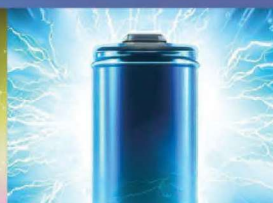




2D NANOMATERIALS



Synthesis, Properties and Applications

Edited by
Subhendu Chakroborty and Kaushik Pal

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Synthesis, Properties and Applications

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WILEY

This edition first published 2024 by John Wiley & Sons, Inc., 111 River Street, Hoboken, NJ 07030, USA and Scrivener Publishing LLC, 100 Cummings Center, Suite 541J, Beverly, MA 01915, USA

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Library of Congress Cataloging-in-Publication Data

ISBN 978-1-394-16649-7

Cover image: Pixabay.Com

Cover design by Russell Richardson

Set in size of 11pt and Minion Pro by Manila Typesetting Company, Makati, Philippines

Printed in the USA

10 9 8 7 6 5 4 3 2 1

Contents

Preface	xvii
Part I: Synthesis of 2D Nanomaterials	1
1 Top-Down Strategies Synthesis of 2D Nanomaterial	3
<i>Pranjyan Dash and Pradeep Kumar Panda</i>	
1.1 Introduction	3
1.2 Top-Down Strategy Synthesis Method	5
1.2.1 Etching	7
1.2.2 Mechanical Milling	8
1.2.3 Sputtering	9
1.3 Laser Ablation	10
1.4 Characterizations and Toxicity of 2D Nanomaterials	11
1.5 Conclusions	12
References	12
2 Bottom-Up Strategies for Synthesis of 2D Nanomaterial	17
<i>Nibedita Nath, Subhendu Chakroborty and Anita Routaray</i>	
2.1 Introduction	17
2.2 Types of 2D Nanomaterial	18
2.2.1 Graphene	18
2.2.2 MXenes	19
2.2.3 Black Phosphorus	19
2.2.4 Hexagonal Boron Nitride	19
2.2.5 Transition Metal Dichalcogenides	20
2.2.6 Graphitic Carbon Nitride	20
2.2.7 MOF and COF	20
2.3 Synthesis Strategies	21
2.3.1 Top-Down	22
2.3.1.1 Mechanical Milling	22
2.3.1.2 Electrospinning	22
2.3.1.3 Lithography	22

2.3.1.4	Sputtering	23
2.3.1.5	The Arc Discharge Method	23
2.3.1.6	Laser Ablation	23
2.3.2	Bottom-Up Method	23
2.3.2.1	Chemical Vapor Deposition	23
2.3.2.2	Sol–Gel Method	24
2.3.2.3	Solvothermal and Hydrothermal Methods	24
2.3.2.4	Soft and Hard Template and Reverse Micelle Methods	26
2.4	Bottom-Up Strategies for Synthesis of 2D Nanomaterial	26
2.5	Conclusion and Outlook	32
	References	33
3	Unveiling the Intricacies: Characterization Techniques for 2D Nanomaterials	43
	<i>Siba Soren, Subhendu Chakroborty, Rudra N. Purusottam and Amiya Ranjan Panda</i>	
3.1	Introduction	44
3.2	Characterization Techniques	45
3.2.1	XRD	46
3.2.2	SEM and TEM	47
3.2.3	Optical Microscope	48
3.2.4	AFM	49
3.2.5	XPS	50
3.2.6	RAMAN	51
3.3	Conclusion	53
	References	53
	Part II: Properties of 2D Nanomaterials	59
4	Crystal Structure, Magnetic and Mechanical Properties of 2D Nanomaterials	61
	<i>Nandini Roy</i>	
4.1	Introduction	62
4.2	Structure of 2D Materials	62
4.2.1	Graphene	62
4.2.2	Black Phosphorous	63
4.2.3	Transition Metal Dichalcogenide (TMDC)	64
4.3	Magnetic 2D Materials	65
4.4	Origin of Magnetization in 2D Materials	65
4.5	Mechanical Properties of 2D Nanomaterials	66
4.6	Conclusion	68
	References	68

5	Electrical, Plasmonic, and Optical Properties of 2D Nanomaterials	73
	<i>Ankita Subhrasmita Gadtya and Srikanta Moharana</i>	
5.1	Introduction	74
5.2	Overview of Two-Dimensional Nanomaterials (2D NMs)	77
5.3	Electrical Properties of 2D NMs	80
5.4	Optical Properties of 2D NMs	84
5.5	Plasmonic Properties of 2D NMs	85
5.6	Recent Applications of 2D NMs	87
5.6.1	2D NMs for BioMedical Application	87
5.6.2	2D NMs in the Field of Energy	90
5.6.3	2D NMs as Lubricant Additive	92
5.7	Challenges and Prospective	93
5.8	Conclusion	93
	Acknowledgments	94
	References	94

Part III: Application of 2D Nanomaterials **103**

6	Challenges Surrounding 2D Nanomaterials and Their Application to Photocatalytic Industrial Wastewater Treatment	105
	<i>Anchit Modi and N. K. Gaur</i>	
6.1	Introduction	106
6.2	Photocatalysis for Industrial Wastewater Treatment	107
6.2.1	Principles of Photocatalysis	108
6.2.2	Photocatalytic Processes for Industrial Wastewater Treatment	108
6.2.3	Advantages and Limitations of Photocatalysis	108
6.3	2D Nanomaterials in Photocatalysis	110
6.3.1	Introduction to 2D Nanomaterials and Types Used in Photocatalysis	110
6.3.2	Key Properties and Characteristics of 2D Nanomaterials	110
6.3.3	Role of 2D Nanomaterials in Enhancing Photocatalytic Performance	111
6.4	Challenges in Utilizing 2D Nanomaterials for Photocatalytic Wastewater Treatment	112
6.4.1	Synthesis and Fabrication Challenges	112
6.4.2	Stability and Degradation Issues	112
6.4.3	Efficiency and Selectivity Considerations	112

6.4.4	Scalability and Cost-Effectiveness Challenges	112
6.5	Strategies to Overcome Challenges	113
6.5.1	Improvement of Synthesis and Fabrication Techniques	113
6.5.2	Enhancement of Stability and Durability	113
6.5.3	Optimization of Photocatalytic Performance	113
6.5.4	Economical and Scalable Production Methods	114
6.6	Case Studies and Applications	114
6.6.1	Examples of Successful Applications of 2D Nanomaterials	114
6.6.2	Case Studies in Photocatalytic Industrial Wastewater Treatment	115
6.6.3	Lessons Learned and Future Prospects	115
6.7	Conclusion	116
	References	117
7	Application of 2D Nanomaterials for Energy Storage <i>Tulasi Barik and Subhendu Chakroborty</i>	121
7.1	Introduction	121
7.2	2D Nanomaterials for Application of Lithium Ion Batteries	122
7.3	Application of 2D Nanomaterials in Sodium Ion Batteries	129
7.4	Application of 2D Nanomaterials in Potassium Ion Batteries	134
7.5	Applications of 2D Nanomaterials in Supercapacitors	137
	Conclusions	141
	References	141
8	Innovation in Photoinduced Antibacterial 2D Nanomaterials <i>Zubaid ul Khazir Rather, Shabnam Kawoosa, Gulam Nabi Yattoo, Mohd Asif Hajam, Sajad Ahmed Bhat and Javid Ahmed Banday</i>	155
8.1	Introduction	156
8.2	Antibacterial Applications Based on Graphene-Induced Photostimulation	159
8.2.1	Nanomaterials for Antibacterial Transition-Metal Dichalcogenides/Oxides	164
8.2.2	Antibacterial Nanomaterials Based on Carbon Nitride	165
8.2.3	Antibacterial Nanomaterials Based on Black Phosphorus	166
8.2.4	Other 2D Antibacterial Nanomaterials	167
8.3	Antibacterial Mechanisms of Graphene-Based Family	168

8.3.1	Physical Contact Destruction	169
8.3.2	Oxidative Stress	169
8.3.3	Disruption of Bacterial Protein Interactions	170
8.3.4	Photo-Induced Mechanisms	170
8.4	Conclusion	172
	References	172
9	2D Nanomaterials for Drug Delivery System	185
	<i>Syed Muzammil Munawar, Dhandayuthabani Rajendiran and Kaleel Basha Sabjan</i>	
9.1	Introduction	186
9.2	2D Material Biosynthesis	190
9.3	Encapsulation of 2D Materials	192
9.4	Hydrogel Encapsulation—2D Materials	193
9.5	2D Material Encapsulation—Liposomes	194
9.6	2D Supply Encapsulation—Micelle	195
9.7	Stimuli Responsive 2D Material SDDSs—Classification	195
9.8	Light-Sensitive SDDSs	196
9.9	Magnetic Field-Responsive SDDSs	196
9.10	Various Response Exhibits Diverse—Advantages/ Disadvantages	197
9.11	2D Material SDDS Therapy—Cancer	197
9.12	Antibacterial	198
	9.12.1 Central Nervous System	198
9.13	Orthopedic	200
9.14	Diabetes Mellitus	200
9.15	2D Materials in Intelligent Drug Delivery System—Advantages	201
9.16	Disadvantages	201
9.17	Conclusion and Future Perspective	202
	Acknowledgements	203
	References	203
10	New Technology 2D Nanomaterials for Neural Tissue Engineering	209
	<i>Banti Baishya, Saurav Paul, Hillol Das, Utsab Singha and Dipyaman Mohanta</i>	
10.1	Introduction	210
10.2	Regeneration of Tissue and Organ Repair in Nature	211
	10.2.1 The ‘Curious Case’ of Lizard: A Nature’s Classic	211

10.2.2	Regenerative Capabilities of Amphibians	212
10.2.3	Regeneration in Humans	213
10.3	Nanotechnology and Neural Tissue Engineering	214
10.3.1	Definition of Nanotechnology	215
10.3.2	Synthesis of Nanomaterials or Nanoparticles	216
10.4	2D Nanomaterials for Tissue Engineering Application	218
10.4.1	Graphene-Based Nanomaterials in Tissue Engineering	219
10.4.2	Black-Phosphorus (BP)-Based Nanosheets in Tissue Engineering	220
10.4.3	Application of 2D Nanoclay in Tissue Engineering	221
10.5	2D Nanomaterials and Peripheral Nerve Engineering	224
10.5.1	Peripheral Nerve	224
10.5.2	Damage and Regeneration in Peripheral Nerve	225
10.5.3	Key Features of Nanomaterials in Neural Tissue Engineering	228
10.5.4	Mechanism of 2D Nanomaterial-Based Neural Regeneration	229
10.5.4.1	Graphene	230
10.5.4.2	Graphene Oxide	230
10.5.4.3	Black Phosphorus (BP)	230
10.6	Application of 2D Nanomaterials in Spinal Cord Repair	231
10.7	2D Nanomaterials for Drug/Gene Delivery	233
10.8	Challenges and Prospects	234
	References	235
11	Theranostic Approach of 2D Nanomaterials in Breast Cancer	241
	<i>Pravati Panda, Subhendu Chakroborty and Kaushik Pal</i>	
11.1	Introduction	242
11.2	Applications	243
	Conclusion	256
	Acknowledgments	256
	References	256
12	2D Nanomaterials for Photocatalytic Hydrogen Production	263
	<i>Grandprix T. M. Kadja, St Mardiana and Arxhel S. F. Nanda</i>	
12.1	Introduction	264
12.2	Basics of Photocatalytic Hydrogen Production	265

12.3	2D Nanomaterials for Photocatalytic Hydrogen Production	267
12.3.1	Graphene-Based	267
12.3.2	Carbon Nitrides	269
12.3.3	Transition Metal Dichalcogenides	271
12.3.4	MXene	273
12.4	Enhancing the Photocatalytic Performance	276
12.5	Conclusion and Outlook	280
	Acknowledgments	284
	References	284
13	Supercapacitor Based on 2D Nanomaterials and Their Hybrid	289
	<i>Anupam Kumar and Arun Rathore</i>	
13.1	Introduction	290
13.2	Structure Design of 2D Nanomaterial-Based Supercapacitors	297
13.3	2D Nanomaterials for Supercapacitor Technology	301
13.3.a	Transition Metal Oxides (TMOs) and Transition Metal Hydroxides (TMHs)-Based Supercapacitor	302
13.3.a.1	Transition Metal Oxides	302
13.3.a.2	Transition Metal Hydroxides	304
13.3.b	Transition Metal Carbide/Carbonitride (MXene)-Based Supercapacitor	304
13.3.c	Transition Metal Dichalcogenide (TMD)-Based Supercapacitor	305
13.3.d	Black Phosphorous-Based Supercapacitor	307
13.4	Conclusions	307
	References	308
14	2D Nanomaterials Based for Electrocatalytic Application	315
	<i>Anchit Modi, D. K. Gupta, Jitendra Malviya and N. K. Gaur</i>	
14.1	Introduction	316
14.1.1	Introduction to 2D Nanomaterials and Their Unique Properties	316
14.1.2	Motivation for Utilizing 2D Nanomaterials in Electrocatalytic Applications	317
14.2	Types of 2D Nanomaterials	318
14.2.1	Graphene	318
14.2.2	Dichalcogenides (TMDs)	318

14.2.3	Brief Overview of Their Structures and Properties	319
14.3	Electrocatalytic Reactions Enabled by 2D Nanomaterials	320
14.3.1	Oxygen Reduction Reaction (ORR)	320
14.3.2	Hydrogen Evolution Reaction (HER)	321
14.3.3	Carbon Dioxide Reduction Reaction (CO ₂ RR)	322
14.3.4	Synthesis and Characterization Techniques	323
14.3.4.1	Synthesis Methods for 2D Nanomaterials	323
14.3.4.2	Characterization Techniques for 2D Nanomaterials	324
14.3.4.3	Relationship Between Synthesis, Structure, and Electrocatalytic Performance	325
14.4	Challenges and Future Perspectives	325
14.4.1	Current Challenges in Utilizing 2D Nanomaterials for Electrocatalytic Applications	325
14.4.2	Potential Strategies to Overcome These Challenges	326
14.4.3	Future Directions and Emerging Trends in the Field	326
14.5	Conclusion	327
	References	329
15	Engineering 2D Nanomaterials for Biomedical Applications	333
	<i>Swaati Sharma, Hardeep Kaur, Mansi Thakur and Shinar Athwal</i>	
15.1	Introduction	334
15.2	Synthesis of Nanomaterials	337
15.3	Nanomaterials for Cancer Treatment	338
15.4	Difference of 2D Materials from Bulk Materials	339
15.4.1	Graphene	340
15.4.1.1	Synthesis of Graphene	341
15.4.1.2	Graphene Properties	343
15.4.1.3	Applications of Graphene	344
15.4.2	Hexagonal Boron Nitride (hBN)	345
15.4.2.1	Hexagonal Boron Nitride (hBN) Synthesis	347
15.4.2.2	Properties of Hexagonal Boron Nitride (hBN)	347
15.4.2.3	Applications of Hexagonal Boron Nitride (hBN)	348
15.4.3	Transition Metal Dichalcogenides (TMDs)	349

15.4.3.1	Synthesis of Transition Metal Dichalcogenides (TMDs)	349
15.4.3.2	Transition Metal Dichalcogenides (TMDs) and its Properties	350
15.4.3.3	Applications of Transition Metal Dichalcogenides (TMDs)	351
15.5	2DNMS for Next-Generation Quantum and Electronic Devices	352
15.6	Functionalized Hybridization of 2D Nanomaterials	352
	References	353
16	The Potential Applications of 2D Nanomaterials for Water Purification	361
	<i>Hardeep Kaur, Swaati Sharma, Shinar Athwal, Mansi Thakur, Meenakshi Verma and Vishal Mutreja</i>	
16.1	Introduction	362
16.2	Contaminants Present in Water	363
16.3	2D Nanomaterial-Based Water Purification Membranes	364
16.4	Solar Desalination Membrane	365
16.5	Filtration Membrane	367
16.6	Properties of Widely Used 2DM for Water Purification	369
16.6.1	MXene	369
16.6.2	g-C ₃ N ₄	372
16.6.3	Black Phosphorus	373
16.6.4	Graphene	374
16.6.5	h-BN	375
16.7	Synthesis of 2DM	376
16.7.1	Top-Down Approach	376
16.7.1.1	Liquid Exfoliation via Oxidation/Ion Intercalation/Mechanical Force	376
16.7.1.2	Mechanical Cleavage	378
16.7.1.3	Ion Exchange	379
16.7.1.4	Selective Etching	380
16.7.2	Bottom-Up Approach	380
16.7.2.1	Chemical Vapor Deposition	381
16.7.2.2	Wet-Chemical (WC) Synthesis	381
16.8	Adsorption of Contaminants From Water	382
16.8.1	Removal of Ions	382
16.8.2	Removal of Heavy Metals	387
16.9	Photocatalytic Purification of Water	387

16.10	Conclusion and Future Prospects	389
	References	389
17	Insights into the Exciton Dynamics of Functionalized 2D Nanomaterials for Robust Photoelectrochemical Sensing Applications	397
	<i>Dipyaman Mohanta, Koushik Barman, Abhinandan Mahanta, Bishal Bhuyan and Arpita Paul Chowdhury</i>	
17.1	Introduction	398
17.2	Basic Theory and Working Principle of Photoelectrochemical Sensing	400
17.3	Experimental Setup of Photoelectrochemical Cell	401
17.4	Importance of Photoactive Material in Photoelectrochemical Sensing	402
17.5	2D Nanomaterials in Photoelectrochemical Sensing	403
17.6	Current Challenges and Future Prospects	407
17.7	Conclusion	408
	References	409
18	Fabrication of 2D Nanomaterials-Based Biosensor	415
	<i>Arpita Paul Chowdhury, M. Dinamani and K. S. Anantharaju</i>	
18.1	Introduction	415
18.2	2D Nanomaterial Synthesis Strategies	417
18.3	Role of 2D Materials in Biosensor	418
	18.3.1 Electrochemical Biosensors	418
	18.3.2 Fluorescence Biosensors	421
	18.3.3 Colorimetric Biosensor	422
	18.3.4 Field-Effect Transistor Biosensor	424
	18.3.5 Surface-Enhanced Raman Spectroscopy (SERS)	425
18.4	Conclusions and Future Prospective	426
	References	426
19	Transition Metal Dichalcogenide (TMD)-Based 2D Nanomaterials for Various Kinds of Rechargeable Batteries	435
	<i>Periyakaruppan Karuppasamy and Varatharaj Rajapanian</i>	
19.1	Introduction	436
19.2	Synthesis of 2D-TMDCs	440
19.3	Applications of 2D-Transition Metal Di-Chalcogenides (2D-TMDCs) in Various Categories of Rechargeable Batteries	443

19.3.1	Li-Ion Batteries (LIBs)	446
19.3.2	Sodium-Ion Batteries	452
19.3.3	Potassium Ion Batteries (PIBs)	456
19.3.4	Magnesium Ion Batteries (MIBs)	457
19.3.5	Zinc Ion Batteries (ZIBs)	457
19.3.6	Lithium Sulfur Batteries (LSBs)	459
19.3.7	Aluminum Ion Batteries (AIBs)	460
19.4	Conclusion	461
	References	461
20	Effect of 2D Nanomaterial Addition to Performance and Emission Characteristics of Diesel Engine	475
	<i>Geetesh Goga and M. V. B. Unnamatla</i>	
20.1	Introduction	476
20.2	Performance Characteristics	477
20.3	Emission Characteristics	481
20.4	Conclusion	484
	References	485
Index		489

Preface

In recent years, 2D nanomaterials have emerged as a remarkable cornerstone in the field of advanced materials research, with their unique properties and versatile applications captivating the attention of scientists and engineers worldwide. This book is a testament to the ever-growing interest and importance of 2D nanomaterials in the realm of material science, nanotechnology, pharmaceuticals, and a myriad of engineering specializations. This book provides a comprehensive overview of the synthesis, modification, characterization, and application of 2D nanomaterials. Since the topic is vast, spanning a wide range of scientific disciplines and technological advancements, it is important to acknowledge that no single book can encompass the entirety of this field. However, this diverse collection of chapters covers essential themes and materials, focusing particularly on the synthesis strategies and potential applications of 2D nanomaterials.

The book is structured into three sections, each delving into different aspects of 2D nanomaterials. The first section explores the synthesis of these materials, providing an overview of both top-down and bottom-up strategies. Understanding the methods by which these materials can be synthesized is crucial for advancing their potential applications. Additionally, this section delves into the structural characterization of 2D nanomaterials, shedding light on their intricate compositions and properties.

The second section examines the diverse characteristics exhibited by 2D nanomaterials. From their magnetic and mechanical properties to their electrical, plasmonic, and optical behaviors, these materials possess an array of intriguing attributes that make them highly attractive for a wide range of applications. This section of the book provides a comprehensive understanding of these properties, enabling readers to appreciate the unique potential of 2D nanomaterials.

The final section focuses on the applications of 2D nanomaterials, highlighting their use in various fields such as energy, water purification, biomedical applications, multimodal tumor therapy, and supercapacitor technology. By showcasing the breadth of their applications, we hope to

inspire readers to explore further and unlock the immense possibilities that lie within these materials.

The editors would like to express sincere gratitude to all the authors and co-authors who contributed their exceptional research to this book. Their expertise and dedication have enriched its content, ensuring its relevance and significance. Furthermore, we extend our heartfelt appreciation to the Wiley-Scrivener publishing team for their unwavering support throughout the challenging process of bringing this book to fruition. Their professionalism and commitment have been invaluable in making this project a reality.

As editors, we firmly believe that this book will serve as a valuable resource for students, researchers, and professionals in material science and related fields. It will spark new ideas, ignite curiosity, and pave the way for groundbreaking discoveries in the realm of 2D nanomaterials. We are confident that this book will find its rightful place in university and institute libraries across the globe and will be a source of inspiration for future research and innovation.

Once again, we extend our deepest gratitude to all who have contributed to this book. We are honored to present this collection of knowledge and discoveries, and we eagerly anticipate witnessing the impact it will have on the ever-evolving landscape of 2D nanomaterial research.

Subhendu Chakroborty

Ph.D.; MRSC

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January 2024

Part I

SYNTHESIS OF 2D NANOMATERIALS

Top-Down Strategies Synthesis of 2D Nanomaterial

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Abstract

The nanotechnology field originated in the 21st century. Especially 2D-based nanomaterials have received a lot of attention since they are easily affordable, non-toxic, and have excellent electrical, optical, thermal, and mechanical properties. Moreover, it is simple to synthesize and can be applied to a wide range of applications. The present chapter mainly focuses on various types of synthesis methods for 2D nanomaterials using top-down strategies. Many top-down strategies have been developed to synthesize 2D nanomaterials, such as etching, mechanical milling, sputtering, and laser ablation. In all methods, we would introduce their synthesis parameters, advantages, disadvantages, and applications. Moreover, its characterization and toxicity were briefly introduced.

Keywords: Nanomaterials, synthesis, 2D nanomaterials, top-down strategy, application of nanomaterials

1.1 Introduction

Nowadays, nanomaterials (NMs) are diverging materials among all research fields [1–4]. Nanomaterials are tiny-sized materials with external diameters up to 100 nm. The nanomaterials are primarily used to make the tubes, rods, and fibers. In addition, the nanoparticles found their physical existence in nature [5]. Nanomaterials possess different physical properties

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as well as chemical properties to form the bulk of their counterparts. As the size of the nanomaterials is too tiny, they cannot be seen with the naked eye. These nanomaterials are added to different materials, such as cloth, cement, and other materials. The tiny size of these materials also makes them useful in electronics, environmental remediation, and neutralizing toxins. The emergent properties of nanomaterials make them beneficial and impart great impacts in electronics, medicine, and other fields [6–8]. The chemical and physical properties of NMs highly depend on the surface atoms. As per the applications, NM size can be controlled by various techniques, such as modification of surface and micelle concentration [9, 10]. Dimensionally, NMs are divided into zero-dimensional (0D), one-dimensional (1D), two-dimensional (2D), and three dimensional (3D). In 0D, all three dimensions merge into a nanoscale range. A schematic diagram of dimension-based NMs is provided in Figure 1.1. In this category, nanospheres, quantum dots, and nanoclusters are included. In 1D, two dimensions merge into one. In this category, nanotubes and nanorods are included. In the 2D category, one dimension is at the nanoscale and the other dimension is outside [11]. In this category, nanofilms and nanolayers are included. 3D NMs are bulk NMs with diameters greater than the nanoscale (1–100 nm). The building blocks for 3D NMs are 0D, 1D, and 2D NMs. Core shells, nanowire bundles, nanotube bundles, and multi-nanolayers are included in this category [12].

Among these, more and more attention has been paid to two-dimensional (2D) NMs due to their unique properties, such as excellent

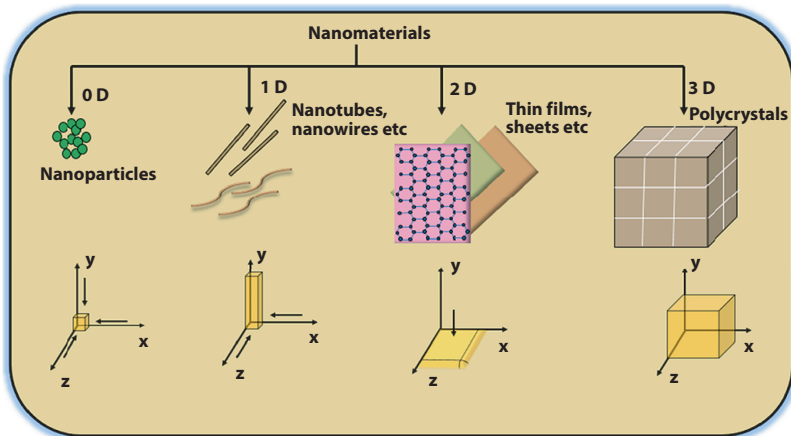


Figure 1.1 NM classification based on dimensions [13].

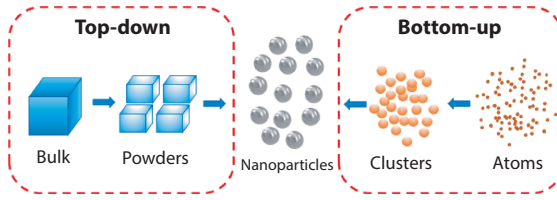


Figure 1.2 Nanoparticle's production strategies [23].

electrical, optical, thermal, and mechanical properties [14–17]. Many strategies have been developed to synthesize it for a specific application. These synthesis methods are broadly divided into two types of strategies: top-down and bottom-up approaches. Figure 1.2 displays an illustration of NM top-down and bottom-up strategies. The distinction between these two general classifications is based on the processes involved in the creation of nanometer-sized structures, and the choice of method depends on the specific requirements of the desired end product and the available techniques and technologies [18]. In the bottom-up approach, nanoscale materials are constructed from atomic or molecular precursors that are allowed to react, grow in size, or self-assemble into more complex structures [19]. By contrast, the top-down approach carves nanoscale structures by controlling the removal of materials from larger or bulk solids [20]. Each strategy has its own advantages and disadvantages [21]. In bottom-up strategies, nanoshells, ultrafine nanoparticles, and even nanotubes can be produced with a size of 1–20 nm. However, massive production is not possible, and synthesized nanomaterials need chemical purification. In a top-down strategy, nanomaterials can be produced massively, and purification is not needed. However, with this strategy, it is difficult to optimize the synthesized parameters [22]. This strategy is also not cost-effective.

In this chapter, we mainly emphasize the synthesis of 2D nanomaterials using a top-down strategy. In this strategy, we introduce the synthesis parameters, advantages, disadvantages, and applications for all methods. Eventually, the characterization and toxicity of 2D materials will be proposed.

1.2 Top-Down Strategy Synthesis Method

In a top-down strategy, bulk material is first converted into powder-based materials, which are then converted into nanomaterials [20]. There are mainly four methods available in this strategy (Figure 1.3), such as etching,

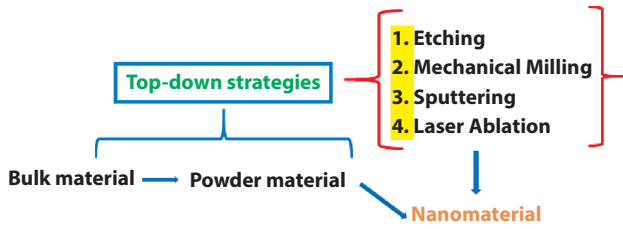


Figure 1.3 Top-down synthesis method of 2D nanomaterials.

mechanical milling, sputtering, and laser ablation. Each method’s advantages, disadvantages, and own perspectives on applications are critically discussed. A summary of 2D NMs is listed in Table 1.1 using different top-down strategy methods.

Table 1.1 Synthesis of 2D NMs by different methods of top-down strategy.

NMs	Method	Parameters	Size	Applications	Ref.
GO	Mechanical milling	Ball size 5 nm; rotation speed 600 rpm, time (6, 16, and 24 h)	5 nm	Dye removal	[33]
GO	Mechanical milling	Ball size 3 mm, rotation speed 200 rpm, time 12 h	40 nm	Drug delivery	[34]
MoO ₃	Laser ablation	Argon emission lines, Oxygen supplied	NA	Cancer therapy	[42]
GO-nano ribbon	Laser ablation	NA	NA	Optical	[43]
WS ₂	Sputtering	Pulsing frequency of 20 kHz, DC power supply	>100	NA	[44]
MoS ₂	Sputtering	Working distance 6 mm, a base pressure 2×10^{-6} Torr prior, 60 W	721	Supercapacitor	[45]

(Continued)

Table 1.1 Synthesis of 2D NMs by different methods of top-down strategy.
(Continued)

NMs	Method	Parameters	Size	Applications	Ref.
MoO ₂	Electro explosion	Surfactant, Thickness, temperatures, and current density	18.2	Antibacterial activity	[46]
MoS ₂	Etching	Cl ₂ plasma and Ar plasma	NA	Nanodevices	[47]
Hexagonal BN	Etching	O ₂ and N ₂ plasma (10 W); pressure: 1 Torr	NA	NA	[48]

1.2.1 Etching

In this method, the 2D NM surface is modified for enhanced physical and chemical properties. Mainly, this method of obtaining materials is applicable to the semiconductor industry. In this approach, a bulk material is treated with an etchant that selectively removes or dissolves certain layers or regions, leaving behind thin layers or flakes with desired 2D NM properties. This method has been used to synthesize 2D NMs such as graphene oxide (GO), transition metal dichalcogenides (TMDs), and boron nitride by selectively oxidizing or etching away layers from their respective bulk materials [24]. In this method, it is necessary to understand growth mechanisms [25]. Etching can be used to create patterns, structures, or features on the nanoscale, and it is a crucial step in the fabrication of various nanodevices and nanosystems [26]. Depending on the material and the desired outcome, several etching techniques can be used for 2D nanomaterials. Here are some commonly used etching techniques for 2D nanomaterials:

Wet etching: Wet etching involves the use of liquid chemicals to dissolve or remove material from a substrate selectively. For 2D nanomaterials, wet etching can be performed by immersing the substrate containing the nanomaterial in a chemical solution that selectively reacts with the material to be etched while leaving other parts of the substrate untouched [27]. Wet etching is relatively simple and can be used for a wide range of 2D NMs, including graphene, transition metal dichalcogenides (TMDs), MoS₂, and WS₂.

Dry etching: Dry etching, also known as plasma etching, involves using reactive gases and plasma to remove material from a substrate. Dry etching

can be used for 2D NMs by exposing the substrate to a reactive gas, typically in a plasma chamber, which reacts with the material being etched and removed [28]. Dry etching offers higher precision and control over the etching process than wet etching, but it may require more complex equipment and processing conditions. Standard dry etching techniques for 2D NMs include reactive ion etching (RIE) and plasma etching.

Chemical vapor etching: Chemical vapor etching involves using reactive gases that are deposited onto the substrate as a vapor, which then reacts with the material to be etched. Chemical vapor etching can be used for 2D NMs by exposing the substrate to the reactive gases in a controlled environment, such as a vacuum chamber, and allowing the gases to react with the material to be etched [29]. Chemical vapor etching offers high control over the etching process and can be used for selective and precise material removal from 2D nanomaterials.

Atomic layer etching: atomic layer etching (ALE) is a specialized technique that offers precise control over the etching process at the atomic scale. ALE involves using sequential, self-limiting reactions to remove material from a substrate, layer by layer, selectively [30]. ALE can be used for 2D nanomaterials by controlling the exposure of the substrate to the reactive gases in a cyclic manner, which allows for highly controlled etching with atomic-level precision.

It is important to note that the choice of etching technique depends on the specific material properties of the 2D nanomaterial, the desired outcome, and the equipment and facilities available for the fabrication process. Careful consideration of the material properties, etching parameters, and safety precautions is necessary to ensure the successful etching of 2D NMs.

1.2.2 Mechanical Milling

Mechanical milling is the simplest top-down strategy. Mechanical milling, also known as mechanical alloying or ball milling, is a technique used to synthesize and process materials at the nanoscale through mechanical means. In this method, NMs are produced by the collision of rigid balls and bulk materials [31]. The whole system works under high pressure and is conducted through a sealed container. Usually, containers are made of ceramic and steel-based materials. The final product size depends upon many factors, such as ball size, milling time, milling duration, rotating speed, amount of bulky raw materials, and milling environment. The milling process is more effective when the ball-to-powder ratio is higher [32]. The milling process have many more advantages such as easy to handle, safety, high capacity,

and long term maintained, even sometime no need solvent [32]. Despite this, disadvantages include disordering the crystal structure, requiring a lot of energy, the chance of contamination, and noise. It has been widely used to fabricate 2D nanomaterials, which exhibit unique properties due to their thickness being limited to the nanometer scale. The process of mechanical milling for 2D nanomaterials typically involves the following steps:

Material selection: Suitable precursor materials, typically in the form of powders, are chosen based on the desired properties of the final 2D nanomaterial.

Ball milling: The precursor materials are loaded into a ball mill and milling balls are usually made of a hard material such as stainless steel or tungsten carbide. The ball mill is then rotated at high speeds, causing the milling balls to collide with the precursor materials, leading to mechanical deformation, grinding, and mixing.

Milling parameter optimization: Various milling parameters, such as milling time, milling speed, and ball-to-powder ratio, are optimized to control the size, shape, and properties of the resulting 2D nanomaterials. These parameters can be adjusted to achieve the desired nanoscale features and properties of the 2D nanomaterial.

Post-milling treatment: Additional post-milling treatments such as annealing, doping, or functionalization may be employed to tailor the properties of the 2D nanomaterials further.

In this method, various types of 2D nanomaterials are synthesized and listed in Table 1.1 with different parameters. Recently, Mahmoud and his co-workers synthesized graphene oxide (GO) MPs through the ball milling method [33]. The obtained NM size was 5 nm. Authors set different parameters such as rotating speed (600 rpm), milling time, ball size, etc. Further authors characterize it using various sophisticated techniques such as BET, UV, XRD, TEM, and Raman. Moreover, synthesized materials are applicable for methylene blue dye removal. Further, Caicedo *et al.* fabricated GO from graphite using this method. The obtained GO NM size was 40 nm in a 12 h processing time and is applicable in drug delivery systems. From the graphite precursor, GO was produced [34]. The main mechanism is the oxidation process.

1.2.3 Sputtering

Sputtering is a well-known synthesis method in the category of top-down strategies. Sputtering is a versatile and widely used technique for synthesizing 2D nanomaterials due to its ability to produce thin films with precise control over their thickness, composition, and properties [35]. It has been

employed to synthesize various types of 2D NMs and 2D layered materials, with applications in areas such as electronics, optoelectronics, energy storage, and sensors [36]. Generally, in this method, thin films are fabricated on a substrate through bombardment of the target with energetic ions [37]. In practice, there are several types of sputtering, such as DC diodes, RF-diodes, magnetron diodes, and ion beam sputtering. In this method, thin films are commonly produced on the substrate. For the production films, an ultra-high vacuum system was needed [38]. The sputtering process for the synthesis of 2D nanomaterials typically involves the following steps:

Target preparation: A solid target material, which is the source of the atoms or ions to be deposited, is selected and prepared. The target material can be a pure element or a compound, depending on the desired composition of the 2D nanomaterial.

Substrate preparation: A substrate, which is a flat surface onto which the atoms or ions will be deposited to form the 2D nanomaterial, is prepared. The substrate can be made of various materials, such as silicon, glass, or a flexible polymer, depending on the intended application of the 2D nanomaterial.

Sputtering process: The target material is bombarded with high-energy ions, which dislodge atoms or ions from the target surface. These atoms or ions are then deposited onto the substrate surface, where they can form a thin film with a thickness in the nanometer range. The substrate is typically placed in close proximity to the target material, and the sputtering process is carried out in a vacuum chamber to minimize contamination and promote uniform deposition.

Control of deposition parameters: Various parameters, such as the sputtering power, gas pressure, and deposition time, can be adjusted to control the thickness, composition, and morphology of the deposited 2D nanomaterial. These parameters can be optimized to achieve the desired properties of the final nanomaterial.

Post-deposition treatments: After the deposition, the 2D nanomaterial may undergo additional treatments, such as annealing or etching, to further modify its properties, such as crystal structure, electrical conductivity, or surface morphology.

1.3 Laser Ablation

The laser concept was first introduced by Einstein. Laser ablation is a straightforward process. Using this process, lasers are the main source for