The Supramolecular Chemistry of Organic-Inorganic Hybrid Materials

Edited by

Knut Rurack and Ramón Martínez-Máñez



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Preface

Supramolecular chemistry, which is basically devoted to the study of the interaction between molecules, and materials chemistry, dealing with the development of solids with specific properties, are two powerful disciplines that have traditionally been poorly interrelated. Only the drive to create ever faster, ever more affordable, and ever more convenient technologies with a myriad of new and advanced features has tempted materials scientists to push the boundaries to ever smaller components and chemical researchers to design ever larger supramolecular structures, both entering into the interfacial zone of nanotechnology and nanochemistry. Function is the keyword here, especially when the aim is to design "smart" or "intelligent" materials. Inorganic supports are often inert and do not display many functions. In contrast, organic molecules can have a rich functionality, yet an ensemble of them in a disordered state—whether in solution or randomly adsorbed on a surface—often does not perform as desired. Thus, at the dawn of nanotechnology research in the late 1980s, chemists and materials scientists realized that a combination of their skills might be more successful in approaching the aims than to stay on the beaten tracks. Hence, the rapidly growing world of organic-inorganic hybrid materials emerged, producing nanoscopic matter with a plethora of novel properties and functions. Although the basic idea to combine inorganic materials with functional organic molecules might sound straightforward, its realization is connected to several challenges. Of course, "smart" hybrid materials cannot be obtained simply by teaching organic molecules to sit on an inorganic support and solve a Sudoku, play tennis, or sing a number-one hit. Organic functions on inorganic supports have to be organized and have to be orchestrated in their action, which often involves sophisticated chemistry and a structuring and patterning of the inorganic partner at molecular dimensions. At this stage, supramolecular concepts and tools from nanotechnology come into play. Only a clever combination of these strategies and techniques allows the creation of tailor-made "hetero-supramolecular" functionalities, showing new synergisms and unprecedented performance. Compared to the vast amounts of macroscopic devices available in society today and molecular biological processes operative in living organisms, naturally, only considerably few active functions have been realized in the young research field covered here. However, this book shows how a plethora of promising ideas has arisen from the combination of supramolecular chemistry, inorganic solids, and nanotechnology and has already accomplished significant advances in many areas such as sensing, controlled motion, or delivery. The objective here is to provide a compendium that gives an overview of the present state and upcoming challenges in this rapidly growing, highly cross- or interdisciplinary research field.

Flanked by three general chapters, the book is divided into five thematic sections. After a brief introduction to basic terms and concepts in the areas of supramolecular chemistry and hybrid (nano)materials in Chapter 1, Ariga et al. sketch general aspects of supramolecular chemistry related to hybrid materials and structures at the mesoscale in Chapter 2. The chapters collected in the first thematic section on Organic—Inorganic Hybrid Nanomaterials provide an in-depth introduction to synthetic strategies, major properties, characterization techniques, key features, and selected applications of today's most important families of hybrid materials: mesoporous organic—inorganic hybrid silica (Chapter 3 by Hoffmann and Fröba), modified gold nanoparticles and surfaces (Chapter 4 by Pengo and Pasquato), and organically functionalized semiconductor nanocrystals (Chapter 5 by Reiss et al.). Chapter 6 by Gu et al. deals with the functionalization of carbon nanotubes and their bioanalytical and biomedical applications and in Chapter 7, Kitagawa and Noro unfurl the world of porous coordination polymers or MOFs.

The second, third, and fourth sections comprise detailed introductions to design strategies, collective properties, signaling aspects, and/or application-oriented features of a broad variety of hybrid materials in the context of major supramolecular concepts such as assembly, sensing, switching, gating, catalysis, and molecular machinery. Part Two, Improvement of Signaling and Sensing by Organization on Surfaces, basically shows how the organization of molecular entities on surfaces can be used to enhance electrochemical or optical signaling and sensing processes for materials such as gold or silica nanoparticles and quantum dots. In Chapter 8, Polsky et al. report on biomolecular—nanoparticle hybrid systems for electrochemical signaling, followed by Guo et al.'s Chapter 9 on the use of modified nanoparticles for electrocatalysis and as amplifying sensors. The use of gold and silica nanoparticles and quantum dots for optical sensing and imaging applications is demonstrated in Chapters 10, 11, and 12 by Wei and Wei, Mancin et al., and Fernández Argüelles et al., respectively.

The section Control of Supramolecular Nanofabrication, Motion, and Morphology is devoted to state-of-the-art applications of certain supramolecular tools and functions on solid supports. In Chapters 13 and 14, Ling et al. and Rożkiewicz et al. discuss different strategies for the directed self-assembly of nanoparticles on surfaces and give an overview of immobilization and patterning techniques for the attachment of biomolecules on surfaces. The other chapters elaborate on the realization of advanced supramolecular functions on solid scaffoldings related to switchable host—guest chemistry (Chapter 15 by Kong et al.), the control of mass transport by gating in mesoporous hybrid silica (Slowing et al. in Chapter 16), the directed motion of molecular machines on surfaces (Chapter 17 by Credi et al.), and controlled changes in morphology of mesostructured hybrid materials (Dunphy et al. in Chapter 18).

The subsequent section Biomimetic Chemistry presents hybrid solids that were developed according to signaling and recognition processes established in nature (Chapters 19 and 20 by Rurack et al. and Collinson) and concludes with Chapter 21 by Kamperman and Wiesner, who show how nature's strategy of combining biomacromolecules and inorganic skeletons can be transferred to block copolymers

and inorganic nanomaterials, yielding hybrid materials with unprecedented properties and functions.

The chapters in the last section have a "wildcard" character, each touching a very particular aspect of the area of nanoscopic hybrid materials in a rather short and concise manner yet each having a background of more general importance. In Chapter 22, Shchukin et al. report on the use of hybrid nanocontainer materials as self-healing anticorrosion coatings. The ways in which adaptive or stimuli-responsive "schizophrenic" materials with a dual character might revolutionize chemo- and biosensing systems is discussed by Byrne and Diamond in Chapter 23. After all the chemistry highlighted in the previous chapters, Chapter 24 sheds light on a particular keyword that is often used by scientists in the field themselves as well as by policy makers, interdisciplinarity. Rafols et al. approach an answer to the question of how far hybrid nanomaterials research really is interdisciplinary with scientometric tools, that is, with a bibliometric analysis of the field as presented in the book. Another short chapter written by the editors completes the book by looking ahead on four exemplary research directions that have developed only in the last two to three years, during the making of the book, or that are on the verge of developing in the near future.

Finally, we would like to thank all the authors of this book wholeheartedly for their enthusiastic participation and the effort they made in preparing such interesting and stimulating chapters. To work on this book has been an exciting and pleasurable experience for us, and we are also grateful to Anita Lekhwani and Rebekah Amos of John Wiley & Sons for their belief in the book and for their help in realizing it. Of course, a book like this cannot be complete yet we hope that through this collection of excellent contributions the reader will gain profound insight into this fascinating and emerging research area, will appreciate what has been already achieved by scientists around the globe, will be captivated to keep an eye on the field in the future, and, perhaps, will be inspired to join in and discover future advances in the supramolecular chemistry of organic—inorganic hybrid materials.

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Abbreviations

2D two-dimensional 3D three-dimensional

3PL three-photon luminescence

4VP 4-vinylpyridine

AA alginic acid

AA2024 trade name of an aluminum alloy

Ab antibody ac acetate

adip 5,5'-(9,10-anthracenediyl)di-isophthalate

ADP adenosine diphosphate
AFM atomic force microscopy
Aib α-aminoisobutyric acid

Ala alanine

AM 1.5 air mass 1.5 conditions AMP adenosine monophosphate

AMS α -methylstyrene

ANA analcime

AOT sodium bis(2-ethylhexyl)sulfosuccinate
APC 2,4-bis(4-dialkylaminophenyl)-3-hydroxy-4-

alkylsulfanylcyclobut-2-enone

apoB-100 ligand of LDL receptor

Apt aptamer

APTES 3-aminopropyltriethoxysilane (frequently abbreviated as APTS

in the literature)

APTMS 3-aminopropyltrimethoxysilane

Asn asparagine

ASTM American Society for Testing and Materials

atp aminoterephthalate ATP adenosine triphosphate

ATR attenuated total reflection (spectroscopy)
ATRP atom transfer radical polymerization

AuNP gold nanoparticle

Az azurin

azpy trans-4,4'-azopyridine

xx Abbreviations

αCP affinity contact printing

 αG α -L-guluronic acid (frequently abbreviated as G in the literature)

b block (in block copolymer nomenclature)

B50-6600 trade name of a block copolymer EO₃₉BO₄₇EO₃₉

BaM barium ferrite nanocrystals

BBDA N,N'-bis(4-tert-butylphenyl)-N,N'-bis(4-((E)-2-

(triethoxysilyl)vinyl)phenyl)biphenyl-4,4'-diamine

bbim 1,3-dibutylimidazolium

bBSA biotinylated bovine serum albumin BCAm benzo[18]crown-6-acrylamide

bdc benzene dicarboxylate
BDD boron-doped diamond
BDMS bis(tert-butyldimethylsilyl)

BEA beta (zeolite beta)

BET Brunauer-Emmett-Teller

BFc biferrocene

BHEEEN 1,5-bis[2-(2-(2-hydroxyethoxy)ethoxy)ethoxy]naphthalene BINAP 2,2'-bis-(diphenylphosphino)-1,1'-binaphthyl (also: binap)

BJH Barrett-Joyner-Halenda

BO butyleneoxide

Boc *tert*-butoxycarbonyl BP benzophenone

bpbp 4,4'-bis(4-pyridyl)biphenyl bpethe 1,2-bis(4-pyridyl)ethene

bpy bipyridine/yl

btb

Brij 56 trade name of a poly(ethylene glycol hexadecyl ether) detergent

and emulsifier

BS³ bis(sulfosuccinimidyl)suberate

BSA bovine serum albumin

btapa 1,3,5-benzenetricarboxylic acid tris[*N*-(4-pyridyl)amide]

btc benzene tricarboxylate
BTEB 1,4-bis(triethoxysilyl)benzene
BTEBP 4,4'-bis(triethoxysilyl)bipheny

BTEBP 4,4'-bis(triethoxysilyl)biphenyl BTEE 1,2-bis(triethoxysilyl)ethane

BTEMEB 1,4-bis(triethoxysilyl)-2-(1-methoxyethyl)benzene

1,3,5-benzenetribenzoate

BTESM bis-triethoxysilylmethane BTET 2,5-bis(triethoxysilyl)thiophene

BTEX benzene, toluene, ethylbenzene, xylenes

BTEY 1,2-bis(triethoxysilyl)ethene
BTME 1,2-bis(trimethoxysilyl)ethane
BTMSEB 1,4-bis(trimethoxysilylethyl)benzene
BTMSPA bis[3-(trimethoxysilyl)propyl]amine

btt 1,3,5-benzenetristetrazolate

Bu butyl bza benzoate

βM β-D-mannuronic acid (frequently abbreviated as

M in the literature)

CA cancer antigen

CALNN Cys-Ala-Leu-Asn-Asn

cAMP cyclic AMP

CASH combined assembly by soft and hard (chemistries)

CB conduction band CB[6] cucurbit[6]uril

CBED convergent beam electron diffraction (patterns)

CBPQT cyclobis(paraquat-p-phenylene)

CCD charge-coupled device

CCM Cornell composition of matter (family of materials)

CD cyclodextrin

CD4 (cluster of differentiation 4) glycoprotein, a co-receptor of the T

(thymus) cell receptor

CDA cell-directed assembly
CE capillary electrophoresis
CE cholesterol esterase
CFU colony forming units
chbt cyclohexylbutyrate

CHI chitosan

CHO Chinese hamster ovary
CMC critical micelle concentration

CMK-n¹ carbon mesostructured by Korea Advanced Institute of Science

and Technology (family of mesoporous carbon materials)

CNT carbon nanotubes

co copolymer
cod cyclooctadiene
COx cholesterol oxidase
CP conjugated polymer

CP/MAS cross-polarization MAS (NMR)
CPB hexadecylpyridinium bromide
CPC hexadecylpyridinium chloride
CPL coordination pillared layer

CPO chloroperoxidase CPU central processing unit

CS core-shell CT charge-transfer

CT computed tomography

CTAB cetyltrimethylammonium bromide

xxii Abbreviations

CTAC cetyltrimethylammonium chloride CTES carboxyethylsilanetriol, sodium salt

CV cyclic voltammetry
CVD chemical vapor deposition

CW continuous wave

CXCR4 CXC chemokine receptor; CXC stands for a C-X-C motif with

C = cysteine and X = arbitrary amino acid

Cy3 carbocyanin 3 Cy3.5 carbocyanin 3.5

Cys cysteine

DA dodecylamine (frequently abbreviated as DDA in the literature)

dabco diazabicyclo[2.2.2]octane DAP diaminopyrimidine

DAR diazo resin

DART direct analysis in real time (ionization technique in MS)

DAT diaminotriazine
DB24C8 dibenzo[24]crown-8
DBM dibenzoylmethane
DBS dibutyl sebacate

DDA discrete dipole approximation

DDAB dilauryldimethylammonium bromide

DFT density functional theory dhbc 2,5-dihydroxybenzoate DHLA dihydrolipoic acid DIEA diisopropylethylamine

diglyme diethylene glycol dimethyl ether dipn N,N-di(3-aminopropyl)amine

diPyNI N,N'-di-(4-pyridyl)-1,4,5,8-naphthalenetetracarboxydiimide

DMAP 4-dimethylaminopyridine

DMB (1R,2S)-(-)-N-dodecyl-N-methylephedrinium bromide

DMF N,N-dimethylformamide
DMPA dimyristoylphosphatidyl
DMSO dimethyl sulfoxide
DNA deoxyribonucleic acid
DOE U.S. Department of Energy
DON 1,5-dioxynaphthalene

DOPA D-/L-3,4-dihydroxyphenylalanine

DOPO-Br *p*-bromobenzyl-di-*n*-octylphosphine oxide

DOX doxorubicin

DPAR 4-*n*-dodecyl-6-(2-pyridylazo)phenol

DPC diphenylcarbazide
DPN dip-pen nanolithography
dpp 4,4'-diphenylphenanthroline
DPV differential pulse voltammetry

dpyg 1,2-di(4-pyridyl)glycol dsDNA double-stranded DNA DSG disuccinimidylglutarate

DsRed mutant of red fluorescent protein

DT dodecanethiol

DTA differential thermal analysis

DTAR 4-*n*-dodecyl-6-(2-thiazolylazo)resorcinol

DTC dithiocarbamate dtoa dithiooxamide

DTPA diethylenetriaminepentaacetic acid

DTT dithiothreitol
DVB divinylbenzene
dx decylxanthate

DZ diphenylthiocarbazone

E energy e electron

eAuNP enlarged AuNP

ECL electrochemiluminescence

EDAC see EDC

EDC 1-ethyl-3-[(3-dimethylamino)propyl]carbodiimide hydrochloride

EDOT 3,4-ethylenedioxythiophene
EDS energy-dispersive spectroscopy
EDTA ethylenediaminetetraacetic acid
EELS electron energy loss spectroscopy

EFTEM energy-filtered TEM
EG4 tetra(ethylene glycol)
EG6 hexa(ethylene glycol)

EGDMA ethylene glycol dimethacrylate
EGFP enhanced green fluorescent protein
EGFR epidermal growth factor receptor
EHTES 5,6-epoxyhexyltriethoxysilane

eim 2-ethylimidazolate

EISA evaporation-induced self-assembly ELISA enzyme linked immunosorbent assay

EO ethyleneoxide

EPR enhanced permeability and retention

ER electrorheological ESR electron spin resonance

Et ethyl

EU European Union ex ethylxanthate

F88 trade name for a Pluronic surfactant F127 trade name for a Pluronic surfactant

xxiv Abbreviations

FA folic acid

FAD flavin adenine dinucleotide Fc ferrocene or ferrocenyl

Fc fragment, crystallizable (of an antibody)

Fc-D ferrocenyl-tethered dendrimer

FCC fluid catalytic cracking

FcN (R)-/(S)-N,N'-dimethylferrocenylethylamine

FDA U.S. Food and Drug Administration

FDM ferrocene dimethanol

FDMDG ferrocene dimethanol diethylene glycol FDTD finite difference time domain (method)

FDU-*n* Fudan University (family of mesoporous silicas)

FET field effect transistor
FGD flue gas desulfurization
FIA fluoroimmunoassay
FITC fluorescein isothiocyanate
Fmoc fluorenylmethoxycarbonyl

FND fluorescent ND

FNP fluorescently doped nanoparticles

FR⁺ receptors for FA ligands

FRET Förster resonance energy transfer (frequently yet inappropriately

referred to as fluorescence resonance energy transfer, see IUPAC

recommendations)²

FSM-*n* (family of) folded sheet mesoporous (materials)

FTIR Fourier transform IR

FUM fumaramide

Fur ferric uptake regulator FWHM full width at half maximum

G generation (of dendrimer) GAG glycosaminoglycan

Gal galactose

GBP gold binding polypeptide
GCE glassy carbon electrode
GCNF graphitic carbon nanofiber

GEPI genetically engineered peptides for inorganics

gFND green FND

GFP green fluorescent protein

Glu glutamine

GLYMO 3-glycidyloxypropyltrimethoxysilane

GMP guanosine monophosphate

GOx glucose oxidase

GSH reduced form of glutathione GSSG oxidized form of glutathione h⁺ hole

h-h head-to-head coupling
HDA hexadecylamine
hdx hexadecylxanthate

HeLa Henrietta Lacks (HeLa cells: immortal cell line derived from

H.L.'s cervical cancer cells)

HETCOR heteronuclear correlation (NMR)

HFBI hydrophobin

HFE gene responsible for hereditary hemochromatosis

HH hexadecyl hexadecanoate
HHCC horse heart cytochrome c
HI hot-injection method

His histidine

HIV human immunodeficiency virus

HMDS hexamethyldisilazane

HOMO highest occupied molecular orbital
HOPG highly ordered pyrolytic graphite
HPLC high pressure liquid chromatography

HREELS high-resolution EELS
HRP horseradish peroxidase
HRS hyper-Rayleigh scattering
HRTEM high resolution TEM

HSMA hydrolyzed poly(styrene-alt-maleic anhydride)

HU heating up method

I inorganic species

IC₅₀ half-maximum inhibitory concentration iCCD intensified charge-coupled device

ICS ion channel sensor ICS isocyanurate

ICTES (3-isocyanatopropyl)triethoxysilane (frequently abbreviated as

ICPES in the literature)

IgE immunoglobulin E
IgG immunoglobulin G
IL ionic liquid

IL ionic liquid im imidazolate

IMAC immobilized metal ion affinity chromatography

IR infrared

IRMOF-*n*³ isoreticular metal–organic framework

ISE ion-selective electrode

ITO indium tin oxide

IUPAC International Union of Pure and Applied Chemistry

xxvi Abbreviations

KIT- n^4 (family of) large mesopore Fm3 m silica(s)

LA lauric acid

LB Langmuir-Blodgett
LbL layer-by-layer

LC liquid chromatography LC liquid crystal/crystalline

LCST lower critical solution temperature

LD lethal dose

LDL low density lipoprotein LD-SAM low density SAM LED light emitting diode

Leu leucine

LOD limit of detection

LPEI linear poly(ethylene imine)

LSPR localized surface plasmon resonance

LTA Linde type A (zeolite A)

LUMO lowest unoccupied molecular orbital

M41S^{5,6} family of mesoporous molecular sieves

MA myristic acid MAA methacrylic acid mAb monoclonal Ab

MagMOON magnetically-modulated optical nanoprobes

MAL maleamide

MALDI-TOF matrix assisted laser desorption/ionization time-of-flight (MS)

MAS magic angle spinning NMR

MAXSORB trade name of an activated carbon adsorbent

MBP maltose binding protein

MBP-zb maltose binding protein with a positive leucine zipper domain

MC merocyanine

MCF-7 human breast cancer cells

MCM-*n* Mobil Composition of Matter (family of mesoporous silicas)

MDMO-PPV poly[2-methoxy-5-(3',7'-dimethyloctyloxy)-1,4-

phenylenevinylene]

Me methyl

MEH-PPV poly(2-methoxy-5-(2'-ethyl-hexyloxy)-1,4-phenylene-vinylene)

MeOTAD 2,2',7,7'-tetrakis(N,N-di-p-methoxyphenylamine)9,9'-

spirobifluorene

MFI Mobil five (ZSM-5; zeolite)

MHDA 16-mercaptohexadecanoic acid (frequently abbreviated as MHA

in the literature)

MHT 1-(6-mercaptohexyl)thymine MHV68 murine gammaherpesvirus 68 $MIL-n^7$ Materials of Institut Lavoisier (family of metal-organic

frameworks)

mim 2-methylimidazolate

MIMIC micromolding in capillaries
MIP molecularly imprinted polymer

MM methyl myristate
MNP magnetic nanoparticles
MO molecular orbital

MOF metal-organic framework
MOR methanol oxidation reaction
MPA mercaptopropionic acid
MPC monolayer protected cluster
MPL multiphoton luminescence

MPMD (mercaptopropyl)methyldimethoxysilane

MPTES 3-mercaptopropyltriethoxysilane

MPTMS 3-mercaptopropyltrimethoxysilane (frequently abbreviated as

MPMS in the literature)

MRI magnetic resonance imaging

MS mass spectrometry

MSH α-melanocyte stimulating hormone MSN mesoporous silica nanoparticles

MSU-*x* Michigan State University (family of mesoporous silicas)

mTERT murine telomerase reverse transcriptase

MTG methanol-to-gasoline MTMOS methyltrimethoxysilane

MTX methotrexate

MUA 11-mercaptoundecanonic acid MWNT multiwalled carbon nanotube

my myristate

 $\begin{array}{ll} \mu CP & \text{microcontact printing} \\ \mu FN & \text{microfluidic network} \\ \mu TM & \text{microtransfer molding} \end{array}$

n wildcard character for numbers

NBIC nanotechnology, biotechnology, information technology, and

cognitive science

NBTC Nanobiotechnology Center (Cornell University)

NC nanocrystal ND nanodiamond

Nd:YAG neodymium-doped yttrium aluminium garnet (laser)

ndc 2,6-naphthalenedicarboxylate

NDR 2-nitro-*N*-methyl-4-diazonium-formaldehyde resin NEST New and Emerging Science and Technology

NEXAFS near-edge x-ray absorption fine structure (spectroscopy)

xxviii Abbreviations

NHA carbon nanohorn aggregate NHS N-hydroxysuccinimide/yl

NHSC₁₁SH 11-mercaptoundecanoyl-*N*-hydroxysuccinimide ester

NIL nanoimprint lithography

NIR near infrared NLO nonlinear optics

NLS nuclear localization sequence (peptide sequence for nuclear

targeting)

NMR nuclear magnetic resonance NN next-neighbor (interactions)

NOM nano-on-micro NP nanoparticle NR nanorod

NTA nitrilotriacetic acid

NTS nonadecenyltrichlorosilane
NV nitrogen vacancy (defect sites)
NVOC nitroveratryloxycarbonyl
NVP naphthalenylvinylpyridine

OA oleic acid OAm oleylamine

OCT optical coherence tomography

ODA octadecylamine ODE 1-octadecene

ODPA octadecylphosphonic acid

ODT octadecanethiol

OFMS organic-functionalized molecular sieve

ON oxynaphthalene

OPTA *o*-phthalic hemithioacetal

OR alkoxy group

ORMOSIL organically modified silicate
ORR oxygen reduction reaction
ORTEP Oak Ridge thermal ellipsoid plot

OT octanethiol

OTAB octadecyltrimethylammonium bromide OTAC octadecyltrimethylammonium chloride

OTF trifluoromethanesulfonate
OTS octadecyltrichlorosilane

ox oxalate

P123 trade name for a Pluronic block copolymer

P3HT poly(3-hexylthiophene)

P3(ODAP)HT poly(3-(6-oxy-2,4-diaminopyrimidine)hexylthiophene) P3TOPA poly(3-(3'-thienyloxy)propyltrimethylammonium)