1021/cm901247t>.
- [239] R. Mangiri, T. Ramachandran, Y. Anil Kumar, A. Ghosh, A.G. Al-Sehemi, A. K. Yadav, D. Mani, Surface engineering of M_5X_4 MXenes for next-gen energy solutions, *Mater. Today Chem.* 48 (2025) 102864.
- [240] X. Zhang, Z. Lai, C. Tan, H. Zhang, Solution-processed two-dimensional MoS_2 nanosheets: preparation, hybridization, and applications, *Angew. Chem. Int. Ed.* 55 (2016) 8816–8838, <https://doi.org/10.1002/anie.201509946>.
- [241] X. Wang, H. Feng, Y. Wu, L. Jiao, Controlled synthesis of highly crystalline MoS_2 flakes by chemical vapor deposition, *J. Am. Chem. Soc.* 135 (2013) 5304–5307, <https://doi.org/10.1021/ja400693z>.
- [242] C. Tan, X. Cao, X.J. Wu, Q. He, J. Yang, X. Zhang, J. Chen, W. Zhao, S. Han, G. H. Nam, M. Sindoro, H. Zhang, Recent advances in ultrathin two-dimensional nanomaterials, *Chem. Rev.* 117 (2017) 6225–6331, <https://doi.org/10.1021/acs.chemrev.6b00558>.
- [243] H. Yuan, H. Wang, B. Lian, H. Zhang, X. Fang, B. Shen, Y. Xu, A.R. Hight Walker, Y. Cui, Generation and electric control of Spin–valley-coupled circular photogalvanic current in WSe_2 , *Nat. Nanotechnol.* 9 (2014) 851–857, <https://doi.org/10.1038/nnano.2014.182>.
- [244] Q.H. Wang, K. Kalantar-Zadeh, A. Kis, J.N. Coleman, M.S. Strano, Electronics and optoelectronics of two-dimensional transition metal dichalcogenides, *Nat. Nanotechnol.* 7 (2012) 699–712, <https://doi.org/10.1038/nnano.2012.193>.
- [245] C. Liu, Y. Wang, J. Li, W. Liu, Y. Gong, J. He, Understanding interfacial behavior in 2D material composites for electrochemical applications, *Adv. Funct. Mater.* 31 (2021) 2009835, <https://doi.org/10.1002/adfm.202009835>.
- [246] C. Zou, C. Chang, D. Sun, K.F. Böhringer, L.Y. Lin, Photolithographic patterning of perovskite thin films for multicolor display applications, *Nano Lett.* 20 (2020) 3710–3717.
- [247] M.J. Sun, C. Zheng, Y. Gao, A. Johnston, A.M. Najarian, P.X. Wang, O. Voznyy, S. Hoogland, E.H. Sargent, Hysteresis-less, high open-circuit voltage perovskite solar cells with minimized charge recombination via surface passivation, *Adv. Mater.* 33 (2021) e2006368.
- [248] S.F. Leung, K.T. Ho, P.K. Kung, V.K.S. Hsiao, H.N. Alshareef, Z.L. Wang, J.H. He, A self-powered and flexible organometallic halide perovskite photodetector with very high detectivity, *Adv. Mater.* 30 (2018) 1704611.
- [249] B. Jeong, I. Hwang, S.H. Cho, E.H. Kim, S. Cha, J. Lee, H.S. Kang, S.M. Cho, H. Choi, C. Park, Solvent-assisted gel printing for micropatterning thin organic–inorganic hybrid perovskite films, *ACS Nano* 10 (2016) 9026–9035.

- [250] Z. Wang, T. Yang, Y. Zhang, Q. Ou, H. Lin, Q. Zhang, H. Chen, H.Y. Hoh, B. Jia, Q. Bao, Flat lenses based on 2D perovskite nanosheets, *Adv. Mater.* 32 (2020) 2001388.
- [251] M.A. Stoeckel, M. Gobbi, S. Bonacchi, F. Liscio, L. Ferlauto, E. Orgiu, P. Samori, Ultrathin flexible organic transistors via ambient electrospray deposition, *Adv. Mater.* 29 (2017) 1702469.
- [252] H. Chen, M. Zhang, R. Bo, C. Barugkin, J. Zheng, Q. Ma, S. Huang, A.W.Y. Ho-Baillie, K.R. Catchpole, A. Tricoli, Laser-induced graphene electrodes for high-performance perovskite solar cells, *Small* 14 (2018) 1702571.
- [253] X. Xu, H. Wu, X. He, M.K. Hota, Z. Liu, S. Zhuo, H. Kim, X. Zhang, H.N. Alshareef, Stretchable MXene-polymer composite films for wearable electronics, *ACS Nano* 14 (2020) 9840–9851.
- [254] C.J. Zhang, L. McKeon, M.P. Kremer, S.H. Park, O. Ronan, A. Seral-Ascaso, S. Barwich, C.Ó. Coileáin, N. McEvoy, H.C. Nerl, B. Anasori, J.N. Coleman, Y. Gogotsi, V. Nicolosi, Additive-free MXene inks and direct printing of micro-supercapacitors, *Nat. Commun.* 10 (2019) 1795.
- [255] S. Zheng, H. Wang, P. Das, Y. Zhang, Y. Cao, J. Ma, S.F. Liu, Z.S. Wu, Stretchable MXene composite films with enhanced mechanical and capacitive performance for wearable energy storage, *Adv. Mater.* 33 (2021) e2005449.
- [256] Thibeorchews Prasankumar, Sujin Jose, Pulickel M. Ajayan, Meiyazhagan Ashokkumar, Functional carbons for energy applications, *Mater. Res. Bull.* 142 (2021) 111425, <https://doi.org/10.1016/j.materresbull.2021.111425>.
- [257] Thibeorchews Prasankumar, Kaaviah Manoharan, N.K. Farhana, Shahid Bashir, K. Ramesh, S. Ramesh, Vigna K. Ramachandaramurthy, Advancements and approaches in developing MXene-based hybrid composites for improved supercapacitor electrodes, *Mater. Today Sustain.* 28 (2024) 100963, <https://doi.org/10.1016/j.mtsust.2024.100963>.
- [258] Soumyabrata Roy, Xiang Zhang, Anand B. Puthirath, Ashokkumar Meiyazhagan, Sohini Bhattacharyya, Muhammad M. Rahman, Ganguli Babu, Sandhya Susarla, Sreehari K. Saju, Mai Kim Tran, Lucas M. Sassi, M.A.S.R. Saadi, Jiawei Lai, Onur Sahin, Seyed Mohammad Sajadi, Bhuvaneswari Dharmarajan, Devashish Salpekar, Nithya Chakingal, Abhijit Baburaj, Xinting Shuai, Aparna Adumbukulath, Kristen A. Miller, Jessica M. Gayle, Alec Ajnsztajn, Thibeorchews Prasankumar, Vijay Vedhan Jayanthi Harikrishnan, Ved Ojha, Harikishan Kannan, Ali Zein Khater, Zhenwei Zhu, Sathvik Ajay Iyengar, Pedro Alves da Silva Autreto, Eliezer Fernando Oliveira, Guanhu Gao, A. Glen Birdwell, Mahesh R. Neupane, Tony G. Ivanov, Jaime Taha-Tijerina, Ram Manohar Yadav, Sivaram Arepalli, Robert Vajtai, Pulickel M. Ajayan, Structure, Properties and applications of two-dimensional hexagonal boron nitride, *Adv. Mater.* 33 (2021) 2101589, <https://doi.org/10.1002/adma.202101589>.
- [259] Salamat Ali, Muhammad Sufyan Javed, Khalid Umer, Jiatai Wang, Yujun Fu, Shunlian Kong, Shaikat Khan, Awais Ahmad, Anand Parkash, Munirah D. Albaqami, Annu, Jing Qi, Deyan He, MoS₂@Ti₃C₂T_x Heterostructure: a new negative electrode material for Li-Ion hybrid supercapacitors, *Chem. Eng. J.* 498 (2024) 155330, <https://doi.org/10.1016/j.cej.2024.155330>.
- [260] Salamat Ali, Xiaofeng Zhang, Muhammad Sufyan Javed, Xiaqing Zhang, Guo Liu, Xuegang Wei, Hao Chen, Muhammad Imran, Jiatai Wang, Weihua Han, Jing Qi, 2H-MoS₂ nanosheets-based binder-free electrode material for supercapacitor, *J. Appl. Phys.* 132 (14) (2022) 145001, <https://doi.org/10.1063/5.0100522>.
- [261] Salamat Ali, Xiaqing Zhang, Muhammad Sufyan Javed, Hidayat Ullah Shah, Awais Ahmad, Munirah D. Albaqami, Mohamed Sheikh, Ahmed M. Hassan, Xuegang Wei, Jiatai Wang, Jing Qi, MoS₂/Ti₃CO₂ heterostructure-based ceramics as promising electrode material for high-performance monovalent energy storage devices, *Ceram. Int.* 50 (3) (2024) 4782–4789, <https://doi.org/10.1016/j.ceramint.2023.11.222>. Part A.
- [262] S. Ali, A. Ahmad, I. Hussain, S.S.A. Shah, S. Ali, A. Ali, M. Sufyan Javed, Experimental and theoretical aspects of MXenes-Based energy storage and energy conversion devices, *J. Chem. Environ.* 2 (2) (2023) 54–81, <https://doi.org/10.56946/jce.v2i2.214>.
- [263] Salamat Ali, Syed Shoaib Ahmad Shah, Muhammad Sufyan Javed, Tayyaba Najam, Anand Parkash, Shaikat Khan, Majed A. Bajaber, Sayed M. Eldin, Roaa A. Tayeb, Mohammed M. Rahman, Jing Qi, Recent advances of transition metal dichalcogenides-based materials for energy storage devices, in view of monovalent to divalent ions, *Chem. Rec.* 24 (2024) e202300145, <https://doi.org/10.1002/tcr.202300145>.
- [264] Salamat Ali, Tauqeer Ahmad, Muhammad Yahya Tahir, Muhammad Usman, Muhammad Chhattal, Iftikhar Hussain, Shaikat Khan, Ahmed M. Hassan, Mohammed A. Assiri, P. Rosaiah, Muhammad Sufyan Javed, Bhargav Akkinepally, Jing Qi, The emergence of density functional theory for supercapacitors: recent progress and advances, *J. Energy Storage* 73 (2023) 109100, <https://doi.org/10.1016/j.est.2023.109100>. Part D.
- [265] Salamat Ali, Muhammad Ahsan Farooq Qaisar, Muhammad Sufyan Javed, Khalid Umer, Saleh S. Alarfaji, Muhammad Mateen, Muhammad Chhattal, Shafqat Ali, Anand Parkash, Tensangmu Lama Tamang, Jing Qi, Two-dimensional MXene based innovative electrode materials for supercapacitors: recent advances and prospects, *Fuel* 377 (2024) 132783, <https://doi.org/10.1016/j.fuel.2024.132783>.