



# MULTIDIMENSIONAL NANOMATERIALS FOR SUPERCAPACITORS: NEXT GENERATION ENERGY STORAGE

Editors:  
**Sanjeev Verma**  
**Shivani Verma**  
**Saurabh Kumar**  
**Bhawna Verma**

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# **Multidimensional Nanomaterials for Supercapacitors: Next Generation Energy Storage**

Edited by

**Sanjeev Verma**

*Department of Battery Manufacturing New Energy  
Reliance Industries Limited, Navi Mumbai-400701  
Maharashtra, India*

**Shivani Verma**

*Department of Chemistry, School of Physical Sciences  
Doon University, Dehradun-248012  
Uttarakhand, India*

**Saurabh Kumar**

*Department of Electronics and Communication Engineering  
National Institute of Technology Hamirpur  
Hamirpur-17700, Himachal Pradesh, India*

&

**Bhawna Verma**

*Department of Chemical Engineering and Technology  
Indian Institute of Technology (Banaras Hindu University)  
Varanasi-221005, Uttar Pradesh, India*

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Next Generation Energy Storage**

Editors: Sanjeev Verma, Shivani Verma, Saurabh Kumar and Bhawna Verma

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## PREFACE

Supercapacitors are a new class of superior energy storage devices that provide both high energy and power densities, bridging the gap between batteries and regular capacitors. The two primary charge storage processes of supercapacitors are the redox process and the electrochemical double layer. Considerable interest is being paid to strategies that would combine both mechanisms in a supercapacitor to improve its electrochemical characteristics. The energy storage capacity of supercapacitors can be greatly impacted by the electrode materials utilized to make these devices. For supercapacitors, a variety of materials are being used, including conducting polymers, carbon-based materials, layered structured materials, metal oxides, and sulfides. The energy and power density of supercapacitors might vary depending on the materials' shape and kind. This book discusses developments in next-generation supercapacitor materials such as Mxene, MOFs, Quantum dots, and graphene-based nanostructures. A brief history of nanostructural materials, chemistry and supercapacitors as energy storage devices is also provided. This technical book can be a very helpful reference for scientists, industrial practitioners, graduate and undergraduate students, and other professionals in the scientific and education domains.

This book attempts to present the most recent as well as future forming materials, and ground-breaking developments in nanostructured materials for supercapacitor applications. The numerous intriguing characteristics of nanoscale materials make them perfect for energy storage applications. Additionally, methods are used to improve their morphological, electronic, and electrical characteristics in order to improve their electrochemical performances. Numerous new nanocomposites based on Mxenes, MOFs, Quantum dots, and variants of graphene are discussed. In-depth descriptions of novel methods for synthesizing and customizing their electrochemical characteristics are provided. With thorough characterization, mechanistic techniques, and theoretical analysis, this book compiles information on the production and applications of nanomaterials for supercapacitors. Recent advances in cutting-edge technology, including flexible and wearable supercapacitors made of nanostructured materials, are discussed. The readers of this book are given both basic and specialized techniques for creating nanostructured materials for supercapacitors.

**Sanjeev Verma**

Department of Battery Manufacturing New Energy  
Reliance Industries Limited, Navi Mumbai-400701  
Maharashtra, India

**Shivani Verma**

Department of Chemistry, School of Physical Sciences  
Doon University, Dehradun-248012  
Uttarakhand, India

**Saurabh Kumar**  
Department of Electronics and Communication Engineering  
National Institute of Technology Hamirpur  
Hamirpur-17700, Himachal Pradesh, India

&

**Bhawna Verma**  
Department of Chemical Engineering and Technology  
Indian Institute of Technology (Banaras Hindu University)  
Varanasi-221005, Uttar Pradesh, India

## List of Contributors

<b>Ayesha Zaheer</b>	Department of Physics “Ettore Pancini”, University of Naples Federico II, Piazzale Tecchio, 80, 80125 Naples, Italy
<b>Ali Raza</b>	Department of Physics “Ettore Pancini”, University of Naples Federico II, Piazzale Tecchio, 80, 80125 Naples, Italy
<b>Asif Mahmood</b>	School of Chemical and Biomolecular Engineering, The University of Sydney, Sydney, Australia Center for Clean Energy Technology, School of Mathematical and Physical Sciences, Faculty of Science, University of Technology Sydney, Sydney, Australia
<b>Ahmad Nawaz</b>	Center for Refining & Advanced Chemicals, King Fahd University of Petroleum and Minerals, Dhahran, 31261, Saudi Arabia Department of Chemical Engineering & Technology, Indian Institute of Technology (Banaras Hindu University), Varanasi-221005, India
<b>Abhishek Pathak</b>	Department of Chemistry, Govind Ballabh Pant Institute of Agriculture and Technology, 263145, India
<b>Ankit Tyagi</b>	Department of Chemical Engineering, Indian Institute of Technology Jammu, Jammu, 181221, J & K, India
<b>Akansha Agrwal</b>	Department of Applied Sciences, KIET Group of Institutions, Delhi-NCR, Meerut Road (NH-58), Ghaziabad 201206, India
<b>Bhawna Verma</b>	Department of Chemical Engineering & Technology, Indian Institute of Technology (Banaras Hindu University), Varanasi-221005, Uttar Pradesh, India
<b>Bhuvaneshwari Balasubramaniam</b>	Department of Material Science Programme, Indian Institute of Technology Kanpur, Kanpur 208016, UP, India
<b>Dinesh Bejjanki</b>	Department of Chemical Engineering, National Institute of Technology, Warangal 506004, India
<b>Himadri Tanaya Das</b>	Centre of Excellence for Advance Materials and Applications, Utkal University, Bhubaneswar 751004, Odisha, India
<b>Hafsa Siddiqui</b>	CSIR - Advanced Materials and Processes Research Institute (AMPRI), Bhopal-462026, India
<b>Jahan Zeb Hassan</b>	Department of Physics, Riphah Institute of Computing and Applied Sciences (RICAS), Riphah International University, 14 Ali Road, Lahore, Pakistan
<b>Lakshmikant Atram</b>	CSIR - Advanced Materials and Processes Research Institute (AMPRI), Bhopal-462026, India
<b>Manisha Devi</b>	Department of Chemistry, Gautam College Hamirpur, Himachal Pradesh, India
<b>Manoj Goswami</b>	Academy of Scientific and Innovative Research (AcSIR), Ghaziabad-201002, India CSIR - Advanced Materials and Processes Research Institute (AMPRI), Bhopal-462026, India

<b>Mani Jayakumar</b>	Department of Chemical Engineering, Haramaya Institute of Technology, Haramaya University, Haramaya, Dire Dawa, Ethiopia
<b>Neeraj Kumar</b>	School of Studies in Chemistry, Jiwaji University, Gwalior (M.P), India IPS group of colleges, Shivpuri Link Road, Gwalior (M.P), India
<b>Nigamananda Das</b>	Centre of Excellence for Advance Materials and Applications, Utkal University, Bhubaneswar 751004, Odisha, India Department of Chemical Engineering, National Taiwan University of Science and Technology, Taipei, 10607, Taiwan
<b>Nikhil Kumar</b>	Functional Materials Group, Advanced Materials & Processes (AMP) Division, CSIR-National Metallurgical Laboratory (NML), Burmamines, East Singhbhum, Jamshedpur, Jharkhand-831007, India
<b>Nisha Gupta</b>	Functional Materials Group, Advanced Materials & Processes (AMP) Division, CSIR-National Metallurgical Laboratory (NML), Burmamines, East Singhbhum, Jamshedpur, Jharkhand-831007, India
<b>Netrapal Singh</b>	Academy of Scientific and Innovative Research (AcSIR), Ghaziabad-201002, India CSIR - Advanced Materials and Processes Research Institute (AMPRI), Bhopal-462026, India
<b>N. Sathish</b>	Academy of Scientific and Innovative Research (AcSIR), Ghaziabad-201002, India CSIR - Advanced Materials and Processes Research Institute (AMPRI), Bhopal-462026, India
<b>Om Prakash</b>	Regional Ayurveda Research Institute, Ministry of Ayush, Gwalior, 474009, India
<b>Payaswini Das</b>	CSIR-Institute of Minerals and Mining Technology, Bhubaneswar, Odisha, India
<b>Pradeep Kumar</b>	Department of Chemical Engineering & Technology, Indian Institute of Technology (Banaras Hindu University), Varanasi-221005, India
<b>Pallab Bhattacharya</b>	Functional Materials Group, Advanced Materials & Processes (AMP) Division, CSIR-National Metallurgical Laboratory (NML), Burmamines, East Singhbhum, Jamshedpur, Jharkhand-831007, India
<b>Priyanka A. Jha</b>	Department of Physics, Indian Institute of Technology (Banaras Hindu University), Varanasi-221005, India
<b>Pardeep K. Jha</b>	Department of Physics, Indian Institute of Technology (Banaras Hindu University), Varanasi-221005, India
<b>Prabhakar Singh</b>	Department of Physics, Indian Institute of Technology (Banaras Hindu University), Varanasi-221005, India
<b>Raju Kumar Gupta</b>	Department of Chemical Engineering, Indian Institute of Technology Kanpur, Kanpur-208016, UP, India Center for Environmental Science and Engineering, Indian Institute of Technology Kanpur, Kanpur-208016, UP, India Department of Sustainable Energy Engineering, Indian Institute of Technology Kanpur, Kanpur- 208016, UP, India



<b>Shailendra Kumar Dwivedi</b>	School of Studies in Physics, Jiwaji University, Gwalior (M.P), India Madhav Institute of Technology & Science, Gola ka Mandir, Gwalior-474005, India
<b>Shivani Verma</b>	Department of Chemistry, School of Physical Sciences, Doon University, Dehradun-248012, Uttarakhand, India
<b>Sampath Kumar Puttapati</b>	Department of Chemical Engineering, National Institute of Technology, Warangal 506004, India
<b>Swapnamoy Dutta</b>	University of Tennessee, Bredeesen Center for Interdisciplinary Research and Graduate Education, Knoxville, TN, 37996, USA
<b>Shipra Jaswal</b>	Department of Chemistry, Gautam College Hamirpur, Himachal Pradesh, India
<b>Swadesh Kumar</b>	Department of Chemistry, Gautam College Hamirpur, Himachal Pradesh, India
<b>Satendra Kumar</b>	Academy of Scientific and Innovative Research (AcSIR), Ghaziabad-201002, India CSIR - Advanced Materials and Processes Research Institute (AMPRI), Bhopal-462026, India
<b>S. Rajveer</b>	Metallurgical and Materials Engineering, National Institute of Technology, Jamshedpur-831014, India
<b>T. Elango Balaji</b>	Department of Chemical Engineering, National Taiwan University of Science and Technology, Taipei, 10607, Taiwan
<b>Venkatesa Prabhu S.</b>	Center of Excellence for Bioprocess and Biotechnology, Department of Chemical Engineering, College of Biological and Chemical Engineering, Addis Ababa Science and Technology University, Addis Ababa, Ethiopia
<b>Vikas Kumar Pandey</b>	Department of Chemical Engineering & Technology, Indian Institute of Technology (Banaras Hindu University), Varanasi-221005, India
<b>Vijay Kumar Juyal</b>	Department of Chemistry, Govind Ballabh Pant Institute of Agriculture and Technology, Uttarkhand 263145, India
<b>Vivek Kumar</b>	Regional Ayurveda Research Institute, Ministry of Ayush, Gwalior, 474009, India
<b>Viveka Nand</b>	Department of Chemistry, Govind Ballabh Pant Institute of Agriculture and Technology, Uttarkhand 263145, India
<b>Zaheer Ud Din Babar</b>	Scuola Superiore Meridionale (SSM), University of Naples Federico II, Largo S. Marcellino, 10, 80138, Italy Department of Physics "Ettore Pancini", University of Naples Federico II, Piazzale Tecchio, 80, 80125 Naples, Italy

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**CHAPTER 1**

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**Introduction of Next-Generation Materials**

**Neeraj Kumar<sup>1,3</sup>, Shailendra Kumar Dwivedi<sup>2,4,\*</sup>, Om Prakash<sup>5</sup> and Shivani Verma<sup>6</sup>**

<sup>1</sup> School of Studies in Chemistry, Jiwaji University, Gwalior (M.P), India

<sup>2</sup> School of Studies in Physics, Jiwaji University, Gwalior (M.P), India

<sup>3</sup> IPS group of colleges, Shivpuri Link Road, Gwalior (M.P) India

<sup>4</sup> Madhav Institute of Technology & Science, Gola ka Mandir, Gwalior-474005, India

<sup>5</sup> Regional Ayurveda Research Institute, Ministry of Ayush, Gwalior, 474009, India

<sup>6</sup> Department of Chemistry, School of Physical Sciences, Doon University, Dehradun-248012, Uttarakhand, India

**Abstract:** The “next-generation materials” are those materials that have high efficiency, high-performance structural stability, easy manufacturability, and multifunctional capabilities. These new materials can be classified based on dimension, shape, composition, and nanostructure like 0D, 1D, 2D, and 3D. These materials have unique enhanced properties *viz.* electronic, optical, mechanical, magnetic, optoelectronics, vitrification, thermal properties, *etc.* Due to these outstanding features, these smart materials could be a game changer for prospects. Tuning the properties of such advanced materials provides a wide variety of fascinating opportunities. This chapter aims to provide a comprehensive overview of materials used to fabricate supercapacitor point of view and several other latest applications. The nanomaterials, discussed in this chapter along with their properties are Graphene, nanotubes, nanocomposites, microwave-absorbing materials, nanoparticles, biomaterials, and self-healing polymers. It also discusses future directions for the development of advanced materials that perform well to anticipate future trends and highlight their relevance in real-world contexts. This chapter could become the torchbearer for new researchers working in the field of multifunctional advanced materials.

**Keywords:** Functional & smart materials, Flexible electronics, Multifunctional, Nanomaterials & nanofluids, Optoelectronics.

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\* **Corresponding author Shailendra Kumar Dwivedi:** School of Studies in Physics, Jiwaji University, Gwalior (M.P), India and Madhav Institute of Technology & Science, Gola ka Mandir, Gwalior-474005, India; E-mail: shail.dew04@gmail.com

Sanjeev Verma, Shivani Verma, Saurabh Kumar & Bhawna Verma (Eds.)  
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## INTRODUCTION

The “Next-generation materials” or advanced materials are those materials that have the properties of high efficiency, high-performance structural stability, easy manufacturability, and multifunctional capabilities. The basic characteristics of these materials include being very light, intelligent, more durable, and active materials that can adjust appropriately to their surroundings. In recent years, the development of new materials and technologies has been associated with innovation, creativity, originality, and forward thinking. A self-assembly process is specifically designed to produce advanced materials comprising nanoscale structures [1, 2]. These materials are of great interest in scientific research efforts and industrial development because of their innovative potential applications in various fields. Advanced materials are future materials with improved properties that are consciously designed for superior performance. The major scientific contributions of the 21st century, and a new understanding of atomic and subatomic levels, laid the foundation for the creation of advanced materials. The development of such advanced future materials can even lead to the design of completely advanced products, such as portable supercomputers, mini electronic gazettes, flexible electronics and optoelectronic devices, automatic lightweight weapons, fire registrant materials, medical implantable devices, gas sensors, lightweight industrial equipment, intelligent robotics, *etc.*

Nowadays, the materials such as graphene, carbon nanotube, men, nanofluids, quantum dots, nanoparticles, metal-organic frameworks (MOFs), aerogel, nanocomposites, microwave absorbing materials, self-healing polymers, artificial spider silk, metal foam, synthetic fuel and lubricants, shrilk and many more have emerged as advanced materials for human beings. These materials have the potential to sort out human futuristic problems and are useful for the better advancement of human civilization. Advanced nanomaterials are very desirable in these domains because of their controllable production and beautiful design. Due to its vast applicability in a range of sectors, such as energy storage, electronics, optics, optoelectronics catalytic, absorption and separation, biomedical, luminescence, sensing, and environment, nanotechnology has gained a lot of attention in recent decades. The key features of advanced nanomaterials are their active surfaces, dimensions, and reaction conditions [3, 4].

The physical and chemical properties of advanced nanomaterials are greatly influenced by their dimensions and reaction conditions [3]. Thus, it is the right time to think about not only synthesizing materials but also tuning their physicochemical properties (Fig. 1a) to develop next-generation materials. The beauty of advanced nanomaterials is their tunable properties; therefore, by changing the shape, size, and reaction conditions of the nanomaterials, one can

change their functionality accordingly. So, to utilize these nanomaterials for the development of a new world, we need to develop advanced synthetic techniques so that more features of those materials can be explored in various fields for human beings.

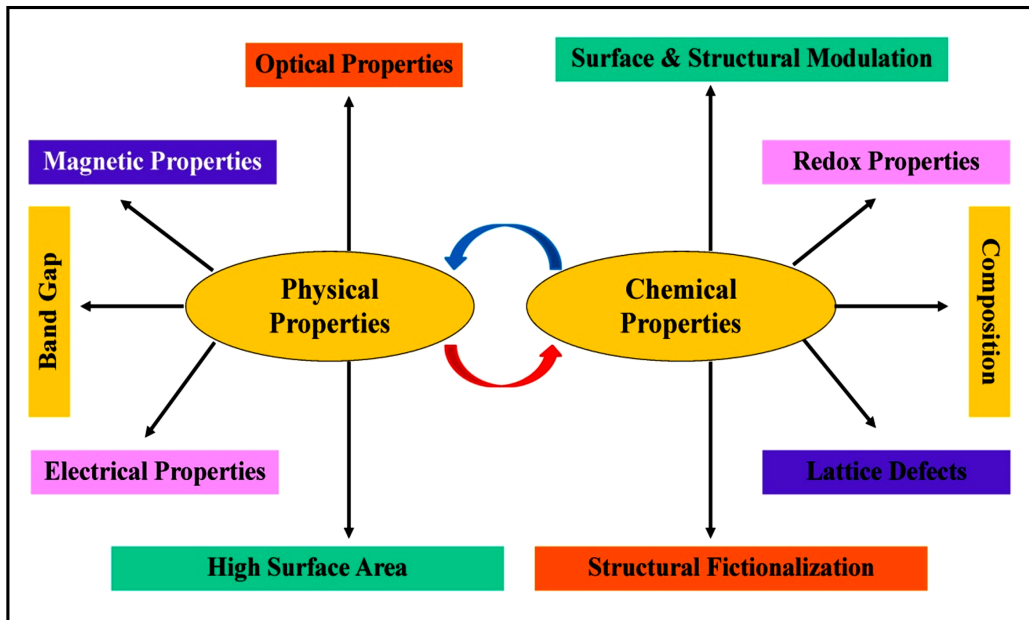


Fig. (1a). Physicochemical properties of nanomaterials.

Scientific legend; Andre Geim and Konstantin Novoselov in 2004 at Manchester University discovered a wonder material called “Graphene” by playing with a lump of graphite and Scotch tape. At that time, both did not know how to deal with and what to do. But nowadays, Graphene has become one of the extraordinary materials for the future world because of its properties like immensely strong, flexible, transparent, and conductivity. Shrilk could be another wonder material for the future world [4]. Shrilk is a biodegradable solution to plastic and is mainly made up of silk proteins and chitin developed by Javier Fernandez and Donald Ingber at the Wyss Institute of Biologically Inspired Engineering, Harvard University [5]. A material with huge absorption capability of electromagnetic radiations (microwave) was discovered named metamaterials. It is an advanced or a new class of materials that can have electromagnetic features including the negative value of permittivity, permeability, and refractive index that do not occur naturally. In 2011, another wonder material called Mxenes was discovered by two research groups led by Y.Gogotsi and M.Barsoum at Drexel University. Generally, Mxene is a 2D transition metal-based compound of

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**CHAPTER 2**

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**Supercapacitor Basics (EDLCs, Pseudo, and Hybrid)****Dinesh Bejjanki<sup>1</sup> and Sampath Kumar Puttapati<sup>1,\*</sup>**<sup>1</sup> *Department of Chemical Engineering, National Institute of Technology, Warangal 506004, India*

**Abstract:** Over the past few years, supercapacitors have been spotlighted because of the challenges faced by other energy storage systems. The supercapacitor possesses excellent power density and long-term durability with an eco-friendly nature. Due to their wide range of advantages, supercapacitors are applicable especially in electric vehicles, heavy-duty vehicles, telecommunication, electric aircraft, and consumer electronic products. As per the charge storage mechanism, supercapacitors are divided into three categories based on their charge-storing method: electric double-layer capacitors (EDLCs), pseudocapacitors, and hybrid capacitors. The electrode materials such as graphene, activated carbon, metal oxides, conducting polymers, *etc.*, were widely applied, for better performance. The electrolyte is a crucial component in the mechanism of the supercapacitor to run the system at a higher voltage and thus there are various electrolytes such as solid, inorganic, and organic based on the application of the materials, and the electrolytes are chosen. However, the supercapacitors suffer from low energy density. Currently, research is more focused on advanced materials and various synthesis methods to overcome the drawbacks. This chapter provides a detailed understanding of supercapacitors with redox and non-redox reactions -the broad classification of the supercapacitor -their charge storage mechanism -various electrode materials -electrolytes (aqueous, non-aqueous, and solid) and current collectors, *etc.* Finally, the parameters that help in estimating the performance of supercapacitors are (specific capacitance, energy density, and power density) included.

**Keywords:** Classification of supercapacitor, EDLC, Hybrid supercapacitor, Pseudo capacitors.

**INTRODUCTION**

Energy consumption has risen drastically over the past decades as well as the diminution of non-renewable fuel sources, leading to work on new approaches to solve the issue [1, 2]. In reality, the rise in energy consumption is brought about by the increasing use of electronic gadgets in society [3]. There are limited energy

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\* **Corresponding author Sampath Kumar Puttapati:** Department of Chemical Engineering, National Institute of Technology, Warangal 506004, India; E-mail: pskr@nitw.ac.in

resources; it is also essential to produce clean, sustainable, and renewable energy sources [4]. Renewable sources with eco-friendly nature are warmly welcomed for the production of power. Environmental-friendly renewable technologies include wind, solar, and tidal energy [5]. At the same time, burning fossil fuels is hugely hazardous because of the release of CO<sub>2</sub> and additional pollutants into the environment [6]. Renewable energy technologies, like solar or wind, only produce energy when the sun is shining or the wind is blowing. Thus energy-efficient gadgets are facing issues. We cannot effectively utilize renewable energy until appropriate energy storage technology is developed. Innovative electrical drive energy storage systems must be coupled with renewable energy storage conversion technology [7, 8]. Batteries and supercapacitors are two potential electrical energy storage solutions that can meet the fundamental needs of storing energy from renewable energy technologies [9]. The term “supercapacitor” also refers to electrochemical capacitors. Consequently, because they can store energy for a long time, electrical energy will be available all the time. Due to their ability to provide continuous power, batteries, and supercapacitors are in high demand in the current scenario [10, 11]. Supercapacitors are energetic devices because of their high-power densities, whereas batteries are powerful because of their high energy densities [12]. Combining renewable energy technologies with supercapacitors or batteries may create a unique hybrid system that can save energy and produce simultaneously. Such a hybrid energy system holds great promise for powering a future electronic device [13]. Heavy-duty vehicles need to have both high-power density and energy density simultaneously. Therefore, hybrid devices must emerge in combination with supercapacitors and batteries. The new module supercapacitor showed excellent performance and they are proper candidates for hybrid energy systems. In the year 1990, the United States Department of Energy actively promoted financing for research on supercapacitors and batteries. It increased global awareness of the supercapacitors’ potential [14]. Ever since, tremendous work has been devoted to supercapacitor research and advancement in hybrid electrode materials, nanocomposites, and appropriate electrolytes to enhance performance at the minimum cost. Simultaneously, a fundamental understanding of the supercapacitor’s design, operation, performance, and component optimization resulted in improved supercapacitor performance and a significant increase in energy density [15, 16]. To enhance energy density, advanced hybrid supercapacitors were created, in which the electrodes are composited with carbon materials and metal oxides. In the hybrid supercapacitor, the electrochemical process of the electroactive substance occurs at the interface of electrode and electrolyte either by oxidation-reduction or adsorption, and intercalation, mechanism. In this manner with the fundamentals of supercapacitors and electrode materials, the capacitance and the energy density of the electrode materials can be boosted [17].

This chapter will give a comprehensive understanding of supercapacitors, their broad classification, and their working mechanism. It also includes the main components of a supercapacitor such as electrode materials, electrolytes, and the current collector. Lastly, the parameters such as specific capacitance, energy density, and power density are being discussed.

## FARADAIC AND NON-FARADAIC

In electrochemical measurement, faradaic and non-faradaic reactions are the main types of electrode characteristics (Fig. 1). Charge transfer occurs at the electrode during the redox reaction in the faradaic process. However, the occurrence of electrochemical reactions at the electrode surface does not entail the presence of a faradaic reaction [18]. An electrical charge is introduced on both electrodes while an electrochemical reaction occurs and the ions are transported away from it because charge should not be retained on the electrode. The faradaic process is commonly found in fuel cells and lead-acid batteries. There is no charge transfer in the non-faradaic process. Similarly, in the adsorption and desorption method, the electric charge and ions stay at or on electrodes.

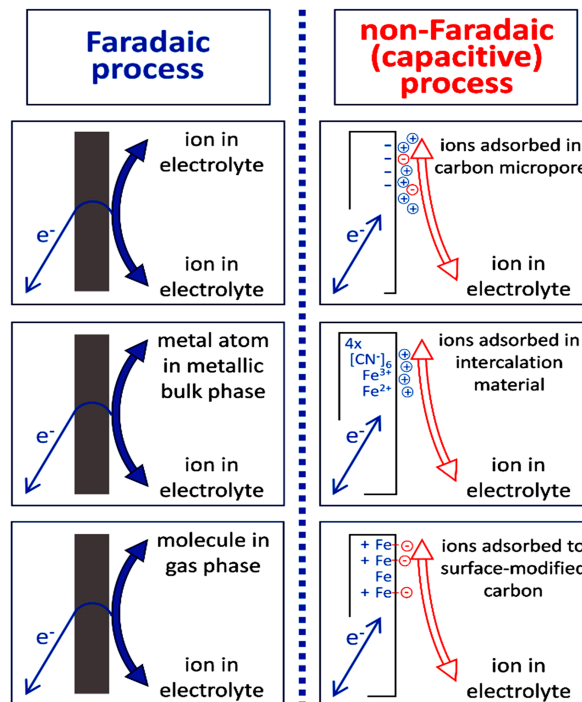


Fig. (1). Difference in charge storage vs charge transfer in faradaic and non-faradaic reactions. Adapted with permission from Reference [18], Copyright (2018), Chemical Physics.

## CHAPTER 3

# Graphene and its Derivatives: Chemistry, Properties, and Energy Storage Application

Om Prakash<sup>1</sup>, Vijay Kumar Juyal<sup>2</sup>, Abhishek Pathak<sup>2</sup>, Neeraj Kumar<sup>3,6</sup>, Vivek Kumar<sup>1</sup>, Shivani Verma<sup>5</sup>, Akansha Agrwal<sup>4</sup> and Viveka Nand<sup>2,\*</sup>

<sup>1</sup> Regional Ayurveda Research Institute, Ministry of Ayush, Gwalior, 474009, India

<sup>2</sup> Department of Chemistry, Govind Ballabh Pant Institute of Agriculture and Technology, Uttarakhand 263145, India

<sup>3</sup> School of Studies in Chemistry, Jiwaji University, Gwalior (M.P), India

<sup>4</sup> Department of Applied Sciences, KIET Group of Institutions, Delhi-NCR, Meerut Road (NH-58), Ghaziabad 201206, India

<sup>5</sup> Department of Chemistry, School of Physical Sciences, Doon University, Dehradun-248012, Uttarakhand, India

<sup>6</sup> IPS group of colleges, Shivpuri Link Road, Gwalior (M.P), India

**Abstract:** Graphene has attracted a lot of attention in recent years since its discovery because of its unique structural, mechanical, optical, electric, and thermal properties, making it a viable candidate for a wide range of applications. Graphene, a 2-dimensional network of carbon atoms with high conductivity and surface area is a potential material for high-performance applications. For conceivably ground-breaking uses in lithium-ion batteries, solar cells, sensing, and photocatalytic applications, graphene is being used as a filler or composite material with polymers, metals, and metal oxides. Graphene's primary derivatives are graphene oxide (GO) and reduced-graphene oxide (rGO). Graphite can be oxidised to produce GO, and it can be reduced to produce rGO. There is a lot of interest in the application of energy storage in different industries because of the fascinating features of graphene and its derivatives. In the last decade, there has been a lot of interest in the energy storage applications of nanomaterials based on graphene, and numerous groups have started working in this area all over the world. Graphene is perfect for the manufacture of energy storage devices due to its exceptional compatibility, solubility, and selectivity. It is possible to do this, especially if they have been exposed to metal oxide, which causes only minor sheet restacking. The high conductivity of the interconnected networks of graphene is another factor influencing it as a material for energy storage applications.

**Keywords:** Graphene, Graphene derivatives, Graphene oxide, Reduced Graphene oxide.

\* Corresponding author Viveka Nand: Department of Chemistry, Govind Ballabh Pant Institute of Agriculture and Technology, Uttarakhand 263145, India; E-mail: imvivekanand@gmail.com



## INTRODUCTION

The graphene material and its derivatives are considered frontier materials for future technology [1]. Graphene was discovered in 2004 by Andre Geim and Konstantin Novoselov by the process of scotch tape peeling [2]. It is a hexagonal or honeycomb-like 2D sheet with  $sp^2$  hybridized carbon atoms having a thickness in the order of atom diameter [3]. With its extended honeycomb network, it can be wrapped to form 0D fullerenes, rolled to form 1D nanotubes, and stacked to form 3D graphite. These display extraordinary electrical, mechanical, and thermal properties because of the long-range  $\pi$ -conjugate system which attracted theoretical studies and has become more exciting for experimentalists in recent years [4]. Strong chemical durability, highly ordered structure, high thermal conductivity, abundant surface areas, high Young's modulus, and high electron mobility are characteristics of the ideal graphene [5]. Its aromatic rings, reactive functional groups, and free electrons make graphene materials and their derivatives a fascinating field of study across multiple disciplines (Fig. 1) [6].

Graphene can be synthesized in two main ways: top-down and bottom-up methods. In top-down graphene production, precursors such as graphite are structurally broken down, then the interlayers are separated and deposited on graphene sheets. Among these methods are oxidation-reduction of GO, arc discharge, liquid phase exfoliation, and mechanical exfoliation. Whereas, carbon source gases are used to synthesize graphene on a substrate in bottom-up methods like epitaxial growth, chemical vapour deposition, and total organic synthesis [1]. The graphene-based materials chemistry, properties, and energy storage application are discussed here. Due to its remarkable optical, electrical, thermal, and mechanical properties, there has been an increase in interest in using them in various biomedical applications during the past several years, including drug delivery systems, biosensors, and imaging systems. Because of this, research using nanomaterials from the graphene family has produced positive outcomes in a number of scientific fields. It is crucial to conduct more research in order to fully understand how these materials interact with biological systems [4]. This chapter has demonstrated the chemistry of graphene and its derivatives with its application in energy storage and other allied fields.

## CHEMICAL EXFOLIATION

When compared to other popular methods like epitaxial growth, micromechanical cleavage, and Hummer's methods, chemical exfoliation is thought to be a very efficient and economical top-down synthesis process. Exfoliation of bulk graphite is used in the chemical exfoliation procedure to create graphene. The number of

layers and lateral dimension of the resulting graphene may be greatly influenced by choosing an appropriate starting material of graphite. Using reducing solvents or oxidation, graphite layers are separated during chemical exfoliation. By widening the interlayer space between graphite flakes, oxidation or reduction solutions primarily aim to decrease the van der Waals force. As reducing agents, solvents like methanesulfonic acid and hydrazine hydrate N-methyl-2-pyrrolidone have been utilized often.

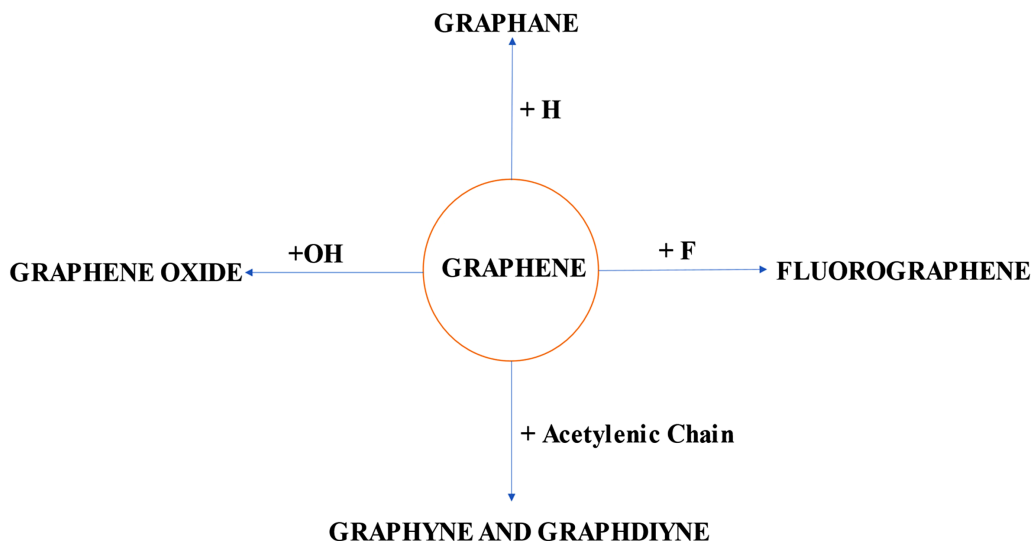


Fig. (1). Schematic diagram of various derivatives of Graphene.

## CHEMISTRY OF GRAPHENE

The chemistry of graphene is the cause of all of its incredible properties. The transparency of graphene is a significant feature. Only 2% of the light shining on it is absorbed by it. This is due to the fact that those atoms are spaced apart because of their thinness. Its strength, which is 200 times stronger than steel, is another significant attribute that may be described using chemistry [7]. Each carbon forms a covalent link with three additional carbon atoms, and these bonds are exceedingly strong. Since it is also incredibly flexible, this is unexpected. One of the best electrical conductors is graphene. This is so that electrons, the building blocks of electricity, can pass through the tightly bound carbon atoms. Because of covalent bonding, and the hexagonal form electrons can flow more quickly. The best heat conductor is known as graphene which is shown in Fig. (2).

**CHAPTER 4****Quantum Dots: Chemistry, Properties, and Energy Storage Applications****Himadri Tanaya Das<sup>1,\*</sup>, T. Elango Balaji<sup>2</sup>, Swapnamoy Dutta<sup>3</sup>, Payaswini Das<sup>4</sup> and Nigamananda Das<sup>1,2</sup>**<sup>1</sup> *Centre of Excellence for Advance Materials and Applications, Utkal University, Bhubaneswar 751004, Odisha, India*<sup>2</sup> *Department of Chemical Engineering, National Taiwan University of Science and Technology, Taipei, 10607, Taiwan*<sup>3</sup> *University of Tennessee, Bredesen Center for Interdisciplinary Research and Graduate Education, Knoxville, TN, 37996, USA*<sup>4</sup> *CSIR-Institute of Minerals and Mining Technology, Bhubaneswar, Odisha, India*

**Abstract:** Currently, Quantum dot nanomaterials have received a lot of attention due to their intriguing features. Most intriguing is how they can be used as electrodes to create safe chemical-free supercapacitor parts and produce clean energy. Due to their high charge storage capacity and stability, quantum dot electrodes are increasingly in demand for high-tech hybrid supercapacitors. This chapter covers the electrochemical performance, physiochemical characteristics, and synthesis of numerous quantum dots. They are also provided with information about the electrochemical characteristics of various supercapacitors. To show readers the potential of this field of study, the best operational factors are highlighted.

**Keywords:** Quantum dots, Nanostructured, Electrochemical, Energy storage devices, Batteries, Supercapacitors.

**INTRODUCTION**

Exploration of materials of different dimensions and their versatile applications have opened up several routes of advancement. In today's research, nanostructured materials have been widely used. Their dimensions as well as morphologies have exhibited a large scope that can be employed in upcoming technologies and quantum dots (QDs) are one of them. In general, QDs can be defined as nanocrystal-semiconducting materials having the smallest diameters

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\* **Corresponding author Himadri Tanaya Das:** Centre of Excellence for Advance Materials and Applications, Utkal University, Bhubaneswar 751004, Odisha, India; E-mail: himadridas@utkaluniversity.ac.in

which vary in between the 2–10 nm nanoscale range. Owing to the smaller particle size, tunable characteristics, and high quantum yield-like significant features, QDs became an attractive choice of material to study in diverse fields of science and engineering such as energy storage (battery, SCs, *etc.*), medicine, and catalysis, solar cells, and sensing. Their adjustable surface area (SA), electro-optical characteristics, and unique quantum confinement effects have made them more impactful. Reduced dimensions of such materials have impacted electronic and optical properties in a great way which further affected applicative purposes. Excitons are restricted to a significantly smaller volume of semiconductor materials in QDs, which is by their exciton Bohr radius. Less energy band splitting occurs as a result, creating an area of quantum confinement. The electronic energy bands are connected to such a region of discrete and quantized electron-hole pairs in various dimensions within a material. To precisely tune the energy bandgap, the size and composition of the QDs can be modified which favors the applications. Specifically, by varying the diameter of QDs typically between 2 to 10 nm, the bandgap of QDs can be adjusted which ultimately results in the improvement of their electrical and optical characteristics. Similar to an atom, the energy levels in QDs are quantized, but compared to the atoms, the distinct factor is that in a QD, the energy level in between separation is lesser; besides, this separation varies based on the size of the QDs having a larger energy level in between separation. The interval between a QD's energy levels must not exceed  $kT$  (*the quantity of heat required to enhance the thermodynamic entropy of a system*)-otherwise, electrons could be excited to higher energy levels by thermal energy [1].

In recent research, QD sizes are precisely controlled and modified which has led to several visions of investigating the physical and chemical properties for different applications. Based on the core type, shape, structure, size like characteristics, QDs are typically sorted. In general, QD cores are prepared with different elements such as cadmium, indium, and carbon encapsulated by chalcogenides [2]. Nevertheless, recent studies have attempted to develop shell structures to enhance the structural stability as well as applicative perspective [3]. Formation of core–shell structure has also been proved useful in lessening the toxicity owing to the existence of an exterior shell layer surrounding the core QDs. One of the recent studies explained that *via* combining two nanocrystal semiconductors having separate bandgap energies, alloyed QDs are developed which have displayed distinguishable characteristics that are different from their parent semiconductor properties [4]. Gradient and homogeneous internal structures are observed in alloyed-QDs, where it was minutely noticed that without tweaking the size of the nanocrystals, their internal structural arrangements and composition vary and so, due to the approach of tuning the optical and electrical characteristics, the result from alloyed QDs of  $CdS_xSe_{1-x}$

x/ZnS formed with different composition emits a wide range of light wavelengths [5]. Several reports have also focused on their shapes such as cylindrical QDs, spherical QDs, tetrahedral QDs, conical QDs, pyramidal QDs, *etc.*, and their resultant experimental impact on different kinds of applications [3d, 6].

### **Synthesis and properties of QDs**

The selection of the synthesis method is useful as it has a profound impact on the size, morphology as well as on the characteristics of the prepared product. QDs are found to be prepared in different ways in reports. Top-down synthesis is a technique where QDs are produced by thinning down bulk semiconductors. Laser ablation, chemical and electrochemical oxidation, ultrasonic, arc discharge, *etc.* are the commonly utilized top-down techniques for fabricating QDs. Bottom-up approaches are also popular techniques and are considered very impactful in the fabrication of QDs. These techniques are mainly categorized into two important segments which are vapor-phase and wet-chemical techniques; and are subdivided into different techniques such as wet chemical techniques like sol-gel, hot-solution decomposition, microemulsion, competitive reaction chemistry, and electrochemical method whereas molecular beam epitaxy, ion sputtering, *etc.* techniques are considered as vapor-phase techniques [7]. In addition, techniques like microwave synthesis, hydrothermal, and thermal decomposition techniques can also be used in the bottom-up approach.

#### ***Laser ablation***

Owing to easy morphology control and better efficiency, laser ablation has been utilized in several studies to form QDs [8]. Importantly, the laser ablation of solids in liquid state is as simple as well as a versatile route to develop nanostructures. Sun *et al.* used a laser ablation technique to synthesize CQDs using graphite and water vapor under inert atmosphere conditions [9]. From several reports, it was confirmed that *via* adjusting laser pulse width, the QD size can be controlled as well and the adjustment affects QD's nucleation and growth [10]. Owing to the interaction between the graphite flakes and the laser beams, an immediate rise in pressure vapor/plasma plume and temperature was noticed at the interface of the liquid medium and the graphite flake. Afterward, the liquid confinement leads to the formation of a bubble at the laser focus which then swiftly expands and spreads out to the highest radius. Subsequently, the completion of a laser pulse width results in the start of the shrinking of the bubble along with the pressure of the surrounding liquid, which leads to the cooling of the inner region and forms a cluster of nuclei. The bubbles containing various cluster densities can be obtained by tuning the width of the laser pulse, thereby obtaining the CQDs with tunable sizes. In another study, Lihe Yan and the group

## Metal-Organic Frameworks (MOFs): Chemistry, Properties, and Energy Storage Applications

Nikhil Kumar<sup>1</sup>, Nisha Gupta<sup>1</sup> and Pallab Bhattacharya<sup>1\*</sup>

<sup>1</sup> Functional Materials Group, Advanced Materials & Processes (AMP) Division, CSIR-National Metallurgical Laboratory (NML), Burmamines, East Singhbhum, Jamshedpur, Jharkhand-831007, India

**Abstract:** The scarcity of natural stocks of fossil fuels and the rising pollutant ions evolved from the burning of carbon-containing fuels, has triggered the necessity for clean, renewable, and sustainable energies to be generated and its subsequent storage in portable form to meet the on-demand consumption. However, the performances of storage materials are still limited for extensive real-world applications due to their sluggish ion diffusion kinetics, lack of efficiency in extreme weather conditions, poor chemical stability, and many more. Therefore, it is highly requisite to discuss the development and assess the performances of new advanced energy storage materials. In this chapter, we are specifically keen to discuss the design, synthesis, chemistry, and properties of various MOFs based electrode materials for energy storage devices like batteries and supercapacitors, which can necessarily store electrical energies by implementing the use of suitable electrode and electrolyte materials through an upright fabrication technique. Generally, MOFs contain both inorganic metal ions and organic ligands/linkers which enable great control over their structural and compositional modifications to optimize the properties like porosity, stability, surface area, redox activity, and electrical conductivity and show great promise to generate high energy storage performances, in the recent past. However, despite the current success, MOFs based electrode materials have faced a lot of challenges in terms of the choice of suitable metals and organic ligand moieties, rich host-guest interactions, preparation of composites with desired morphology and properties, control over composite composition, scalability of the process and many more which needs to be addressed for its full-proof use in the real-world application as energy storage materials and thusly, this chapter is important to discuss.

**Keywords:** Active Sites, Batteries, Compositional flexibility, Composites, Conductivity, Electrochemical performances, Efficiency, Durability, Energy Storage, Functionality, Hydrothermal, MOFs, Metal nodes, Organic ligands,

\* Corresponding author Pallab Bhattacharya: Functional Materials Group, Advanced Materials & Processes (AMP) Division, CSIR-National Metallurgical Laboratory (NML), Burmamines, East Singhbhum, Jamshedpur, Jharkhand-831007, India; E-mail: pallab.b@nmlindia.org

Sanjeev Verma, Shivani Verma, Saurabh Kumar & Bhawna Verma (Eds.)  
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Porosity, Physicochemical properties, Power density, Secondary building units, Surface area, Supercapacitors.

## INTRODUCTION

Given the widespread global demand for clean energy consumption in today's modern world, the development of such clean electrochemical energy storage devices remains a challenge. To address the need for upcoming energy storage devices, MOFs are gaining the attention of researchers due to their porous crystalline nature. Using inorganic metal ions or clusters and multidentate organic ligands, a promising class of porous solid-state materials has been developed over the last two decades with innovative chemistry. MOFs also known as metal-organic materials (MOMs), porous coordination network materials (PCN), porous coordination polymers (PCPs), and metal-organic coordination network (MOCN) constructed with inorganic metal ions or clusters and multidentate organic ligands, have emerged as the most promising class of solid-state porous materials over the last two decades with innovative chemistry. The term "MOF" came into existence after the pioneering publications of mainly three scientists Omar M. Yaghi [1], G. Ferey [2], and S. Kitagawa [3]. These multifunctional materials have contributed extensively to the interests and benefits the society by diminishing health, energy, and environmental issues. MOFs are composed of two different kinds of components: metal inorganic clusters and organic linkers, which act as "joints" and "struts", respectively [4]. The possible connectivity of organic ligands with different metal nodes, secondary building units (SBUs), or supra-molecular building blocks (SBBs) leads to the creation of predictable MOFs with versatile architectures by reticular chemistry. SBUs and the topology of the target framework are the only criteria to synthesize application-specific framework materials with desired characteristics. The excellent features of MOFs such as ultrahigh porosity (till approx. 90% free volume), huge internal surface area (beyond almost  $10000 \text{ m}^2\text{g}^{-1}$ ), and an infinite number of possibilities with a degree of variability for inorganic and organic components, make MOFs suitable candidates for numerous potential applications in adsorption, catalysis, clean energy, gas storage and gas/vapor separation, thin-film and membrane devices, drug delivery and biomedical imaging, luminescence, sensors, conductors, pharmaceuticals, spintronic, energy generation, energy conversion and energy storage devices [4, 5].

Importantly, the flexibility and geometry of organic building blocks play a key role in the structural engineering and functionality of targeted framework materials with desired topologies. These kinds of frameworks exhibit high mechanical and thermal strength and can sustain the same porosity even after the removal of guest solvent molecules. In this rationale, severe benzene di-, tri-, tetra-

and hexa-carboxylates, azolates, and their related derivatives are frequently utilized as organic building units and resulted in a series of advantageous MOFs with enviable features [6]. The versatile coordination modes and adaptable conformations of organic ligands with multi-donor sites have been widely used in developing polynuclear MOFs. In addition to this, several chiral organic molecules namely peptides, amino acids, and their related derivatives can construct chiral centers based on MOFs [7]. Nevertheless, MOFs demonstrate a few weak points like poor chemical stability which impede their applicability with full potential. Hence, it is highly desirable to further improve activities, enhance properties, and incorporate new functionalities. In this context, a variety of active functional materials like graphene, carbon black, and carbon nanotubes have been projected to overcome the shortcomings of pristine MOFs. This new approach opens a lot of opportunities for chemists and researchers to do innovations with creative expression and amazing multidimensional properties. The multifunctional character of novel MOF composites will surely stimulate the appearance of inventive, ground-breaking, diversified technologically important industrial applications. These composites are directly used as precursors for inorganic solids or pioneer advanced materials with potential practical applications in sensing, catalysis, protective coatings, separation, bio-medical as well as energy generation, conversion, and storage [8 - 11]. This chapter focused on the recent noteworthy progress in the advancement of MOFs or MOFs-based composites, their classification, synthesis, and properties along with their performance in energy storage applications. The selectivity of ligand and metal ions leads to the formation of flexible MOFs with dynamic features towards different kinds of electrochemical measurements (Fig. 1).

The responsible key factors, synergistic effects of functional metal nodes, and active ligand sites for energy storage applications have also been discussed in detail. The authors believed that the present chapter would inspire and motivate the readers to adopt a sustainable way of making industrially active MOF composites employed for decorating new energy storage devices for future generations.

## **FUNDAMENTALS OF MOFS: CLASSIFICATION, SYNTHESIS AND PROPERTIES**

MOFs are a unique class of highly porous crystalline polymeric materials with metal ions or clusters linked mutually by bridging organic ligands. Usually, the ligands are an organic molecule that possesses functional groups (amine, pyridine, carboxylic acid, and others) in their structure where a lone pair of electrons can be easily donated to the next metal, normally termed Lewis Base [12]. The porous crystalline MOF framework depends on the dimensionality and length of the



# MXene: Chemistry, Properties, and Energy Storage Applications

Manisha Devi<sup>1\*</sup>, Shipra Jaswal<sup>1</sup> and Swadesh Kumar<sup>1</sup>

<sup>1</sup> Department of Chemistry, Gautam College Hamirpur, Himachal Pradesh, India

**Abstract:** The growing interest and demand for energy storage applications have significantly encouraged the development of a broad range of functional 2D materials. Owing to the extraordinary properties including 2D lamellar structure, larger interlayer space, mechanical strength, high thermal and electrical conductivity, and negative zeta-potential, carbides/nitrides/carbonitrides of transition metal, usually known as MXenes have received the interest of researchers in the development of environmentally friendly materials for storage and conversion of energy. In this chapter, we focused on the MXene, their methods of preparation, the progress of development of various MXenes, and their modification for the storage of energy. Here, we have discussed the various storage devices for energy including batteries and superconductors. This chapter offers scientific inspiration and literature for the rational design and synthesis of high-capacity MXenes and their composites that can fulfill the increased demand for next-generation energy storage devices.

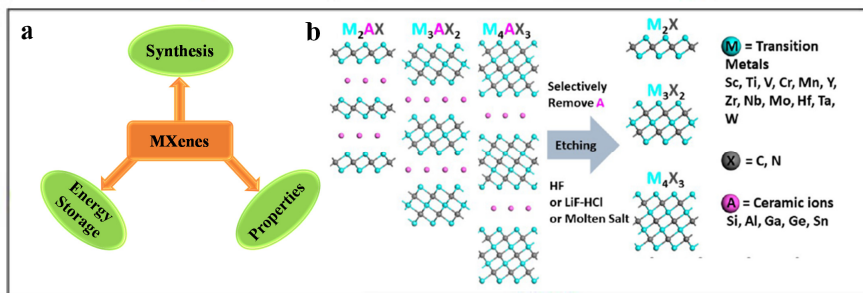
**Keywords:** Batteries, Energy storage, MXene, MXene-based nanocomposites, Supercapacitors.

## INTRODUCTION

With the increased world population and industries, energy-related issues are of major concern for today's generation [1]. In this regard, the development of high-performance energy storage systems is essential to integrate various renewable energy resources into high-power energy devices. Up to now, various electrical energy storage devices (ESDs) and technologies have been developed for example batteries, capacitors, and supercapacitors [2, 3]. ESDs are very important in developing smart electronics and wearable textiles. Metal ions-based rechargeable batteries and supercapacitors are being considered as potential ESDs.

\* Corresponding author Manisha Devi: Department of Chemistry, Gautam College Hamirpur, Himachal Pradesh, India; E-mail: manishathakur04@gmail.com

In previous years, 2D materials have emerged as attractive materials for several applications depending upon their physio-chemical, optical, and electrical properties [4, 5]. Therefore, 2D materials have been widely investigated and received the Nobel Prize for the groundbreaking discovery of the first 2D material graphene [6, 7]. Due to single/few atomic layers, 2D materials exhibit properties such as nanosheet-like structures, large specific surface areas, very high aspect ratios, short ion diffusion path, and low energy barrier for electron transportation. These properties make them a potential material for the development of electrodes for electrochemical storage of energy to deal with current energy issues. 2D materials including graphene, transition metal oxides, transition metal dichalcogenides, hexagonal boron nitride, hydroxides, organics, MXenes, and black phosphorous have been extensively explored in the field of energy storage [8 - 11]. Among various 2D materials, a family of 2D materials comprising transition metal nitrides, carbides, and carbonitrides commonly recognized as MXenes, have attracted huge consideration because of their extraordinary properties including easily modulating surface chemistry, rich chemical composition possibilities, unusual combination of metallic hydrophilicity and conductivity, good thermal stability, tunable bandgap, charge carrier mobility, excellent electrical conductivity ( $2 \times 10^5 \text{ Sm}^{-1}$ ), fast diffusion and easy dispersion in various solvents including water, large spacing between layers and surface area [12]. In 2011, the first MXene,  $\text{Ti}_3\text{C}_2\text{T}_x$ , was discovered by Yury Gogotsi *et al.* at Drexel University, USA [13]. General chemical representation  $\text{M}_{n+1}\text{X}_n\text{T}_x$  corresponds to MXenes, where M = early transition metal (Sc, Ti, V, Cr, Mo, Nb, Hf, Ta, Zr, *etc.*), X = carbon and/or nitrogen atom,  $\text{T}_x$  = surface termination groups (F, O or OH) and  $n = 1-4$ . Versatile properties render MXenes as a potential candidate for energy storage in the form of batteries and supercapacitors. In this chapter, we focused on the MXene, different methods of preparation of MXenes, different properties, and utilization of MXenes and their composites in energy storage applications. Fig. (1a) demonstrates an overview of the full chapter.



**Fig. (1).** (a) Overview of the chapter. (b) Schematic representation of the preparation of MXene (bottom) from MAX (top). Adapted with permission from Reference [11], Copyright (2019), American Chemical Society.

### Structure of MXene and MAX Phase

MXene are collectively referred to as 2D layered transition metal nitrides, carbides, or carbonitrides [10]. MXenes ( $M_nX_n$ ) were prepared by the removal of the A layer from their precursor MAX phase. The term “MAX” corresponds to the chemical composition  $M_nAX_n$ , where M corresponds to an early transition metal, A corresponds to an element from group 13 or 14 (Al, Si, Ga, *etc.*), X corresponds to carbon or nitrogen, and  $n = 1-4$  (Figs. **1b** & **2a**) [10]. Etching is an essential step for the synthesis of MXenes. Here, the M-X bond is ionic, covalent, and metallic while the M-A bond is metallic; this leads to a higher binding energy of M-X than M-A. This suggests that the M-X bond is highly stable in comparison to the M-A. The metallic nature of both bonds inhibits mechanical exfoliation. The chemical stability of M-X bonds over M-A bonds and the difference in the binding energies of the bonds facilitate the selective etching of the A layer to prepare MXene. On removing the A layer, the corresponding MXene retains the hexagonal lattice of parent MAX. Fig. (**1b**) demonstrates the preparation of MXene ( $M_2X$ ,  $M_3X_2$ , and  $M_4X_3$ ) by etching of their respective MAX phases. Thin films of MXene are generally organized horizontally, which were inherited from their parent MAX phase. Multilayer MXene (m-MXene) is further converted into few-layer MXene nanosheets by delamination using intercalating agents (DMSO and LiF) that lead to increased surface area for providing redox active sites which allow the 2D MXene to be a part of energy storage applications. First MXene  $Ti_3C_2$  was prepared by dissolving the parent MAX phase ( $Ti_3AlC_2$ ) in a hydrofluoric acid (HF) solution [13]. After this, different MXenes have been prepared and explored for various applications [14, 15].

### DIFFERENT APPROACHES FOR THE SYNTHESIS OF MXENES

In the synthesis of MXene etching plays a crucial role. As discussed in the previous section, due to the metallic character of M–A bonds of MAX precursor, isolation of  $M_nX_n$  layers by mechanical shearing is difficult. However, treating more chemically active M–A bonds of MAX with etching agents prompts the removal of A layers selectively. Usually, hydrofluoric acid (HF) is used to extract the “A” layer from MAX [16]. Van der Waals force of attractions between layers causes the aggregation of 2D nanosheets that restrict the high-quality 2D MXenes synthesis. Normally, two synthetic strategies, bottom-up and top-down synthesis have been employed for the preparation of 2D MXenes. Top-down synthesis is mainly the exfoliation of bulk crystalline material into single or few-layer sheets whereas bottom-up is based on the preparation of material from atoms or molecules. In general, MXene synthesis involves 3 steps; i) preparation of MAX precursor, ii) etching out the A layer, iii) intercalation and exfoliation (Fig. **2b**).

## Different Supercapacitors' Characterizations

Satendra Kumar<sup>1,2</sup>, Hafsa Siddiqui<sup>2</sup>, Netrapal Singh<sup>1,2</sup>, Manoj Goswami<sup>1,2</sup>, Lakshmikant Atram<sup>2</sup>, S. Rajveer<sup>3</sup>, N. Sathish<sup>1,2</sup> and Surender Kumar<sup>1,2,\*</sup>

<sup>1</sup> Academy of Scientific and Innovative Research (AcSIR), Ghaziabad-201002, India

<sup>2</sup> CSIR - Advanced Materials and Processes Research Institute (AMPRI), Bhopal-462026, India

<sup>3</sup> Metallurgical and Materials Engineering, National Institute of Technology, Jamshedpur-831014, India

**Abstract:** The development of new materials and technologies that can efficiently store energy while delivering power quickly has been the subject of numerous investigations. In an electrochemical supercapacitor (E-SC), the electric charge is stored in a double-layer formed at the electrode/electrolyte interface (EEI), which is based on the surface area as well as pore size availability. The high surface area provided by the micropores (pore diameter: 2 nm) is essential for charging the E-SCs and calculating the capacitance values. Mesopores (2 nm < pore diameter < 50 nm) allow good electrolyte penetration and offer a high-power density (2 nm pore diameter 50 nm). However, because a lot of non-carbonaceous materials are used to make E-SC electrodes, more *in-situ* analytical characterisation tools along with electrochemical techniques are needed. It is crucial to have at least a brief understanding of the electrochemical processes occurring at the EEI of E-SC electrodes (or devices). Variations in electrochemical, morphological and surface, and crystallographic properties will be used to categorise the data gathered by the state-of-the-art characterisation techniques. This chapter also provides a resource for researchers by outlining the methods to learn more about E-SCs and opportunities to achieve additional functionalities beyond those related to energy storage.

**Keywords:** Analytical tools, Electrochemical tools, Electrode/electrolyte interface, Supercapacitor, Pseudocapacitor.

### INTRODUCTION OF SUPERCAPACITORS

Research into E-SCs has been a popular topic in the academic community for the better part of two decades due to their exceptional cycle stability and high power density [1]. E-SCs are intended to serve as a secondary energy storage option alongside batteries in a variety of contexts, including portable gadgets, electric

\* Corresponding author Surender Kumar: Academy of Scientific and Innovative Research (AcSIR), Ghaziabad - 201002, India; CSIR - Advanced Materials and Processes Research Institute (AMPRI), Bhopal - 462026, India; E-mail: surenderjanagal@gmail.com

vehicles, backup generators, and so on [2]. There have been numerous attempts to assemble high-performance E-SCs using a wide range of materials and methods (surface area/pore control, doping, heterostructure, engineering of crystal structure, and current collector modification) [3]. E-SCs have achieved enhanced levels of capacitance, rate performance, energy density, and cycling stability. Electrode materials [EMs] undergo complex structural/physical/chemical fluctuations during the charge-discharge process. Modifications to lattice parameters, ion adsorption/desorption, chemical bonds, dimensional/mass change, and other changes are all part of the electrochemical process [4].

Device performance can be estimated by using any or combination of the electrochemical techniques: cyclic voltammetry (CV), time constant ( $\tau$ ), galvanostatic charge-discharge (GCD), leakage current ( $I_{\text{leak}}$ ), and electrochemical impedance spectroscopy (EIS) [5, 6]. This time constant defines the shortest time period over which the cell will accept a significant perturbation. It can be obtained through equation  $\tau=1/2\pi fC_{dl}$  at half of the charge-transfer resistance in the high frequency region of EIS profile. Beyond device testing, morphology and surface analysis of EMs can be analysed by scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), transmission electron microscopy (TEM), Auger electron spectroscopy (AES) and so on [7]. X-ray diffraction (XRD), electrochemical quartz crystal microbalance (EQCM), nuclear magnetic resonance (NMR), scanning electrochemical microscopy (SECM), Raman spectroscopy (RS), Fourier transform infrared (FT-IR) spectroscopy, *etc.* are some of the *in-situ* characterisation methods that have been used to investigate E-SCs phase and dynamics [8, 9]. Diverse characterization methods (Fig. 1) yield different data, but they all work together to shed light on the underlying mechanisms of EEI of E-SCs. While E-SCs are primarily used to store energy, it is important to note that new features can be created into E-SCs by modifying their configuration or integrating them. Mechanically deformable, flexible, self-healing, colour-tunable, self-charging E-SC, *etc.*, have all been tried [10]. However, an understanding of the electrochemical phenomenon behind the various EMs is still lacking at the EEI level. Exploring new functions for E-SCs can expand their potential application domains while learning more about E-SCs can improve their understanding of their core operations at EEI level [11]. This chapter will review the advanced characterisation approaches used in E-SC investigations and provide instances of E-SCs that serve many purposes.

### **Double-layer Formation and Faradaic Process**

As per working principles, E-SCs can be electric double-layer capacitors (EDLCs), pseudocapacitors (PCs), and hybrid capacitors (HCs) [12]. Classifications of E-SCs are shown schematically in Fig. (2). EDLCs are capable of storing

energy through the physical absorption of ions upon that electrode, while PCs use redox processes. The charged layer comprises an innermost tight layer (also called the Stern layer) and an exterior diffusion layer. The stern layer has an innermost Helmholtz plane (IHP) with solvent and adsorbed electrolyte ions and another outer Helmholtz plane (OHP) with solvated electrolyte ions [13]. On thermal disturbance, non-typically adsorbed ions may diffuse from OHP to bulk solution. Equation (1) estimates the capacitance of EDLCs:

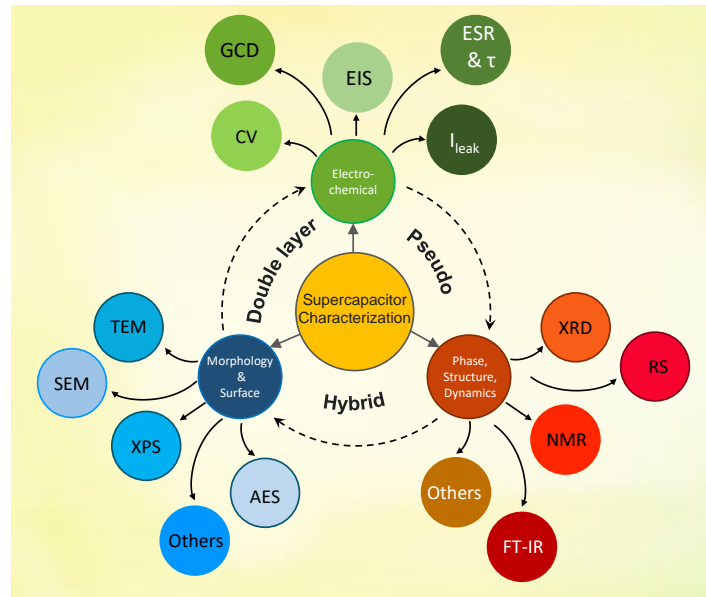


Fig. (1). A schematic representation of different characterization tools for E-SCs.

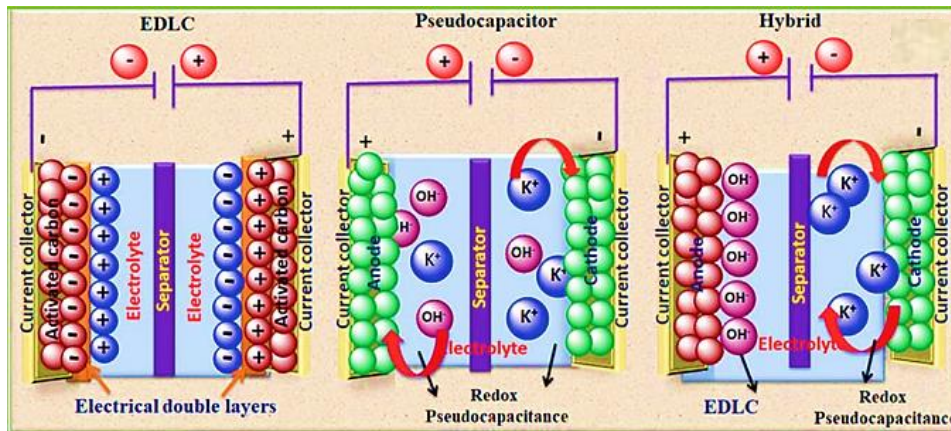


Fig. (2). Classification and charge storage mechanisms of E-SCs: EDLC, PC, and HC. Adapted with permission from reference [14], Copyright (2022), The Royal Society of Chemistry.

## CHAPTER 8

# Electrolytes for Electrochemical Energy Storage Supercapacitors

Priyanka A. Jha<sup>1,\*</sup>, Pardeep K. Jha<sup>1</sup> and Prabhakar Singh<sup>1</sup>

<sup>1</sup> Department of Physics, Indian Institute of Technology (Banaras Hindu University), Varanasi-221005, India

**Abstract:** In this chapter, the types of electrolytes and the alteration in capacitance with pore size, their power density, and energy density along with the interaction of electrolytes with current collectors are discussed. The electrolytes' electrochemical stability broadly estimates the working cell voltage provided that the electrodes are stable under operating cell voltage. The electrolytes are divided into various categories such as liquid electrolyte, solid-state, and redox-active electrolyte. The liquid electrolytes are further categorized into aqueous and non-aqueous electrolytes. The critical performance parameters such as stability, lifetime, operating temperature, operating voltage, *etc.* are believed to be affected by electrolytes. Moreover, the electrolytes are believed to interact with the current collectors, additives, binders, separators, and electrode material to affect the practical performance of supercapacitors. However, the capacitance of the electrolyte depends upon the ion size and the matching between the electrode pore size and electrolyte ion size. The power density and energy density depend upon the potential window, ionic conductivity, and electrochemical stability along with concentration, respectively. Further, the ion-electrode interaction is supposed to affect the cycle life and power density as well. The thermal stability of electrolytes depends upon their boiling points, freezing points, and salt solubility and the equivalent series resistance depends upon ion conductivity, mobility, and viscosity.

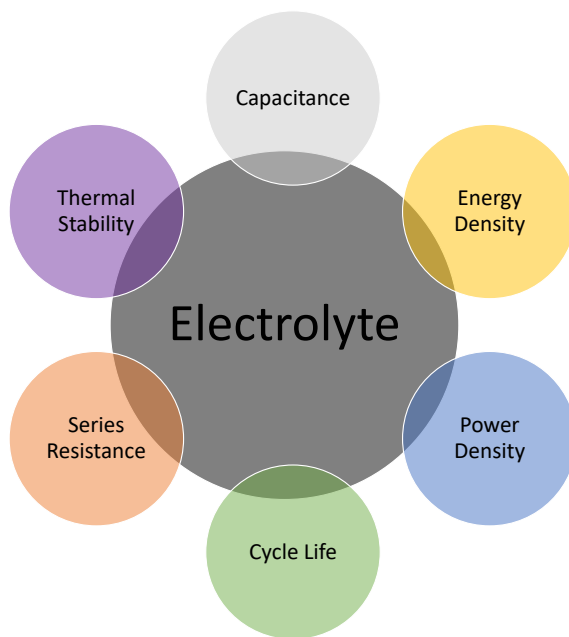
**Keywords:** Electrolytes, Energy storage, Electrochemical supercapacitors.

## INTRODUCTION

An electrolyte is an essential component (located in the center) of the electrochemical supercapacitors. It comprises solute solvated in solvent or solvent-free salt-like ionic liquids [1]. It is important because it provides (a) ionic conductivity allowing charge compensation; (b) a charge storage process *via* reversible redox process and (c) formation of double layer (electrical) in the

\* Corresponding author Priyanka A. Jha: Department of Physics, Indian Institute of Technology (Banaras Hindu University), Varanasi-221005, India; E-mail: priyankajha.dce@gmail.com

double layer capacitors in pseudo capacitors. The electrolytes affect the power density, equivalent series resistance, lifetime, cycling stability, operational temperature range, and self-discharge rate of electrochemical supercapacitors [2 - 4]. Further, the performance of electrochemical supercapacitors depends upon the interaction of electrolytes with the other components of supercapacitors like the electrodes, current collectors, separators, binders, and additives. The factors affecting the energy density of the supercapacitors are cell voltage and capacitance as shown in Fig. (1). The capacitance in turn depends upon electrolyte ion size and the combination of electrode pore size and electrolyte ion size [5]. Moreover, the electrolyte and current collector interaction reduces the energy density of electrochemical supercapacitors by decreasing the operative cell voltage. Other than these, energy density is observed to depend upon the concentration of electrolytes and the electrochemical stability. It is observed that a higher concentration of the electrolyte reduces ion mobility due to less hydration (Fig. 1) [6].



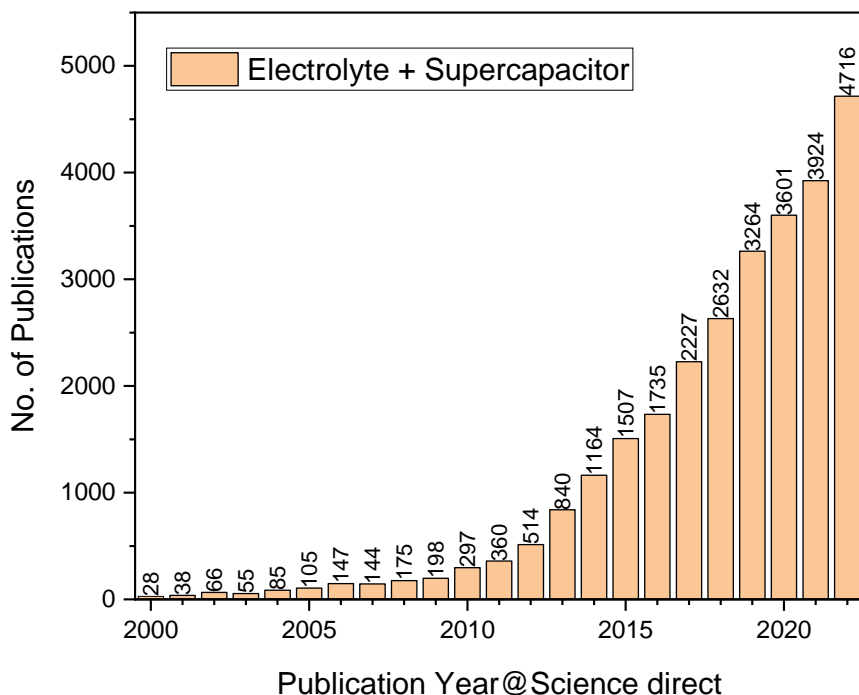
**Fig. (1).** The factors affecting the electrolyte and performance of electrochemical supercapacitors.

Furthermore, the number of publications based on electrolyte materials has largely increased over the last decade as shown in Fig. (2).

Of the well-known electrolytes, most of the supercapacitors employ organic electrolytes with acetonitrile solvents having a cell voltage of 2.5-2.8V [7, 8].



While, the electrochemical supercapacitors with graphitic carbon have shown a potential of 3.3- 3.8 V. Further, a safe, low-cost, and green electrolyte *i.e.* aqueous electrolyte possesses a ceiling voltage of 1.23V. The operational voltage depends upon the electrochemical stability of electrolytes and affects the power and energy density. Further, the ion type and the size of the electrolyte; solvent and ion concentration; interactions between solvent and ion, and the electrode-electrolyte interfacial interactions are believed to affect the pseudo capacitance and double layer capacitance. The ionic conductivity of the electrolyte affects the equivalent series resistance. The features required for a good electrolyte are (i) wide electrochemical stable window; (ii) ionic conductivity should be high; (iii) wide operating temperature range; (iv) less volatile and viscous; (v) environment-friendly; (vi) availability with high purity and (vii) less cost [9].



**Fig. (2).** Number of publications in Science Direct in the last twenty years with the keyword “electrolyte + supercapacitor”.

Fig. (3) shows the classification of electrolytes into various categories and subcategories. Based on the literature survey, the electrolytes are majorly categorized into three groups solid, liquid, and redox active electrolytes (Fig. 3). Liquid electrolytes are comprised of water-in-salt, aqueous, and no-aqueous electrolytes. Quasi-solid state/solid-state comprises inorganic, dry, and gel-based polymer electrolytes. Further, Redox-active electrolytes are classified into gel

## Graphene-Based Fiber Shape Supercapacitors for Flexible Energy Storage Applications

Ankit Tyagi<sup>1,\*</sup>, Bhuvaneshwari Balasubramaniam<sup>2</sup> and Raju Kumar Gupta<sup>3,4,5</sup>

<sup>1</sup> Department of Chemical Engineering, Indian Institute of Technology Jammu, Jammu, 181221, J & K, India

<sup>2</sup> Department of Material Science Programme, Indian Institute of Technology Kanpur, Kanpur 208016, UP, India

<sup>3</sup> Department of Chemical Engineering, Indian Institute of Technology Kanpur, Kanpur-208016, UP, India

<sup>4</sup> Center for Environmental Science and Engineering, Indian Institute of Technology Kanpur, Kanpur-208016, UP, India

<sup>5</sup> Department of Sustainable Energy Engineering, Indian Institute of Technology Kanpur, Kanpur-208016, UP, India

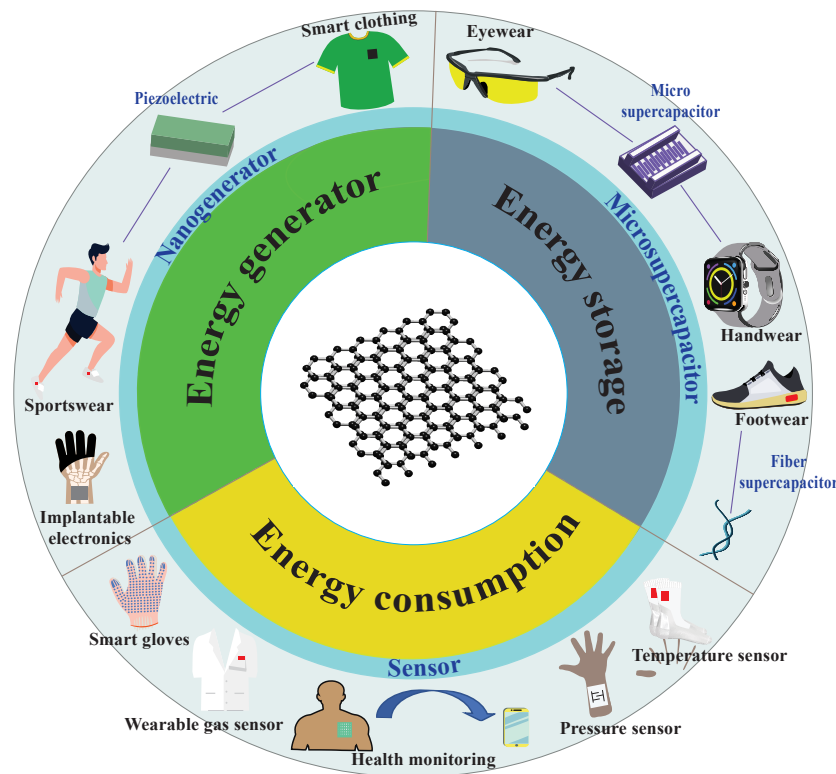
**Abstract:** Energy storage devices are essential because of ever-worsening fossil fuel depletion, increasing energy demand, and increasing environmental pollution. Maxwell Technologies, NessCap, Ashai Glass, and Panasonic commercialize carbon-based conventional supercapacitor devices. Carbon materials like graphene, carbon nanotubes, and activated carbon, are considered favourable materials for bendable and wearable electronic devices. The graphene, because of its high conductivity (thermal  $\sim 5 \times 10^3 \text{ W m}^{-1} \text{ K}^{-1}$ , electrical  $\sim 10^2$  to  $10^8 \text{ S m}^{-1}$ ), extraordinary surface area (theoretically  $\sim 2630 \text{ m}^2 \text{ g}^{-1}$ ), outstanding electrochemical performance (100 to 200 F  $\text{g}^{-1}$ ), less weight compared to transition metal oxides (because of less molecular weight of the carbon), outperform every other carbon material [1, 2]. The fiber-shaped supercapacitors are considered a potential future candidate for electrochemical energy storage systems and have gained considerable attention from the energy storage research community. This chapter discusses the importance of fiber-shaped supercapacitors, their evolution, various forms of their device structures, and electrolytes used for fiber-shaped supercapacitors. Further, the wet-spinning technique for synthesizing graphene fibers and their composites with pseudo-capacitive materials are also discussed.

**Keywords:** Graphene, Supercapacitor, Carbon-based material, Energy storage, Flexible devices.

\* Corresponding author Ankit Tyagi: Department of Chemical Engineering, Indian Institute of Technology Jammu, Jammu, 181221, J & K, India; E-mail: ankit.tyagi@iitjammu.ac.in

## INTRODUCTION

Continuous technological improvements pave the way for developing flexible, wearable, and lightweight electronic devices that can be rolled, folded, bent, and stretched [3]. Flexible electronic textiles and fiber-shaped storage devices have been developed for smart cloth, electronic paper, environmental monitoring devices, implantable medical devices, entertainment, health monitoring sensors, and fashion technologies (Fig. 1) [4]. The rapid development of wearable displays, memory devices, sensors, and transistors on different textiles has recently gained enormous attention [5]. The latest global market survey report projected that the market for wearable electronic devices will grow at a rate of ~15% annually from 2022 to 2030, which was evaluated at USD 52.14 billion in 2021. After COVID-19, the demand for health monitoring sensors built into wristwear, eyewear, and headwear will be expected to increase [6]. The main challenge is developing a large energy density light weight energy storage system with large areal capacitance, without sacrificing wearable properties.



**Fig. (1).** Graphene-based electronics devices for future applications.

The traditional supercapacitors (SCs) contain two active material-coated metal current collectors, with a separator, and an electrolyte (Fig. 2a). The conventional SCs based on liquid electrolytes are rigid, too heavy, and have large-size device structures to ensure safety, making them unsuitable for portable and flexible electronics [7, 8]. Alternative to conventional SCs, thin film Li-ion batteries, flexible micro-supercapacitors, and micro batteries with various shapes and sizes have been developed extensively to power flexible electronic devices, which are two-dimensional (2D) planar structures [9]. Micro batteries significantly improve charge storage performance because of their smaller size in the micrometer range *via* reducing transportation length and enhancing active material surface area exposed to the electrolyte solution [10]. However, micro-supercapacitors (MSCs) are considered the utmost favorable micro-energy storing unit for a range of miniaturized and portable devices because of their extraordinary power density, outstanding cycling (greater than 100,000 cycles), fast charging-discharging (within seconds or several minutes), flexibility, ease of fabrication, and environment-friendly nature compared to micro batteries [11, 12].

Although in-plane interdigital MSCs are flexible and portable, there is an extensive need to further improve these MSCs for wearable and stretchable electronic devices [3]. When in-plane interdigital MSCs are woven into textiles or cloth, they require more space than 1D fiber shape SCs, limiting their potential in wearable electronics. Further, in-plane interdigital MSCs devices can also restrict the natural airflow or convection necessary for a person's health [13].

To address these issues, the focus of scientists has considerably shifted from developing 2D planar structures to fabricating 1D fiber shape devices having lightweight, increased flexibility, and foldability [14, 15]. 1D fiber shape SC devices are better for flexible, stretchable, and wearable electrical devices than traditional 2D planar devices because of their shape diversity, outstanding foldability, twisting-ability, lightweight, and ease of weaving into textiles to develop smart fabrics for wearable electronics [14]. They have omnidirectional flexibility, making them suitable for portable and flexible electronics [16].

Hydrothermal techniques, laser-reduction techniques, and wet spinning techniques are used to synthesize graphene fibers. However, the wet-spinning method outclassed other synthesis techniques due to its considerably lower cost, lightweight and mechanically robust fibers, control over properties of fibers (electrical and mechanical), and the capability of large industrial production [17].

This chapter highlights the topics related to synthesizing graphene oxide (GO) fibers following wet-spinning techniques and the reduction and modification of GO fibers to reduced GO (rGO) fibers for developing rGO-based fiber shape SCs.

## Quantum Dots-Based Nanostructures for Supercapacitors

Himadri Tanaya Das<sup>1,\*</sup>, Swapnamoy Dutta<sup>2</sup>, T. Elango Balaji<sup>3</sup> and Nigamananda Das<sup>1,3</sup>

<sup>1</sup> Centre of Excellence for Advance Materials and Applications, Utkal University, Bhubaneswar 751004, Odisha, India

<sup>2</sup> University of Tennessee, Bredesen Center for Interdisciplinary Research and Graduate Education, Knoxville, TN, 37996, USA

<sup>3</sup> Department of Chemical Engineering, National Taiwan University of Science and Technology, Taipei, 10607, Taiwan

**Abstract:** Recently, Quantum dot nanomaterials have been explored to a great extent for their exciting properties. Their application as electrodes to produce clean energy and hazardous chemical-free components of supercapacitors is the most interesting. The quantum dot electrodes are found to offer high charge storage capacity as well as stability that ultimately boosts their demand in advanced hybrid supercapacitors. This chapter discusses the synthesis, physiochemical features, properties, and electrochemical performance of various quantum dots. Additionally, insights into their electrochemical properties in different supercapacitors are illustrated. The best operational parameters are highlighted to provide readers with the future scope of this research area.

**Keywords:** Electrochemical, Energy storage devices, Nanostructured, Supercapacitors, Quantum dots.

### INTRODUCTION

In the present and future global progress, the development of efficient and sustainable energy sources is and will be playing a great role. Both the environment and economy are presently getting impacted due to excessive dependency on fossil fuels, and this problem needs to be deciphered as soon as possible to ensure stable situations. High-performance and eco-friendly renewable energy sources and storage devices have rapidly developed which gave signs of

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\* Corresponding author Himadri Tanaya Das: Centre of Excellence for Advance Materials and Applications, Utkal University, Bhubaneswar 751004, Odisha, India, E-mail: himadridas@utkaluniversity.ac.in

hope to remove the dependency on conventional fuels. Electrochemical energy is a vital segment that plays a revolutionary role in the advancement of energy scenarios. Most advanced and efficient energy devices like supercapacitors (SCs), batteries, and fuel cells are the major fractions that are constructed based on the concepts of electrochemical energy storage and conversion. Owing to excellent characteristics and benefits such as high specific capacitance ( $C_s$ ), durability, safety, rapid charge-discharge, very negligible maintenance cost, almost no memory effect, and the bridge between power-energy difference which prevails in the capacitor and fuel cells/batteries, *etc.*, SCs have grabbed the attention of several researchers [1 - 4]. Structures of SCs can be altered depending on their target usage; for instance, compact, lightweight, and flexible structures can be used for devices like smartphones, tablets, laptops, smartwatches, and digital cameras; on the other hand, they can also be used in the form of heavier structures to use as the power supplier for the applications which require immediate release of energy for the shorter duration like in solar arrays, wind turbines, automotive industries, *etc.* However, the major issue with supercapacitors is their lesser energy density (ED). Since the ED of capacitors is strongly correlated with  $C_s$  and voltage, if both increase then ED will also increase

SCs consist of two electrodes which are separated by a separator to prevent the connection between the electrodes and also provides good ionic transportation in the electrolyte. SCs accumulate charge in the electrode-electrolyte interface, typically the charge storage process highly relies on the structure and characteristics of the electrode material. For instance, a non-faradaic double-layer charge storage process was proposed for the carbon-based electrodes, while a faradaic rapid, reversible redox reaction mechanism was proposed for metal oxide-based electrodes. So, modifying electrode materials and electrolytes can lead to achieving better outcomes like fast charging, high power capabilities, high  $C_s$ , a proper life cycle with high-capacity retention, *etc.* Electrode materials became a largely concentrated topic in SCs which are broadly investigated either by chemical composition alteration or *via* changing their physical factors like surface area, pore size, *etc.* Due to adjustable electronic and optical properties, high cycling, and excellent biocompatibility, QDs are considered one of the prominent electrode materials. For example, carbon quantum dots (CQDs) are quasi-spherical particles that have shown good conductivity and efficient supercapacitor performances. Similarly, they exhibit strong quantum confinement and great capacitance outcome. For both graphene quantum dots (GQDs) and CQDs, performance can be elevated by using various surface functional groups [5]. Apart from these 0D, 1D, and 2D, QDs like g- $C_3N_4$ , h-BN,  $MoS_2$ , silicene, antimonide, TMDCs, MXenes-QDs, *etc.* have also shown great results [6 - 8].

### Synthesis of Quantum Dots for Electrodes in Supercapacitors

H. Devendrappa and the group prepared QDs composited with a conjugated polymer by employing a combination of hydrothermal synthesis and in situ chemical polymerization techniques. Firstly, a hydrothermal approach was utilized to prepare QDs where distilled water was used to dissolve 12 wt% of glucose precursor, heated at 180 °C for a period of 3 h with the pressure maintained at 110 psi inside an autoclave. Finally, they were centrifuged to derive QDs. Afterward, the chemical-oxidative polymerization technique was implemented to prepare polypyrrole, FeCl<sub>3</sub>, and HCl. QDs were mixed, stirred and heated, and cooled by placing them in an ice bath. PPY–QDs composite was obtained after the oxidizing agent FeCl<sub>3</sub> was poured and stirred, followed by polymerization by keeping the mixture at rest for 24 h [7, 9]. In another work, Peihui Luo and the group utilized hydrothermal treatment of QDs and graphene oxide to incorporate QDs into a 3D graphene (3-DG) framework to form QDs/3DG hydrogels [10]. Firstly, 3-DG hydrogels were prepared by using aqueous GO as a precursor under hydrothermal conditions of 180 °C for 4 h. Afterwards different concentrations of QDs (prepared by oxidizing carbon fibers) were added to the same GO solution and autoclaved at 180 °C for 4 h to get the QDs/3DG hydrogels which were used as a supercapacitor electrode. In the study, several composites were developed by compositing QDs/GO with various weight ratios such as 10%, 20%, 40%, and 80%. Hydrothermal synthesis was also used by M. Ashourdan *et al.*, to synthesize CuMnO<sub>2</sub>/graphene QD composite [11]. The author used citric acid, and sodium hydroxide to synthesize QDs, and Cu(NO<sub>3</sub>)<sub>2</sub>. Mn(NO<sub>3</sub>)<sub>2</sub> precursors were used to prepare CuMnO<sub>2</sub>. Afterward, both the QDs and CuMnO<sub>2</sub> were mixed and allowed for a hydrothermal reaction for 24 h at 80 °C. Finally, the product was purified using water and ethanol and then dried in an oven at 55 °C for 8 h to obtain the composite. Not only the QDs, nickel-cobalt oxide (NiCo<sub>2</sub>O<sub>4</sub>) QDs were also prepared using the hydrothermal method. Hydrothermal method has also been used to develop graphitic carbon nitride (g-C<sub>3</sub>N<sub>4</sub>) QDs/graphene hydrogel where melamine is used to produce g-C<sub>3</sub>N<sub>4</sub> QDs [12]. Poonam Siwath *et al.* [13], used DI water and ethanol to dissolve the following precursors Ni/Co-acetate tetrahydrate in a urea solution and kept for stirring. Afterward, the mixture was hydrothermally heated at 125 °C for 6 h, then washed with DI water and ethanol and dried in a vacuum oven for 10 h. Finally, it was calcined for 4 h at various temperatures ranging from 300 °C to 500 °C, however the author found that the sample calcined at 300 °C exhibited superior capacitive behavior. Although hydrothermal is the frequent method employed to prepare QDs for supercapacitor applications. Few other synthesis approaches are also employed. Abu Jahid Akhtar and the group used the wet-precipitation method to prepare different Co-doped ZnO QD samples [Zn<sub>1-x</sub>Co<sub>x</sub>O (x = 0, 0.02, 0.04)] [14]. In this method,

## CHAPTER 11

# Metal-Organic Frameworks (MOFs) Based Nanomaterials for Supercapacitor Applications

Pardeep K. Jha<sup>1,\*</sup>, Priyanka A. Jha<sup>1</sup> and Prabhakar Singh<sup>1</sup>

<sup>1</sup> Department of Physics, Indian Institute of Technology (Banaras Hindu University), Varanasi-221005, India

**Abstract:** In the last two decades, nanomaterials with enhanced active sites and better surface kinetics as compared to their bulk counterpart, have been significantly studied for supercapacitor electrode materials. Contemporarily, Metal-organic frameworks (MOFs) by virtue of versatile structure, charge conduction, high porosity, and redox-active functionality have also emerged as the most potential materials for next-generation energy storage technologies. Despite these excellent features, the bulk phase inorganic-MOFs have some chemical and physical limitations that hinder cell performance and thus novel materials are required. Recently, MOFs-based nanomaterials (nMOF) got due attention leading to the discovery of a variety of properties not observed or relevant in bulk systems, such as well-defined 3D structures, permanent porosity, and accelerated adsorption/desorption kinetics. That's why nMOFs are considered an emerging class of modular nanomaterials. However, understanding of nMOFs is still in its infancy, film uniformity along with the unstable structure in a highly corrosive electrolyte is still a bottleneck problem. In this chapter, the recent developments of pristine MOF and MOF-derived porous nanocomposites for the next-generation supercapacitor applications will be discussed.

**Keywords:** Metal-organic frameworks (MOFs), Nanomaterials, Supercapacitors.

## INTRODUCTION

Supercapacitors are an electrochemical device that has the best energy storage performance features of batteries and capacitors. It has higher power density, charge/discharge efficiency and life cycle than batteries, and higher energy density than capacitors. The first electrochemical cell was patented in 1957 (by General Electric's H.I. Becker) and commercially viable by 1975. Since then, supercapacitors have been serving society with their applications in the automotive industry, hybrid transportation systems, traction, and grid stabilization. Supercapacitors are combined with various other energy sources to

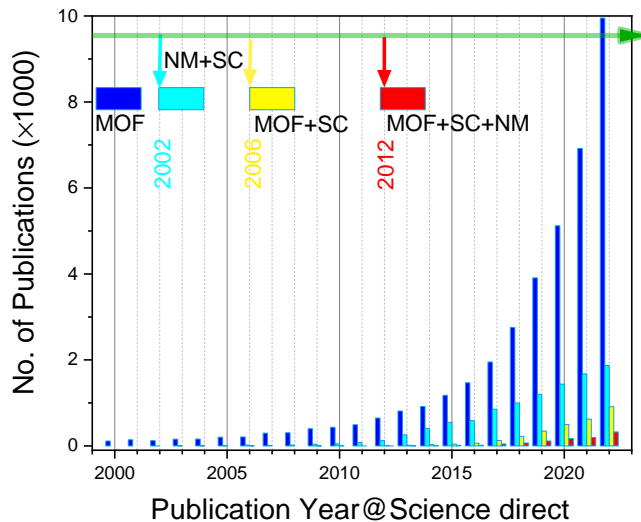
\* Corresponding author Pardeep K. Jha: Department of Physics, Indian Institute of Technology (Banaras Hindu University), Varanasi-221005, India; E-mail: pardeepjha.jit@gmail.com



improve performance. Their combination with fuel cells helps in rapid charging. Its essentiality will soon be reflected at charging stations with the expansion of electrical vehicles in the global market. However, for next-generation supercapacitors, we still require better performance than recent supercapacitors in use at a lower cost.

The supercapacitors consist of an electrolyte in which two porous electrodes (along with its separator in between and current collectors on both ends) are immersed/placed. For better capacitive performance, porosity and surface area of electrodes play pivotal roles. On the basis of charge storage mechanism capacitive or Faradaic, electrode materials for supercapacitors can be broadly categorized into three groups: capacitive (electrochemical double layer capacitor, EDLC), battery-like materials, and pseudo-capacitive (pseudo-capacitor) [1, 2]. The carbon materials-based electrode (CE) is generally capacitive in nature and therefore used in EDLC. This is one of the basic features of CE, by which, we, in general, distinguish EDLC from PC [1 - 3]. The capacitive features of EDLCs are a function of electrical conductivity and surface area. The active electrode materials employed in EDLCs are porous carbons like carbon nanotubes, activated carbon, and cross-linked or holey graphene [4, 5]. However, the fostering of carbon materials synthesis technique is not only a way to improve EDLCs but requires out of the box thinking, *i.e.*, a conception of entirely different class materials with optimized surface area and electrical conductivity. Considering this, the nanomaterials (NMs) were used for SCs fabrication in the year ~2002 as shown in the timeline in Fig. (1). Contemporarily metal-organic frameworks [6 - 8] (MOFs, with surface areas exceeding  $7,000 \text{ m}^2 \text{ g}^{-1}$  [7]) and another innovative class of porous crystalline materials have been developing since 1995 [9, 10]. MOFs with their versatile structure with open metal sites, high porosity, electrical conductivity, and redox-active functionality turned up as promising materials for energy storage technologies. That is why soon after NMs, MOFs-based SCs were also introduced in ~2006 (see timeline in Fig. (1)). MOFs are the structural combination of metals and organic linkers. This structural modularity permits not only creative design but also gives plenty of scope to engineer the porous structure, morphology, stability, and functionality for desired applications [11, 12]. The MOFs are directly employed as supercapacitor electrodes or as a template for fabricating different MOF-composites for supercapacitor electrodes. Some MOFs (pristine) are listed in Table 1. In order to increase the functionality of these MOFs, understanding the structure-property relation in these MOFs plays a pivotal role. For instance, MOF-5, being one of the earliest MOFs, possess a  $\text{ZnO}_4$ - tetrahedral-type entity originating from Zn ions and 1,4-benzene-dicarboxylate (BDC). It is also termed IRMOF-1 (here IRMOFs means isorecticular MOFs) as its frameworks are formed with the same structural topology, by organic linkers with different lengths and functionalities [13]. For

instance, pore size in MOF-5 is altered *via* using BDC and its derivative linkers and hence functionality.



**Fig. (1).** Journey of MOF in supercapacitors along with nanomaterials.

In spite of excellent features, the bulk phase MOFs have some physiochemical limitations that hinder cell performance. Very soon in ~2012 as shown in Fig. (1) it was realized that MOF-based nanomaterials (nMOF) could enhance the storage capacity after the introduction of MOFs to superconductors and therefore, got due attention. Consequently, many novel observations were found which were absent in the bulk, and it was understood that the decreased particle size of nMOF is the prime requisite for the creation of large surface area electrodes along with small diffusion paths [11]. For instance, UiO-66-based supercapacitors exhibited significantly higher charge storage for smaller particles [12]. That is why nMOFs are considered an emerging class of modular nanomaterials.

In this chapter, we will first cover some basics of nanomaterials synthesis for MOFs. It will be followed by morphological classification of MOFs: dimensional morphology (0-3D nanomaterials) and compositional morphology (pristine MOF, carbon and non-carbon based nanocomposites). In the last, we will discuss and summarize the recent developments for the next-generation supercapacitor applications.

## MXene-based Nanomaterials for High-performance Supercapacitor Applications

Zaheer Ud Din Babar<sup>1,2</sup>, Ayesha Zaheer<sup>2</sup>, Jahan Zeb Hassan<sup>3</sup>, Ali Raza<sup>2</sup> and Asif Mahmood<sup>4,5,\*</sup>

<sup>1</sup> *Scuola Superiore Meridionale (SSM), University of Naples Federico II, Largo S. Marcellino, 10, 80138, Italy*

<sup>2</sup> *Department of Physics “Ettore Pancini”, University of Naples Federico II, Piazzale Tecchio, 80, 80125 Naples, Italy*

<sup>3</sup> *Department of Physics, Riphah Institute of Computing and Applied Sciences (RICAS), Riphah International University, 14 Ali Road, Lahore, Pakistan*

<sup>4</sup> *School of Chemical and Biomolecular Engineering, The University of Sydney, Sydney, Australia*

<sup>5</sup> *Center for Clean Energy Technology, School of Mathematical and Physical Sciences, Faculty of Science, University of Technology Sydney, Sydney, Australia*

**Abstract:** Technological advances in recent decades have augmented the demand for durable and inexpensive energy storage devices with higher charge capacity. Owing to their unique charge storage and surface capability, a recent class of two-dimensional (2D) materials known as MXenes has been widely used in energy storage devices. MXenes are the layered transition metal carbides, nitrides, and/or carbonitrides produced *via* selective etching of interleaved “A” layers from parent MAX phases. Unlike other 2D materials, MXenes earned great attention because of their intrinsic surface functional groups, hydrophilicity, unique electrochemical nature, high conductivity, and superior charge storage capacity. Such features render MXenes as the ultimate material from the 2D family, thus inspiring researchers to delve further into experimental and theoretical realms. Numerous attempts have been made to elucidate synthesis strategies to produce MXene and its fundamental characteristics. The current chapter emphasizes the recent advancements in MXene-based electrochemical energy storage applications using supercapacitors which are recognized as a dominant source. The effect of MXene's morphology and electrode growth on the charge-storage mechanism has also been highlighted in subsequent sections. In addition, this chapter outlines the current state-of-the-art on the supercapacitors comprised of the MXene-based composites. A discussion of relevant challenges associated with such materials for energy storage applications is also presented, and future perspectives provide additional insight into their practical aspects.

\* **Corresponding author Asif Mahmood:** School of Chemical and Biomolecular Engineering, The University of Sydney, Sydney, Australia, Center for Clean Energy Technology, School of Mathematical and Physical Sciences, Faculty of Science, University of Technology Sydney, Sydney, Australia.; E-mail: asif.mahmood@uts.edu.au

**Keywords:** Composite, Energy storage, MXene, Supercapacitor, 2D material.

## INTRODUCTION

MXenes are 2D layered nitrides, carbides, or carbonitrides of primary transition metals produced *via* selective etching of interleaved “A” layers from their parent MAX phases. They were named “MXene” due to their structural and morphological similarities with graphene [1]. MAX phases serve as the precursor to MXene. They are denoted as  $M_{n+1}AX_n$  (where  $n = 1$  to 4), where “M” is the transition metal that belongs to the elements from groups III-VI of the periodic table (*i.e.*, Nb, Mo, V, Mn, Ta, Cr, Ti, Zr, *etc.*), “A” represents group IIIA/IVA elements (*i.e.*, In, Ga, Ge, Al, As, Pb, Sn, and Si, *etc.*). At the same time, “X” indicates carbide (C), nitride (N) and/or carbonitride (CN). The layers of “M” atoms are interweaved with the “A” layers, whereas the “X” atoms reside in octahedral sites in MAX phases with  $D_{6h_4}$ - $P6_3/mmc$  close-packed symmetry [2]. As the layers in transition metal dichalcogenide and Graphene have van der Waals (vdWs) bonding, the MX layers in MXenes have mixed ionic, metallic, and covalent nature. In contrast, MA layers have only metallic bonds. Due to the reactive nature and faint bonding among M-A layers for IIIA/IVA elements with fluoride-containing acids, it is easy to remove the A layers selectively *via* a wet-chemical etching route to yield MXenes [3]. MXenes are formed after this discriminating chemical engraving of A layers (covalently associated) using MAX phases and represented as  $M_{n+1}X_nT_x$ . After etching, the MXenes surface is terminated with a distinct surface group, *e.g.*, -Cl, -F, -OH, or -O as represented by “T” whereas the “n” value specifies the number of layers [4]. These surface terminal groups impart hydrophilic behaviour to MXene's sheets [5]. Furthermore, ion transport and other electronic features (*e.g.*, bandgap) of MXene are essentially governed by surface termination groups [6]. Also, the environmental stability of MXenes (*e.g.*, oxidation) depends on the “n” values. Higher values of “n” result in the yield of more stable MXene [7].

In 2011, a research group at Drexel University led by Professors M. W. Barsoum and Yuri Gogotsi described the experimental synthesis of  $Ti_3C_2T_x$  [8] *via* selective removal of “Al” by immersing MAX phase precursor ( $Ti_3AlC_2$ ) in hydrofluoric acid as an etchant [9].  $Ti_2CT_x$ ,  $Zr_3C_2T_x$ ,  $Nb_2CT_x$ ,  $Nb_4C_3T_x$ ,  $V_2CT_x$ ,  $Ti_3CNT_x$ ,  $Mo_2CT_x$ ,  $Ti_4N_3T_x$ ,  $Mo_4VC_4T_x$ ,  $Mo_2ScC_2T_x$ ,  $(Ti_{0.5}, Nb_{0.5})_2CT_x$ ,  $(Nb_{0.8}, Ti_{0.2})_4C_3T_x$ , and  $(Nb_{0.8}, Zr_{0.2})_4C_3T_x$  are examples of the MXenes that produced experimentally till date. Due to its rare blend of ceramic (C/N atoms) and metallic characteristics, a wide range of applications have been studied using MXenes. More than 100 MAX phases have been designed using different recipes of MAX phase components [10]. Nevertheless, about 30 MXenes are synthesized experimentally, with their characteristics thoroughly evaluated. They created a pathway for research and the

MXene community to realize new MXene structures with distinct features. Due to better surface chemistry, MXene has large metallic conductivity ranges from  $\sim 6000\text{-}8000\text{ S}\cdot\text{cm}^{-1}$  [11], better thermal conductivity [12], exceptional mechanical stability [13], promising optical properties [14], excellent electric and magnetic properties [15], intrinsic hydrophilicity [16], abundant surface terminations [17]. Moreover, MXenes can sufficiently intercalate external species among its layers [18, 19]. These distinctive qualities and easy post-processing have made MXenes ideal candidates for numerous applications, including nuclear waste treatment [20], antibacterial activities and water purification [21], sensors [22], photocatalysis [23], solid lubricants [24], electromagnetic interference (EMI) shielding [25], energy conversion devices and transparent conductors in electronics [26], among others. Owing to the micro-scale lateral dimension, higher hydrophilicity, and atomic layer thickness of MXenes, it is possible to recognize thin layers of MXene *via* vacuum-aided filtration or simply by spray coating/printing. The hydrophilicity of  $\text{Ti}_3\text{C}_2\text{T}$  was studied by Ghidui *et al.* by measuring the contact angle of water on MXene film [11]. As compared to semiconductor-type metal oxides, MXene has a greater power density due to strong conductivity, thus facilitating the devices to charge quickly [27]. In addition to these features, the strong electronegativity and rich surface chemistry of MXenes have made it possible to fabricate composite electrodes [28, 29]. In addition, MXenes are better substrate materials for catalysis applications because of the fast electron transport between their layers [30]. Also, MXenes exhibit various characteristics depending on their compositions, elemental stoichiometries, synthesis routes, interlayer spacing, and lateral flake sizes. For example, according to Seh *et al.*,  $\text{Mo}_2\text{CT}_x$  MXenes have higher catalytic activity than  $\text{Ti}_2\text{CT}_x$  [31]. Nitride-assisted MXenes show better electrical conductivity than carbide-assisted MXenes; conversely, carbide-assisted MXenes have superior structural stability [32]. Moreover, the configuration of the exterior terminal groups affects the characteristics of MXene. Amid several surface terminations on  $\text{Ti}_3\text{C}_2$  MXene, O-terminated MXene ( $\text{Ti}_3\text{C}_2\text{O}_2$ ) is more stable than the others, while H-terminated ( $\text{Ti}_3\text{C}_2\text{H}_2$ ) is the least stable. Moreover, the order of stability is  $\text{Ti}_3\text{C}_2\text{F}_2 > \text{Ti}_3\text{C}_2(\text{OH})_2 > \text{Ti}_3\text{C}_2\text{H}_2 > \text{Ti}_3\text{C}_2$  [33]. Various devices have been proposed through the advancement of technology, from microchips to massive electronic devices that require energy to function.

To overcome this, devices with adequate energy storage must be durable and have minimal production and processing costs. It includes a wide-ranging use of batteries for energy storage applications, including sodium-sulfur [34], lithium-ion [35], sodium-ion batteries [36], and supercapacitors [37]. Batteries have an enhanced energy density but low power density though the case of capacitors conflicts with batteries. Supercapacitors perform far better as energy storage devices than batteries owing to their increasing power density, significant energy

## Recent Developments in the Field of Supercapacitor Materials

Mani Jayakumar<sup>1</sup> and Venkatesa Prabhu S.<sup>2,\*</sup>

<sup>1</sup> Department of Chemical Engineering, Haramaya Institute of Technology, Haramaya University, Haramaya, Dire Dawa, Ethiopia

<sup>2</sup> Center of Excellence for Bioprocess and Biotechnology, Department of Chemical Engineering, College of Biological and Chemical Engineering, Addis Ababa Science and Technology University, Addis Ababa, Ethiopia

**Abstract:** Energy storage is one of the crucial requirements for today's life to store energy for later use. Energy storage critically reduces the dependence on backup power supplies. In recent times, fascinatingly, energy storage systems have been developed in compactable sizes and shapes with sustainable and appreciable backup power. However, due to some challenges in conventional storage systems, supercapacitors are gaining a huge interest which satisfies the increasing demands of energy storage devices. Supercapacitors are well-recognized for their long cycle life, high power density, and the ability for less charge and discharge time. Accordingly, in supercapacitor electrodes, activated carbon is extensively used as a base material. Current research documented that the amorphous mixed metal oxides and nanostructured oxides are also used in supercapacitor devices to exhibit high performance. Keeping this in view, this chapter is reviewed to provide information on the different types of recent developments in supercapacitors and their performance. In addition, recent developments in green approaches to supercapacitor applications in MnO<sub>2</sub>-based electrodes and composites, supercapacitors performance, power capability, and cycle life are also discussed.

**Keywords:** Activated carbon, And capacity performance, Composites, Metal oxides, Power capability, Supercapacitors.

### INTRODUCTION

Nowadays, environmental pollution significantly influences climate change resulting in different impacts on the global economy, which triggers the utilization of fossil fuels like coal and natural gas [1]. To address these issues and appropriate exploitation of existing energy, developing a strategy for utilizing

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\* Corresponding author Venkatesa Prabhu S.: Center of Excellence for Bioprocess and Biotechnology, Department of Chemical Engineering, College of Biological and Chemical Engineering, Addis Ababa Science and Technology University, Addis Ababa, Ethiopia; E-mail: venkatesa.prabhu@aastu.edu.et

renewable forms of energy (wind, tidal, and solar) is much required. However, some concerns hinder the use of resources for renewable energy, hence, the production of electricity seems to be inconsistent. Accordingly, there is an urgent need for developing sustainable, and effective energy storage systems [2]. Undeniably, there may be an unexpected imbalance between the demand and supply of electricity that may create unwanted temporal gaps and nonexistent spatial grid systems for the end users. To address this issue, there is a crucial requirement for energy storage systems. In recent times, supercapacitors (SCs), are a new form of energy storage devices that are gaining substantial attention since they exhibit a fast rate of discharge, comparatively long cycle life, and high-power density. Studies show that supercapacitors have prominent potential applications in hybrid power automobiles, electronic devices on portable scale, and even in renewable energy systems. By definition, supercapacitors are energy source devices that can be positioned between capacitors and batteries [3]. Compared to normal capacitors, SCs have a good capacity to hold larger energy and can deliver it at larger power units. In addition to this, SCs have well-appreciable cyclability and long-lasting stability. The application of SCs is still being utilized in different energy sectors and industries. In general, the SCs are classified as pseudo capacitors and electrical double-layer capacitors. Normally, electrical double-layer capacitors accumulate the charge on the interface between the electrode and electrolyte. Differently, for the pseudo capacitors, the energy is stored *via* the process, called, reversible faradaic redox [4]. The two key factors, operative voltage, and capacitive voltage, are significantly influencing the energy density. In such a way, energy density could be promoted towards the improvement of the capacitance performance of the electrode and raising the potential window. So far, several investigations have been carried out toward ameliorating the electrochemical activity of SCs which was found to be dependent on the design and synthesis procedures of materials. In this line, the materials for preparing electrodes, specifically for SCs, show a substantial impact on their performance [5].

In general, carbon materials, conductive polymers, and transition metal oxides are the three different materials used for electrode preparation. Carbon-based materials are generally used in electrical double-layer capacitors as electrodes [6]. They have high specific surface area and well-distributed pore size that can be adjusted to attain better electrical conductivity. In realization, studies revealed that carbon-based materials can provide high power density with deprived energy density. This is because of the method followed for storage in electrical double-layer capacitors that restricts the overall outcomes [7]. Accordingly, graphene, carbon nanotubes, and carbon nanofibers have been well-explored carbon-based materials as electrodes. Further investigations explicated that carbon-based materials have significantly affected the capacity of electrical double-layer

capacitors. Additionally, the high cost of such materials limits the usage of these materials. The conductive polymers are known to be good, but they exhibit low stability. Recently, transition metal oxide-based electrode materials have been found to have much better energy density, chemical stability, and specific capacity than conductive polymers. Up to now, various transition metal oxides, such as  $\text{MnO}_2$ ,  $\text{ZnO}$ , and  $\text{Co}_3\text{O}_4$  are being studied for achieving extraordinary capacitance materials [8].

However, some metal oxides, like  $\text{Co}_2\text{O}_4$ ,  $\text{Ni}_2\text{O}_4$ ,  $\text{Mn}_2\text{O}_4$ , and  $\text{Mo}_2\text{O}_4$  show comparatively low electrical conductivity due to their synergistic effects of elements and metal ions. Interestingly, spinel cobaltates ( $\text{CuCo}_2\text{O}_4$ ,  $\text{MnCo}_2\text{O}_4$ ,  $\text{NiCo}_2\text{O}_4$ , *etc.*) and metal molybdates are gaining potential interest because of easy availability, low cost, and enhanced electrochemical activity [9]. Numerous classes of supercapacitors are illustrated in Fig. (1).

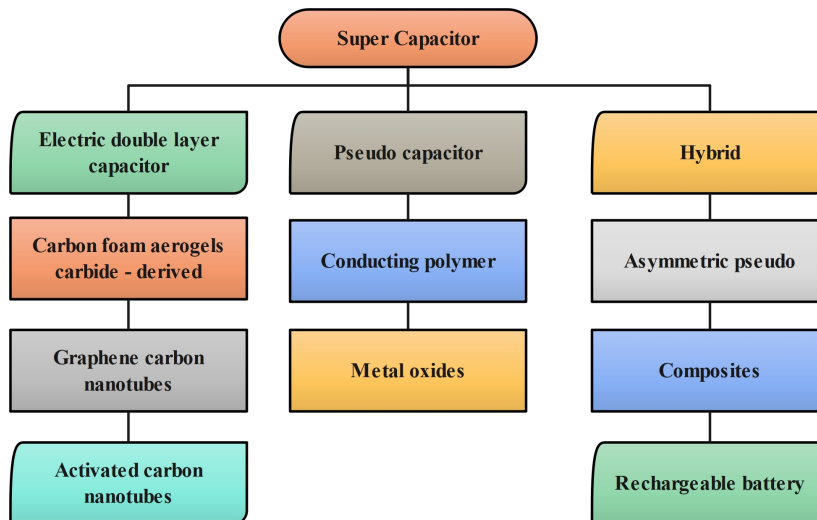


Fig. (1). Different types of Supercapacitors.

### Carbon composite-based Supercapacitor Electrode Materials

Kim and co-researchers [10] have fabricated stretchable SC electrodes using a network of graphene/CNT. The fabricated electrodes have structures like a vertically aligned honeycomb. They adopted a crystallization process followed by radial compression to prepare reentrant structures. Fascinatingly, such a porous network provides better stretchability. Moreover, the resulting fabrication shows excellent conductivities. The studies revealed that such vertically aligned structures can overcome some issues based on the ion-accessible and tensile properties of the electrode. The stretchable property of the electrode is essential for designing compactable epidermal electronics. Li and his co-workers [11]



## Supercapacitor Materials: From Research to the Real World

Ahmad Nawaz<sup>1,2</sup>, Vikas Kumar Pandey<sup>2</sup> and Pradeep Kumar<sup>2,\*</sup>

<sup>1</sup> Center for Refining & Advanced Chemicals, King Fahd University of Petroleum and Minerals, Dhahran, 31261, Saudi Arabia

<sup>2</sup> Department of Chemical Engineering & Technology, Indian Institute of Technology (Banaras Hindu University), Varanasi-221005, India

**Abstract:** Supercapacitors are gaining prominence in the realm of energy storage devices due to their high power density, extended cycle stability, and fast charge/discharge rates. Supercapacitors are widely used in industries such as service grids, transportation, consumer electronics, wearable and flexible systems, energy harvesting, *etc.* Due to their remarkable high-power performance, high reliability, and extended lifetime, they are a key electrochemical device for energy storage; as a result, the worldwide supercapacitor market is rapidly developing. Supercapacitors have a straightforward basic construction, but different products for various applications require cells in various configurations. The application of supercapacitors from the perspective of the industry is the subject of this chapter.

**Keywords:** Electrolytes, Energy storage, Electrochemical supercapacitors.

### INTRODUCTION

Supercapacitors (SCs) first became commercially available in 1957 when General Electric obtained a patent for a specific type of electrolytic capacitor (EC) which included a porous carbon electrode, known as a double-layer capacitor. However, the Standard Oil Company (SOC) obtained a patent for a capacitor that resembled a disc and was made from carbon paste that had been electrolyte-soaked. Nippon Electric Corporation (NEC), who later produced the first commercially effective supercapacitor, received a license to use the patent in 1971. The primary use of these early supercapacitors was generally as memory backup for electronic applications. Several businesses signified by Panasonic have underway to make supercapacitors with a focus on memory backup devices. These early results generated interest in additional uses, likewise in electric vehicles of the hybrid

\* Corresponding author Pradeep Kumar: Department of Chemical Engineering & Technology, Indian Institute of Technology (Banaras Hindu University), Varanasi-221005, India; E-mail: pkumar.che@itbhu.ac.in

type. After that, supercapacitors' dependability and lifecycle have substantially improved [1].

Supercapacitors are classified based on their charge storage techniques such as ELDCs (electric double-layer capacitors), pseudocapacitors, and hybrids. EDLCs use adsorption to accumulate charges on the electrolyte-electrode interface; on the other hand, pseudocapacitors utilize reversible redox (Faradaic) reactions. The hybrid supercapacitors employ a combination of EDLC and pseudocapacitance to store electrochemical energy [2 - 4]. Currently, supercapacitors are highly advanced amongst the pivotal patents in the middle of the 20<sup>th</sup> century, because of their higher performance in terms of power, outstanding reliability, and lifecycle. They have played an immensely and continuously expanding role in the development of modern society. It has evolved into a substantial category of electrochemical energy-storage devices on its own; they are commonplace in daily life and come in a variety of shapes depending on the particular application scenarios. This chapter will provide an introduction to the process of making supercapacitors, focusing mostly on the viewpoint of the industry including their device designs, manufacturing procedures, and problems to take into account when attempting to improve performance.

A supercapacitor's fundamental design consists of two porous dielectric films that operate as a separator. Current collectors covered with active electrode materials are typically constructed of metals and are found between separators. The sandwiched structure is then infused with an electrolyte, which can be an organic or aqueous solution containing a significant amount of ions. The size of the basic unit is controlled by the needed capacitance and voltage, and it can be fixed to form a flat pouch cell or rolled to form a radical structure. Carbon electrodes and organic electrolytes are now the industry's typical material options. The device design may be briefly divided into small, medium, and large cells and is significantly dependent on the desired applications. Supercapacitors are made in the following ways: electrode fabrication, cell assembly, separator location, electrolyte impregnation, external connection, and system sealing. The major objectives of supercapacitor manufacturing's industrialization efforts are to boost device performance and decrease ESR (equivalent series resistance), increase resilience, and lower prices as well as the lifecycle of the entire cell. Supercapacitor applications can be broadly classified into 2 categories: (i) High capacitance supercapacitors (HCS) utilized in elevators, uninterruptible power supplies (UPS), and other applications. Supercapacitors are typically built into modules for these purposes and coupled within an electrical balancing circuit. (2) Low capacitance supercapacitors (LCS) are utilized for a variety of electrical functions such as voltage stability and backup. In these applications, supercapacitors are commonly directly welded into the circuit board and are

typically the same size as other electronic components such as electrolytic and dielectric capacitors.

## COMPONENTS

### Electrodes

#### *Current Collector*

Most commercial supercapacitors need the deposition of active materials on a metallic current collector in order to lower the resistance, with the exception of a few assemblies that use self-support electrodes instead of current collectors.

An electrode that can stand on its own is made up of conductive carbon fabric. The equivalent series resistance (ESR) is frequently too high in the latter scenario for the effectiveness of high power density. Yet, the self-supporting electrode method is more preferential when manufacturing flexible supercapacitors. The electrode is connected to the external circuit through the current collector. The increase in ESR during cycling is mostly caused by the active material being detached from the current collector. The supercapacitors (SCs) used for commercial purposes are crucial to optimize the mechanical and chemical adherence of electrode material to current collectors. The most extensively used technique in the sector involves coating the current collector with an aqueous or organic slurry that contains activated carbon. Additionally, activated carbon can be directly laminated onto the current collector [5]. The fabrication of thick electrodes is not appropriate for this technique. The different functional groups attached to activated carbon can be used in chemical processes to increase adhesion [6]. Physical methods such as plasma can also be employed among activated carbon and aluminium current collectors to increase adhesion.

Stability in electrolytes is one of the factors to consider when selecting a current collector. Processability, density, and cost are essential to select appropriate current collectors. In an organic electrolyte, aluminium is a good choice for the collector as it is inexpensive, has a low density, and in typical organic electrolytes, and good electrochemical stability. Furthermore, its surface can be given a particular treatment to improve the adhesion between active substances. For such applications, there are two main types of Al: (1) Standard Low-cost aluminium foil is used. It is challenging to preserve material and current collector using traditional binders like polyvinylidene fluoride and polytetrafluoroethylene.

**CHAPTER 15****Future Outlook and Challenges for Supercapacitors****Vikas Kumar Pandey<sup>1</sup> and Bhawna Verma<sup>1,\*</sup>**<sup>1</sup> *Department of Chemical Engineering & Technology, Indian Institute of Technology (Banaras Hindu University), Varanasi-221005, India*

**Abstract:** The contemporary research environment calls for developing next-generation devices using cutting-edge technologies for energy storage applications. Supercapacitors are becoming burgeoning contenders in the energy sector due to their increased durability and quicker charge storage capacity. In contrast to batteries and fuel cells, supercapacitors are a less realistic solution for practical applications. Additionally, there is a pressing need for fabrication techniques that must be addressed to deliver an appropriate supercapacitor electrode. The book chapter will better describe the difficulties encountered during various supercapacitor research, development, and commercial application phases. Finally, a conclusive prognosis has been given on how the discussion above will deliver essential insights and create chances to expand the possible application of new-generation supercapacitors.

**Keywords:** EDLCs, Energy storage, Pseudocapacitors, Supercapacitors.

**INTRODUCTION**

The most active research area for the current generation, second only to battery development, is electrochemical supercapacitors, also known as ultracapacitors. Between conventional capacitors and batteries, supercapacitors act as a bridge. There are different types of supercapacitor devices such as wearable supercapacitors, flexible supercapacitors, hybrid supercapacitors, and conventional supercapacitors as illustrated in Fig. (1). The in-depth research and development into new materials for supercapacitor component components have enhanced the electrochemical performance and feasibility of the technology in recent years. Significant technical breakthroughs in supercapacitors have been made in the last ten years thanks to significant advancements in material development. The supercapacitor electrode and electrolyte materials are two important domains that require attention. Higher energy storage capacities will be

\* **Corresponding author Bhawna Verma:** Department of Chemical Engineering & Technology, Indian Institute of Technology (Banaras Hindu University), Varanasi-221005, India; E-mail: bverma.che@itbhu.ac.in

made possible by increasing the capacitances of active electrode materials. However, cost and cyclability difficulties must be considered while creating these high-capacitance materials. Important parts of supercapacitor devices include electrolyte materials (solvents and ionic species). Aqueous electrolyte solutions have typically been employed as they are easy to handle and non-hazardous. On the contrary, the electrochemically stable operating potential windows provided by aqueous electrolytes are very small ( $\sim 1.0$  V).



**Fig. (1).** Different types of supercapacitor devices.

Alternative electrolyte solutions with significantly larger operating voltage stability windows can significantly increase the energy storage capacity of supercapacitors since the energy storage capacity is directly proportional to the square of the operating voltage. Researchers are examining organic and ionic liquid electrolytes on that front. Organic electrolytes are used in the majority of commercially marketed supercapacitor systems. However, there are several material handling and technological challenges with organic and ionic liquid electrolytes that must be resolved.

Several difficulties persist despite significant advancements in the supercapacitor electrode and electrolyte material fields in recent years. This chapter will first

cover the market obstacles to supercapacitor development efforts, then it will go into great depth about the development of electrode and electrolyte materials, including the advances made and the difficulties encountered. Also computational techniques will be covered that can be used to support efforts at material development. Finally, a few viewpoints and future directions for study and development will be examined.

**MARKET CHALLENGES**

Although a small number of supercapacitor devices are currently available, they continue to have several drawbacks, including high cost, a lack of performance capabilities, and issues with long-term endurance. Future energy systems will undoubtedly include supercapacitors as integral components, but to get around the challenges and increase this emerging technology's economic viability, considerable efforts are needed to build new system components. A comparison between specific power density vs. specific energy density (Ragone plots) for different electrochemical energy storage and conversion devices has been illustrated in Fig. (2).

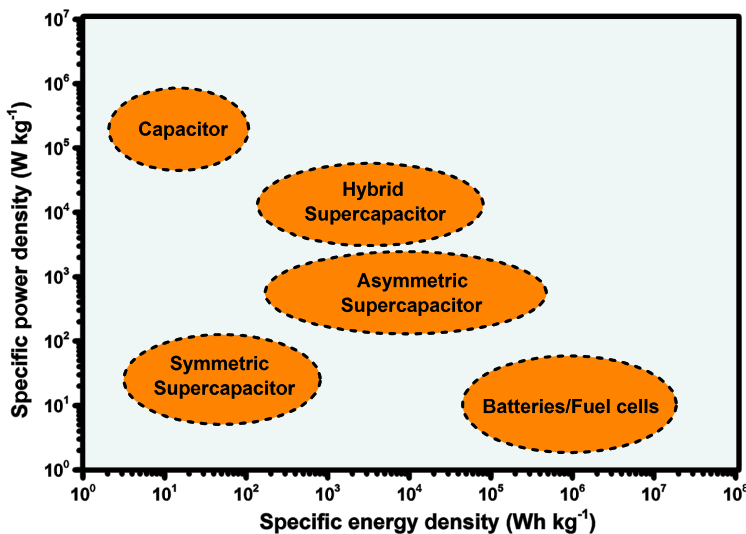


Fig. (2). Ragone plot showing energy density vs. power density for various electrochemical energy storage and conversion devices.

If supercapacitor devices are to preserve their competitive advantages over current battery technology, energy density must be significantly increased. Recent developments in this area, including using organic and alternative electrolytes with higher operational voltages, and significant advancements have been

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## Sanjeev Verma

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Prof. Sanjeev Verma received his Ph.D. in chemical engineering from the Indian Institute of Technology (BHU), Varanasi in 2022. During his Ph.D., he worked in the energy storage materials for supercapacitor energy storage application which has broader application in the energy storage, mobility, and electric vehicle industries. Currently, Dr. Sanjeev Verma is a manager working in the department of battery manufacturing new energy, at Reliance Industries Limited, India. He has authored around 15 research publications and book chapters.



## Shivani Verma

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Prof. Shivani Verma is an assistant professor in the Department of Chemistry at the School of Physical Sciences, Doon University, Dehradun, India. She obtained a Ph.D. in chemical sciences division, specialization in organic synthetic chemistry and agricultural chemistry from G. B. Pant University of Agriculture and Technology, Pantnagar, India. Her research interests include energy storage materials, synthetic chemistry, heterocyclic compounds, heterogeneous catalyst, ionic liquids, green chemistry, agricultural chemistry, and pesticide residue analysis. She has more than 10 research papers and 4 book chapters.



## Saurabh Kumar

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Prof. Saurabh Kumar received a Ph.D. degree in RF and Microwave Engineering from PDPM Indian Institute of Information Technology, Design and Manufacturing, Jabalpur in June 2017. He is silver medalist in Ph.D. and gold medalist in M.Tech degree. Currently, Prof. Kumar is working as an assistant professor in the Department of Electronics & Communication Engineering, NIT Hamirpur, HP, India, since 2018. He has authored more than 25 research articles in reputed journals and conferences. He serves as a reviewer to many journals and conferences of repute. His research interests include energy harvesting circuits, supercapacitors, planar and microstrip patch antennas, artificial materials like meta-materials, electromagnetic theory, RF and microwave engineering, nanophotonics and plasmonics etc.



## Bhawna Verma

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Prof. Bhawna Verma is an associate professor in the department of Chemical Engineering & Technology at IIT(BHU), Varanasi. She holds a Ph.D. (IIT (BHU)), M. Tech. (IT BHU) and B.Tech. (NIT Raipur) in chemical engineering. Her research interests include: advanced composite/nanomaterials for enhanced capacitance, waste water treatment, biodiesel, catalysis, and heat transfer in narrow channels. She has authored around 50 research publications, conference papers and book chapters.