

<<纳米科学与技术大全5>>

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## <<纳米科学与技术大全5>>

### 内容概要

纳米科学与技术大全5：自组装与纳米化学（导读版）作者将生态学不同领域的理论和实验进展与新方法相结合，在个体层次与群落结构、生态系统功能间，微观世界与宏观生态间的融合做了很好的尝试，为我们理解生态现象、生态过程以及生态功能展示了一个很好的途径。

纳米科学与技术大全5：自组装与纳米化学（导读版）可作为研究生生态课程的参考书，对于从事生态学教学和研究的教师和科研人员及生态学者有非常高的参考价值。

<<纳米科学与技术大全5>>

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## 书籍目录

5.01 多孔金属有机骨架5.01.1 Introduction5.01.2 Inorganic SBUs and Organic Linkers5.01.3 Architecture of the Networks5.01.4 Porous Structures5.01.4.1 0D Cage5.01.4.2 1D Channels5.01.4.3 2D Layers5.01.4.4 3D Channels5.01.5 Synthesis of MOFs5.01.5.1 Influencing Factors5.01.5.2 Solvent-Evaporation Synthesis5.01.5.3 Diffusion Synthesis5.01.5.4 Hydrothermal (or Solvothermal) Synthesis5.01.5.5 Microwave-Reaction Synthesis5.01.5.6 Ionothermal Synthesis5.01.5.7 Electrochemical Synthesis5.01.5.8 High-Throughput Synthesis5.01.6 Functions of MOFs5.01.6.1 Gas Storage5.01.6.1.1 Hydrogen Storage5.01.6.1.2 Methane Storage5.01.6.1.3 Carbon Dioxide Storage5.01.6.2 Selective Gas Adsorptions and Separations5.01.6.3 Catalysis5.01.6.4 Magnetism5.01.6.5 Optics5.01.6.6 Sensor5.01.6.7 Drug Delivery5.01.7 Summary and OutlookReferences5.02 纳米粒子配体5.02.1 Introduction5.02.2 Ligands, Chief Cook, and Bottle Washer5.02.2.1 Ligands Control the Synthesis of NPs5.02.2.2 A Brief Introduction to Classical Nucleation Theory5.02.2.3 Ligands Stabilize NP Suspensions5.02.2.4 Ligands and the Shape of NPs5.02.2.5 Ligands Give NPs Physicochemical Functionality5.02.3 What to Expect, Ab Initio Calculations5.02.4 Experimental Observation of NP Ligands5.02.4.1 Indirect Probing of Ligand Exchange5.02.4.2 Direct Probing of Ligands5.02.5 Observing NP Ligands with Solution NMR Spectroscopy5.02.5.1 Solution NMR Techniques for Observing QD Ligands5.02.5.1.1 A brief introduction in solution NMR spectroscopy5.02.5.1.2 Pulsed field gradient NMR spectroscopy5.02.5.1.3 Nuclear Overhauser effect NMR spectroscopy5.02.5.2 The Tightly Bound Ligand5.02.5.2.1 What to expect?5.02.5.2.2 The basic experiment:1D <sup>1</sup>H NMR5.02.5.2.3 Tracing down the ligand resonances by diffusion NMR5.02.5.2.4 Identifying ligands, proton-carbon correlations5.02.5.2.5 A note on relaxation rates and peak broadening5.02.5.3 Adsorption-Desorption Equilibria, <sup>1</sup>H NMR as a Quantitative Technique5.02.5.3.1 Quantitative NMR5.02.5.3.2 Observing adsorption-desorption equilibria by NMR5.02.5.3.3 Understanding the adsorption isotherm5.02.5.4 Adsorption-Desorption Kinetics, Exploiting the NOE5.02.5.4.1 Dodecylamine stabilized Q-CdTe, does the tightly bound ligand model work?5.02.5.4.2 Observed NMR resonances, a story of timescales5.02.5.4.3 Tightly bound ligands have strongly negative NOEs5.02.5.4.4 Rapidly exchanging ligands show strongly negative transfer NoEs5.02.5.5 In Situ Monitoring of NP SynthesisReferences5.03 纳米粒子组装5.03.1 Introduction5.03.2 Assembly Methods for 1D NPs5.03.2.1 Assembly of NPs for Nanorod and Nanowire Formation5.03.2.2 Assembly of 1D NPs on Polymer Templates5.03.3 Assembly of NPs to Form 2D Nanocomposites5.03.4 Biomolecules as Templates for Assembling NPs in 1D and 2D Architectures5.03.5 Modulation of the Properties of 1D and 2D Structures5.03.5.1 Optical Response5.03.5.2 Electronic Behavior5.03.5.3 Magnetic Properties5.03.6 Summary and OutlookReferences5.04 周期的介孔材料:充满机遇的孔道5.04.1 Introduction5.04.2 Hierarchical Organization of Mesoporous Materials5.04.2.1 Self-Assembly of Sol-Gel Precursors and Templates-From Micro to Meso5.04.2.2 Growing Complexity: Powder, Films, and the Importance of Form5.04.3 Bringing Function into Voids5.04.3.1 Grafting5.04.3.2 Co-Condensation5.04.3.3 Periodic Mesoporous Organosilicates5.04.4 Nonsiliceous Mesoporous Materials5.04.4.1 Mesoporous Metal Oxides and Phosphates5.04.4.1.1 Synthesis strategies and objectives5.04.4.1.2 Realized compositions5.04.4.1.3 Perspectives I: Toward crystallized mesoporous oxides5.04.4.1.4 Perspectives II: Form and function5.04.4.2 Mesoporous Metals and Semiconductors5.04.4.2.1 Mesoporous semiconductors5.04.4.2.2 Mesoporous metals5.04.4.3 Mesoporous Carbon5.04.4.3.1 OMCs obtained by hard templating5.04.4.3.2 OMCs obtained by soft templating5.04.4.4 Mesoporous Ceramic Materials5.04.4.4.1 Silicon-based mesoporous ceramics5.04.4.4.2 Mesoporous carbon and boron-based ceramics5.04.5 Mesoscience to Mesotechnology-Why Meso?5.04.5.1 Sorbents and Separation Science5.04.5.2 Catalysis5.04.5.3 Drug Delivery5.04.5.4 Sensing5.04.5.5 Low-k Materials5.04.5.6 Photovoltaics5.04.6 Conclusion and OutlookReferences5.05 单层自组装5.05.1 Molecular Self-Assembly and Nanoscience5.05.2 Driving Forces for Molecular Assembly: Molecular Interactions in Self-Assembled Monolayers5.05.3 Overview of Previous Studies of Molecular Self-Assembled Monolayers5.05.4 Brief Summary of Synthetic Methods of 2D Self-Assembled Monolayers and the Main Techniques to Study them5.05.5 Molecular Self-Assembly on Au(111)5.05.5.1 CH<sub>3</sub>(CH<sub>2</sub>)<sub>n</sub>SH5.05.5.2 CH<sub>3</sub>(CH<sub>2</sub>)<sub>n</sub>CS<sub>2</sub>H<sub>5</sub>5.05.5.3 C<sub>6</sub>H<sub>5</sub>(CH<sub>2</sub>)<sub>n</sub>SH5.05.5.4

CH<sub>3</sub>-(C<sub>6</sub>H<sub>4</sub>)<sub>2</sub>-(CH<sub>2</sub>)<sub>n</sub>-SH5.05.5.5 CF<sub>3</sub>(CH<sub>2</sub>)<sub>n</sub>SH5.05.5.6 Diamidothiols5.05.6 Organic Monolayers on Ag(111)5.05.7 Self-assembly of Organic Molecules on Cu,Al,Hg,Al<sub>2</sub>O<sub>3</sub>,and SiO<sub>x</sub>/Si Substrates5.05.8 Molecular Self-Assembly on Highly Oriented Pyrolytic Graphite5.05.8.1 Single-Component Long-Chain Molecules:Linear Packing and Molecular Distortion5.05.8.1.1 Molecular parallel packing5.05.8.1.2 Molecular distortion5.05.8.2 Multicomponent Self-Assembly and Formation of Nanostructures5.05.8.3 Molecular Chirality upon Self-Assembly5.05.9 SummaryReferences5.06 纳米晶体合成5.06.1 Introduction5.06.1.1 Milestones of Progress in Nanocrystal Synthesis5.06.1.2 Synthetic Methods5.06.1.2.1 High-temperature organo-metallic method5.06.1.2.2 Single-source molecular precursor method5.06.1.2.3 Solvothermal/hydrothermal method5.06.1.2.4 Water-phase synthesis5.06.1.2.5 Template-assisted growth methods5.06.1.2.6 Synthesis of semiconductor nanocrystals in microfluidic reactors5.06.2 Size Tuneability of Nanocrystals5.06.2.1 Introduction5.06.2.2 Mechanisms of Size Control5.06.2.2.1 Nucleation and growth of nanocrystal5.06.2.2.2 Concepts in size control5.06.3 Shape,Phase,and Composition Control of Nanocrystals5.06.3.1 Shape Control of Nanocrystals5.06.3.1.1 Dynamic-induced anisotropic growth5.06.3.1.2 Seed-mediated growth5.06.3.1.3 The Oriented attached method5.06.3.2 Composition Control5.06.4 Overview of the Nanocrystal Synthesis by Material5.06.4.1 II-VI Semiconductor Nanocrystals5.06.4.2 III-V Semiconductor Nanocrystals5.06.4.3 IV-VI Semiconductor Nanocrystals5.06.4.4 IV Semiconductor Nanocrystals5.06.4.5 III-VI and I-III-V Nanocrystals5.06.4.6 Metal Oxides5.06.4.6.1 Sol-gel method5.06.4.6.2 Nonhydrolytic route5.06.5 New-Generation Semiconductor Nanocrystals5.06.5.1 Nanocrystal Heterostructures5.06.5.1.1 Synthetic techniques for the preparation of nanocrystal heterostructures5.06.5.1.2 Synthesis of 0D core-shell Nanocrystal heterostructures5.06.5.1.3 Synthesis of anisotropic and more complex nanocrystal heterostructures5.06.5.2 Doped Nanocrystals5.06.5.2.1 Synthesis of doped nanocrystals5.06.6 SummaryReferences5.07 纳米粒子自组装基元5.07.1 Introduction5.07.1.1 Self-Assembly Principle5.07.1.2 NBB Classification5.07.2 NBB Self-Assembly Approaches5.07.2.1 Self-Assembly on a Substrate5.07.2.2 Interfacial Assembly5.07.2.3 Template-Assisted Assembly5.07.3 Self-Assembly of Complex-Shaped NBBs:Tetrapods5.07.4 Computational Approach to Nanoparticle Self-Assembly5.07.4.1 Computational Framework for Nanoparticle Self-Assembly5.07.4.2 Computational Studies on the Self-Assembly of NBBs on a Substrate5.07.4.3 Computational Studies on the Interfacial Assembly of NBBs5.07.4.4 Computational Studies on NBB Self-Assembly on a Templated Surface5.07.4.5 A Proposed Approach for Modeling Tetrapod Self-Assembly5.07.5 SummaryReferences5.08 组装嵌段共聚物的化学过程5.08.1 Introduction5.08.2 Work Prior to 1992 on Chemical Processing of Self-Assembled Block Copolymers5.08.3 Our Research Program and Activities5.08.4 Architectures from Chemically Processing Assembled Block Copolymers5.08.4.1 Cyclic Polymers5.08.4.2 Thin Films Containing Nanochannels5.08.4.3 Cell-Like Microspheres5.08.5 Block Copolymer Nanofibers and Nanotubes5.08.5.1 Nanofiber Preparation5.08.5.2 Nanotube Preparation5.08.5.3 Dilute Solution Properties5.08.5.4 Chemical Reactions5.08.5.4.1 Backbone modification5.08.5.4.2 Surface grafting5.08.5.4.3 End functionalization5.08.6 Concluding RemarksReferences5.09 生物模版制备半导体纳米晶体5.09.1 Introduction5.09.2 Living Cells as Semiconductor Nanocrystal Factories5.09.3 Peptides and Proteins as Templates for Semiconductor-Based Nanomaterials5.09.4 Nucleic Acids as Templates for Semiconductor-Based Nanomaterials5.09.4.1 Monomeric Nucleotides as Semiconductor Nanocrystal Ligands:Roles of Base and Backbone5.09.4.2 Oligomeric Nucleotides as Semiconductor Nanocrystal Ligands:Roles of Length and Sequence5.09.4.3 Studies of Nucleic Acids with 3D Structure as Semiconductor Nanocrystal Ligands:Control of Nanomaterials Properties with Biomolecular Structure5.09.4.4 One-Step Synthesis of Biofunctionalized Semiconductor Nanocrystals Using Nucleic Acids Ligands5.09.5 Summary and OutlookReferences5.10 高分子层状硅酸盐纳米复合物5.10.1 Introduction and Historical Perspective5.10.2 Basic Structures of Layered Silicates and Polymers5.10.2.1 Layered Silicate Structure5.10.2.2 PLSN Structure: Degree of Silicate Layer Dispersion5.10.2.3 Polymers Used in PLSNs5.10.3 Synthetic Methods5.10.3.1 In Situ Polymerization5.10.3.2 Solution Intercalation/Exfoliation5.10.3.3 Melt Processing5.10.4 Characterization and Properties of PLSN Structures5.10.4.1 Structure of Modified Silicates5.10.4.1.1 XRD and TEM5.10.4.2 Thermal and Mechanical Properties of PLSNs5.10.4.2.1 Mechanical properties5.10.4.2.2 Thermal and flame-retardant properties5.10.4.3

Other Properties5.10.4.3.1 Gas-barrier properties5.10.4.3.2 Electrical properties5.10.4.3.3 Compatibilization of polymer blends5.10.5 ConclusionsReferences5.11 介晶和介相5.11.1 Introduction5.11.1.1 Classification of Thermotropic Mesophases5.11.1.2 Classification of Mesogens5.11.1.3 Self-Assembly of Mesogens to Mesophases5.11.1.4 Alignment,Self-Healing,and Fixation5.11.1.5 Length Scales5.11.2 Thermotropic Mesophases5.11.2.1 Carbon Allotropes-from Conventional Mesogens based on Polycondensed Aromatics to Hybrid Systems of Carbon Nanoparticles5.11.2.1.1 Molecular structure of graphenes,synthetic strategies and interaction motifs5.11.2.1.2 The supramolecular self-assembling of discotic or sanidic mesogens5.11.2.1.3 Applications of graphene LCs5.11.2.1.4 Macrocycles5.11.2.1.5 CNTs-mesogens from enrolled graphenes5.11.2.1.6 LCs and fullerenes5.11.2.1.7 Miscellaneous carbon mesogens5.11.2.2 Supramolecular Mesogens5.11.2.2.1 Hydrogen-bonded systems5.11.2.2.2 Mesogens formed by halogen bonds5.11.2.2.3 Metallomesogens5.11.2.2.4 Ionic liquid crystals5.11.2.2.5 Donor?acceptor interactions,charge transfer,and polytopic interactions5.11.2.3 Bolaamphiphiles and Facial Amphiphiles-Nanostructured Mesophases by Multicolor Tiling5.11.2.4 Star-Shaped Mesogens5.11.2.5 Dendrons and Dendrimers5.11.2.5.1 Supramolecular dendromesogens5.11.2.5.2 Side-chain liquid-crystalline dendrimers5.11.2.5.3 Main-chain liquid-crystalline dendrimers5.11.3 Lyotropic Mesophases5.11.3.1 Lyotropic Phases-Templates for the Synthesis of Nanomaterials5.11.3.2 Cubosomes,Hexosomes,Lamellarsomes:Nanostructured Reverse Phases Stable in Excess Solvent5.11.3.3 From Mineral LCs to LCs of Nanobiomolecules5.11.3.3.1 Introduction5.11.3.3.2 Colloidal suspensions: The Derjaguin?Landau?Verwey?Overbeek theory,steric stabilization,and Onsager theory5.11.3.3.3 Mineral LCs5.11.3.3.4 Applications5.11.3.3.5 From nanobiomolecules toward viruses5.11.4 Nanoparticles and LCs5.11.4.1 Synthesis of Nanoparticles from LC phases5.11.4.2 LC Phases from Nanoparticles5.11.4.3 Nanoparticle Doped LCsReferences5.12 层层自组装胶囊在生物医药的应用5.12.1 Introduction5.12.2 LbL Assembly:Background5.12.3 Engineering the Capsule Layers5.12.3.1 pH-Responsive Capsules5.12.3.2 Redox-Responsive Capsules5.12.3.3 Light-Responsive Capsules5.12.3.4 Temperature-Responsive Capsules5.12.3.5 Enzyme-Responsive Capsules5.12.3.6 Chemically Responsive Capsules5.12.3.7 Other Stimuli-Responsive Capsules5.12.4 Engineering the Capsule Surface5.12.5 Encapsulating Cargo5.12.6 Applications of LbL Capsules5.12.6.1 Glucose-Responsive Systems-Delivery and Sensing5.12.6.2 LbL Drug Delivery Systems5.12.6.3 Bioreactors5.12.7 ConclusionReferences5.13 功能化石墨烯:合成和性能5.13.1 Introduction5.13.2 Chemical Functionalization of Fullerenes5.13.2.1 Nucleophilic Additions to Fullerenes5.13.2.2 Nucleophilic Addition?Elimination Mechanism:The Bingel-Hirsch Reaction with Fullerenes5.13.2.3 Cycloaddition Reactions to Fullerenes5.13.2.4 1,3-Dipolar Cycloaddition of Azomethine Ylides to Fullerenes5.13.2.5 Diels-Alder Cycloaddition Reactions with Fullerenes5.13.3 Molecular Machines5.13.4 Molecular Charge-Transfer Conjugates5.13.5 Molecular Wires5.13.6 Conclusion and OutlookReferences5.14 微乳制备法(综述)5.14.1 Introduction5.14.1.1 Basic Concepts:Micelles and Microemulsions5.14.1.2 Properties and Applications5.14.2 Synthesis of Nanoparticles(NPs)Microemulsion5.14.2.1 Synthesis of Metal and Mixed-Metal NPs5.14.2.2 Synthesis of Semiconductor NPs5.14.2.3 Synthesis of Magnetic NPs5.14.2.4 Synthesis of Oxide NPs in Microemulsion5.14.3 Synthesis of Rare Earth(RE)Nanocrystals(NCs)in Microemulsion5.14.3.1 Synthesis of Regular RE NCs with Well-Defined Facets5.14.3.2 Synthesis of 1D RE NCs5.14.3.3 Synthesis of RE Super Nanostructures5.14.4 Silica Coating of NPs5.14.4.1 Hydrophilic Metallic NPs5.14.4.2 Hydrophilic Semiconductor NPs5.14.4.3 Hydrophilic Magnetic NPs5.14.4.4 Miscellaneous Hydrophilic NPs5.14.4.5 Hydrophobic Metallic NPs5.14.4.6 Hydrophobic Magnetic NPs5.14.5 Direct Coating of Hydrophobic Semiconductor QDs5.14.5.1 Hydrophobic Multifunctional NCs5.14.5.2 Silica Coating of RE NCs5.14.6 Conclusions and OutlookReferences5.15 纳米技术、社会和环境5.15.1 Introduction5.15.2 Twenty-first Century Relationships between Science,Technology,Society,and the Environment5.15.3 Economy5.15.3.1 Technoscience and Business5.15.3.2 Nanoproducts and Society5.15.3.3 Patenting Nanoproducts5.15.3.4 Military Applications5.15.4 Ecology5.15.4.1 Nanotechnology and the Environment5.15.4.2 Nature,Technology,and Public Discourse5.15.4.3 NGOs and Local Communities5.15.5 Health5.15.5.1 Nanotechnology and Health5.15.5.2 Nanotoxicity5.15.6 Equity5.15.6.1 Global Equity and Rights:Implications for Developing Countries5.15.6.2

<<纳米科学与技术大全5>>

Power5.15.6.3 Identity5.15.6.4 Gender5.15.6.5 Privacy5.15.7 Governance5.15.7.1 Science and Technology  
Policy:Funding Nanotechnology Research and Development5.15.7.2 Nanotechnology Regulatory  
Capacity5.15.7.3 Public Attitudes and Media Coverage5.15.7.4 Nanotechnology Public Engagement and  
Democracy5.15.8 Imagined Futures5.15.8.1 Fact and Fiction:Social and Cultural Influences5.15.8.2 The  
Construction of Utopias and Dystopias5.15.8.3 Scenario Planning5.15.9 Conclusion: Nature and  
NanotechnologyReferences

## 章节摘录

版权页：插图：5.02.4.2 Direct Probing of Ligands To address the NP ligands more directly, two techniques have been recently applied in the literature: synchrotron X-ray photoelectron spectroscopy (XPS) and solution NMR [34,35]. With XPS, the various elements present in the nanocrystal core and on the surface can be probed and information on their local environment is obtained. For instance, in the case of PbS, Lobo and coworkers observed a set of S- and Pb emission peaks which they could attribute to various surface species, either unpassivated, linked to a specific ligand, or oxidized [34]. A closely related technique that has been used to identify NP ligands is Rutherford backscattering spectroscopy (RBS) [34,35]. This experiment does not provide information on the exact location of the atoms of interest (in the core or on the surface) but nevertheless, the presence of, for instance, phosphorous atoms in the sample can be taken as evidence for TOP ligands. Both XPS and RBS are ex situ analysis techniques, carried out on nanocrystal solids. This means that traces of unreacted precursors or free ligands might lead to spurious results, since they can be hardly distinguished from bound ligands. Therefore, a careful assessment of the sample purity is crucial in these measurements. In addition, little information on the ligands is obtained, and a study of their dynamical behavior in solution is not possible. The drawback of ex situ analysis can be overcome by solution NMR. This has been applied to the study of NP suspensions for more than 15 years, yet it has mainly been limited to one-dimensional (1D)  $^1\text{H}$  NMR. In that case, NMR raises similar problems of interpretation as XPS and RBS, discrimination between nanocrystal ligands and free molecules is not straightforward. Nevertheless, solution NMR has the advantage of being an in situ technique that involves only a minimal perturbation of the sample, and many early studies at least suggested that NP ligands can be observed with NMR. In the remainder of this chapter, we discuss how the various pitfalls of ligand identification can be overcome with solution NMR, and show that it provides much more information on the NP ligands than a positive ligand identification only.

### 5.01.5.1 Solution NMR Techniques for Observing QD Ligands

#### 5.02.5.1.1 A brief introduction in solution NMR spectroscopy

With solution NMR spectroscopy, one investigates molecular species dissolved or suspended in a liquid sample. Only atoms that carry a nuclear spin can be observed, and only isotopes with a nuclear spin  $1/2$ , such as  $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{15}\text{N}$ , and  $^{31}\text{P}$ , are considered here as they afford high-resolution spectra to be obtained. We will explain the technique for  $^1\text{H}$ , yet the concepts apply to the other nuclei as well. The sample is placed in a homogeneous magnetic field  $B_0$ , directed along the z-axis. This field lifts the spin degeneracy of, in the case of protons, the two possible spin states (up and down). The energy difference is small, yet it results in a slight excess of spin-up protons producing a net longitudinal magnetization  $M$  along the z-axis of  $B_0$  (Figure 12(a)). The phase of the precessing spins remaining randomly distributed with respect to the xy plane, no net transverse magnetization is generated. Next, a radio-frequency magnetic field pulse  $B_1$ , along the x-axis, is used to rotate  $M$  by  $90^\circ$  into the xy plane, here along the y-axis (Figure 12(b)).



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