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Design strategies and applications of multi-macrocyclic hosts for supramolecular functional materials

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ABSTRACT

Macrocyclic compounds play a fundamental role in supramolecular chemistry, demonstrating significant prospect in functional materials owing to their precise cavities and tunable molecular recognition and assembly capabilities. However, the only cavity of the mono-macrocyclic hosts limited the development of traditional mono-macrocycle-based supramolecular materials in assembly dimensionality, recognition selectivity, and synergistic performance. Therefore, multi-macrocyclic hosts attracted increasing attention due to their unique advantages originated from multi-cavity cooperativity, which include more abundant guest suitability, multiple-dimension assembly, precise collaborative effect, and so on. Based on these merits, introducing multi-macrocyclic hosts into supramolecular materials can efficiently improve and enrich the properties of these kinds of materials. This review summarizes recent innovative design and constructing strategies for multi-macrocyclic hosts, including covalent chain bridged, covalent fused, coordinated, and mechanically interlocked pathway. And their applications in recognition, luminescence, catalysis, biomedical and chiral materials are highlighted, while the advantages of multi-macrocycles in enhancing binding affinity, selectivity, and stimulus-responsive behavior are elucidated. Furthermore, we discuss future directions, such as novel multifunctional hosts design, optimization of assembly strategies, and interdisciplinary applications, providing theoretical insights and practical guidance for the development of multi-macrocycles-based high performance supramolecular materials.

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1. Introduction

Macrocyclic compounds are quintessential supramolecular hosts, playing a pivotal role in supramolecular chemistry [1–15]. To date, numerous macrocyclic families have been developed, with crown ethers [16,17], cyclodextrins [18–20], cucurbiturils [21,22], calixarenes [23,24], and pillararene [25–29] serving as representative examples. These architectures exhibit distinctive host-guest recognition and supramolecular assembly properties based on their tunable binding capabilities which stems from their curved, twisted or rigid cyclic cavities. Such structural features facilitate selective molecular recognition through mechanisms of size complementarity, geometric constraints, and electronic interactions [30–40]. Therefore, these compounds show nice application prospect in various fields [41–49].

Conventional mono-macrocyclic molecules have demonstrated various advantages in molecular recognition and self-assembly, however, the mono-macrocyclic structure limit their functional diversity and restrict their applications in complex systems [50–56]. Specifically, during self-assembly processes, mono-macrocyclic molecules only can supply one cavity, which limit the assembly dimensionality for constructing sophisticated supramolecular architectures [57,58]. When interacting with guest molecules, the lack of multi-site synergistic effects often results in insufficient binding strength and selectivity to meet the requirements of high-precision recognition [59]. Furthermore, in response to external stimuli such as light, heat, or pH changes, mono-macrocyclic molecules generally suffer from poor tunability induced low responsiveness [60].

In order to overcome the limitations of mono-macrocyclic molecules, researchers have proposed construction strategies for multi-macrocyclic hosts in recent years [61–63]. These molecular systems, formed by linking multiple macrocyclic units *via* covalent or non-covalent interactions, are assuming an increasingly

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prominent role in supramolecular chemistry [64–82]. Compared to mono-macrocyclic systems, these architectures exhibit unique structural features that enable multi-cycle-synergy-based distinctive recognition, multi-dimensionality assembly behaviors, leading to widespread applications in molecular recognition [83–87], supramolecular self-assembly [88,89], and advanced materials [90–95].

This review systematically summarizes the latest research advancements in bi-macrocyclic, tri-macrocyclic, and multi-macrocyclic hosts over the past decade. It elaborates on their innovative design and construction strategies based on distinct connection methods including covalent chain bridged, covalent fused, coordinated, and mechanically interlocked pathway. Further presents the most recent research progress on the applications of these systems in fields such as sensing and recognition, luminescent materials, supramolecular catalysis, biomedical and chiral materials. Furthermore, the work provides an outlook on future development of multi-macrocyclic supramolecular systems.

2. Strategies on construction multi-macrocyclic hosts

Multi-macrocyclic supramolecular functional materials, as hierarchically ordered architectures assembled through noncovalent or dynamic covalent interactions, exhibit structural and functional properties critically governed by their interconnection modes [96]. These materials are currently classified into four primary paradigms: covalent chain-bridged multi-macrocycles [97], covalent fused multi-macrocycles [98], coordinated multi-macrocycles [99], and mechanically interlocked macrocycle [100], each imparting distinct synergistic effects that enable precise modulation of assembly morphology, stimuli-responsive behavior, and functional performance. Systematic exploration and optimization of these topological strategies not only establish fundamental design principles but also advance the development of high-performance smart materials with transformative applications in biomedicine, energy storage, and adaptive devices [101–105].

2.1. Covalent chain bridged multi-macrocycles

Covalent chain-bridged macrocyclic compounds are multi-macrocyclic systems formed by connecting a mono-macrocyclic molecule through flexible or rigid connecting groups such as alkyl chains, aryl groups, polyether, demonstrating significant potential in the fields of molecular recognition, catalysis, biomedicine and material science. Its core advantage is that by designing the chemical composition and spatial configuration of the connecting group, the microenvironment of the macrocyclic cavity (such as size, electronic effect and steric hindrance) can be accurately regulated. More importantly, intermolecular or intramolecular adjacent multi-macrocycles can supply synergistic effect through collaboration of them, which enhance the guest binding ability of the host and provide a unique platform for the construction of smart molecular devices and the development of high-performance materials [106].

In 2018, Stang research team designed and synthesized a tetra-crown ether macrocyclic compound **1** based on a tetraphenylethylene (TPE) rigid linker (Fig. 1) [107]. This study employed a metal coordination-driven self-assembly strategy to first construct a square prism-shaped metallacage structure functionalized with four 21-crown-7 moieties. Subsequently, through host-guest interactions between the 21C7 groups and a diammonium salt linker $\mathbf{G_1}$, a three-dimensional (3D) supramolecular polymer network was successfully fabricated, which could form a stable supramolecular gel system at high concentrations. Notably, owing to the incorporation of the TPE fluorophore, the resulting gel

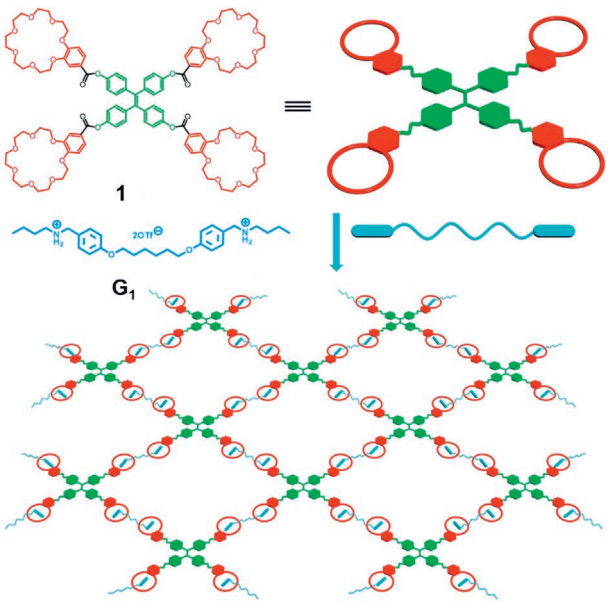


Fig. 1. Molecular structure (left) and cartoon representation (right) of the TPE-bridged tetra-crown ether macrocyclic compound **1**. and achematic illustration of the two-dimensional supramolecular grid structure formed through orthogonal self-assembly between **1** and $\mathbf{G_1}$. Copied with permission [107]. Copyright 2018, American Chemical Society.

exhibited remarkable fluorescence emission properties. This work not only expanded the functionalization of fluorescent metallacage through sophisticated ligand design but also provided a methodology for preparing stimuli-responsive and self-healing supramolecular gels.

In 2021, our group introduced the concept of "synergistic effects" into the design of pillar[5]arene-based materials [108]. We designed and synthesized a novel linear-bridged tri-pillar[5]arene receptor **2** (Fig. 2), in which tri-pillar[5]arene units were rationally interconnected within a single receptor molecule. As anticipated, the linear pillar[5]arene **2** not only exhibited fluorescence responsiveness but also demonstrated high-efficiency separation and adsorption capabilities toward methyl viologen ($\mathbf{G_2}$). Compared with conventional adsorbents such as activated carbon and mono-pillar[5]arene receptors, compound **2** showed superior adsorption performance for methyl viologen ($\mathbf{G_2}$), which was attributed to the synergistic interactions between its adjacent pillar[5]arene moieties. The **2** could serve as a paraquat adsorbent material and convey greatly potential application in the field of removal of paraquat. The concept "employ collaboration effect to enhance the host-guest interactions" is a useful way for the development of adsorption materials.

In 2023, Chen and co-workers synthesized a novel bi-macrocyclic molecule **3** by covalently linking two distinct macrocyclic hosts, water-soluble pillar[5]arene and β-cyclodextrin, *via* a flexible spacer (Fig. 3) [109]. Through orthogonal host-guest recognition and co-assembly, **3** specifically interacted with a photosensitizer ($\mathbf{G_3}$) and a camptothecin prodrug ($\mathbf{G_4}$) to form supramolecular nanoparticles (NPs) **3-NPs**. Notably, the ratio of $\mathbf{G_3}$ to $\mathbf{G_4}$ in **3-NPs** could be precisely tuned as needed. Owing to the host-guest interactions and stable self-assembly, **3-NPs** exhibited exceptional colloidal stability and resistance to photobleaching. Under reducing conditions, camptothecin could be released for chemotherapy, while efficient singlet oxygen generation upon light irradiation enabled photodynamic therapy. This bi-macrocycles-based host-guest assembly strategy provides a new approach for constructing combined chemo-photodynamic therapeutic systems or even dual-drug combination therapies.

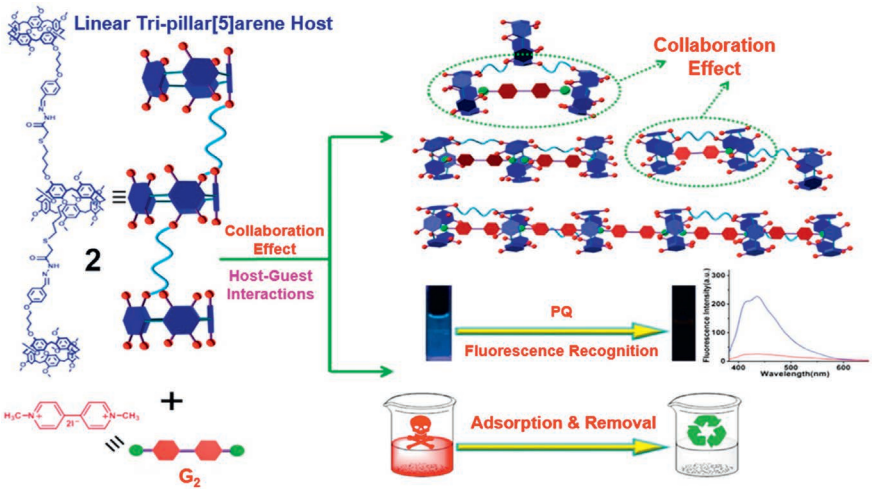


Fig. 2. Schematic illustration of the fluorescence sensing and adsorption mechanism of bridged tri-pillar[5]arene **2** toward paraquat. Copied with permission [108]. Copyright 2021, Elsevier.

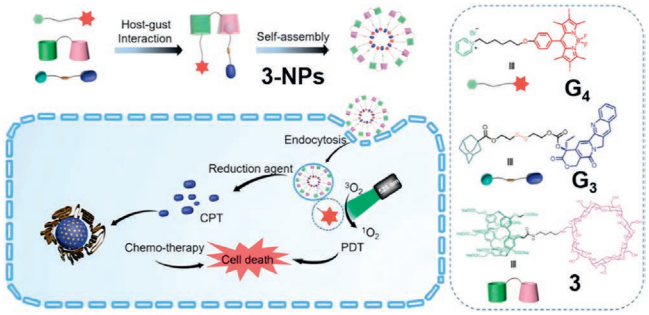


Fig. 3. Chemical structures of the bi-macrocyclic molecule **3** and guest molecules **G3**/**G4**, and schematic illustration of supramolecular nanoparticle assembly for photodynamic therapy. Copied with permission [109]. Copyright 2023, Elsevier.

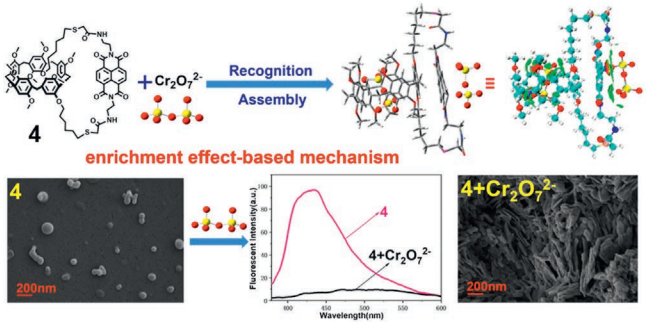


Fig. 4. Schematic illustration of the highly selective and sensitive recognition of $Cr_2O_7^{2-}$ by fused bi-macrocyclic hosts **4**. Copied with permission [111]. Copyright 2024, Elsevier.

2.2. Covalent fused multi-macrocycles

Covalent fused multi-macrocyclic compounds are covalently linked multi-macrocyclic systems formed by macrocycle-to-macrocycle condensation. Its core advantage is that by regulating the interaction between macrocycle, it can break through the limitations of traditional mono-macrocyclic performance and provide a characteristic platform for the design of intelligent molecular devices [110].

Later in 2024, our group designed and synthesized a novel fused bi-macrocyclic hosts molecule **4** (Fig. 4) based on pillar[5]arene [111]. In **4**, the electron-rich cavity of pillar[5]arene is designed to complex cationic species accompanying dichromate anions ($Cr_2O_7^{2-}$), while the methoxy groups on the pillar[5]arene provide multiple hydrogen-bonding sites for $Cr_2O_7^{2-}$ binding. Furthermore, a naphthalenediimide (NDI) unit and one phenyl group of the pillar[5]arene are bridged by an alkyl chain to form a side loop, which offers additional supramolecular interaction sites for enhanced $Cr_2O_7^{2-}$ binding. Based on the individual double macrocyclic hosts **4**, multiple $Cr_2O_7^{2-}$ anions can be bound. The work provided an easy way to improving the sensitivity and selectivity of chemosensor by inducing enrichment effect.

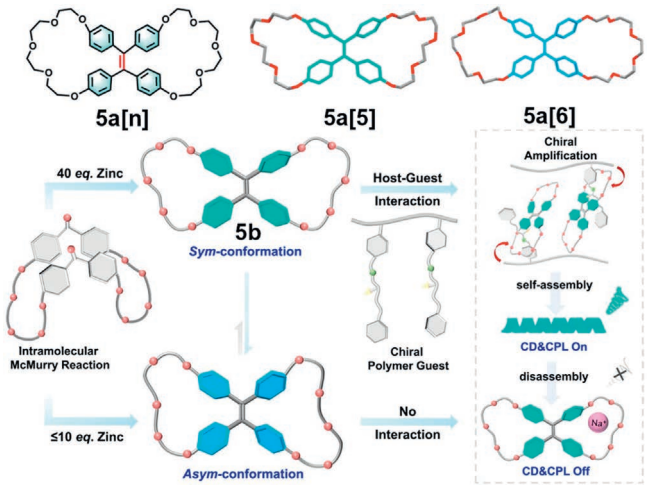


Fig. 5. Molecular structure of fused bi-macrocyclic hosts **5a** and schematic illustration of guest-induced chiral supramolecular assembly formation through host-guest interactions. Copied with permission [112]. Copyright 2024, Nature Publishing Group.

In 2024, Hu *et al.* reported an intramolecular coupling strategy to synthesize a series of TPE-based butterfly-shaped bi-crown ether macrocyclic (**5a[n]**, $n = 4–7$), featuring two crown ether side loops and a central TPE core [112]. The variable-length flexible crown ether chains modulate molecular conformations through multiple intramolecular interactions, yielding two distinct semi-rigid stereostructures with specific symmetry/asymmetry (Fig. 5). These conformational isomers exhibit divergent fluorescence properties and host-guest binding capabilities. Significantly, only **5b** functions as a chiral polymeric hosts, inducing the formation of

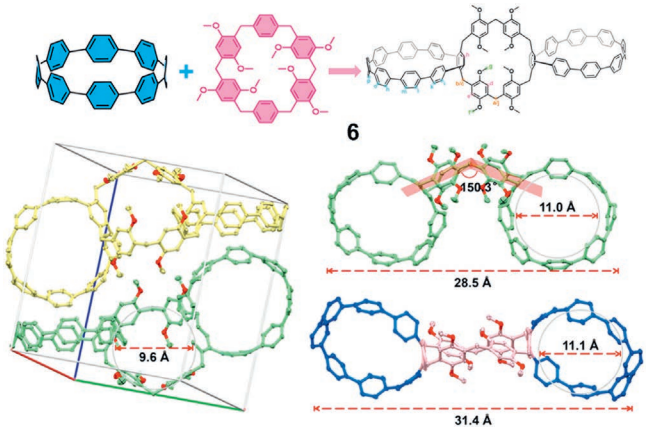


Fig. 6. Molecular and crystal structures of rigid fused tri-macrocyclic hosts molecule **6**. Copied with permission [113]. Copyright 2025, American Chemical Society.

chiral supramolecular assemblies through host-guest interactions with L- and D-type amino acid derivatives. Furthermore, both the circular dichroism (CD) and circularly polarized luminescence (CPL) signals of these chiral assemblies can be effectively "switched off" by Na^+ addition. The work not only provides fundamental insights into structure-property relationships but also offers practical guidance for developing advanced materials with tailored optical and chiral recognition functionalities.

Very recently, Cheng's team reported a rigid fused tri-macrocyclic hosts molecule **6**, consisting of two [8]cycloparaphenylene ([8]CPP) units and one pillar[6]arene unit (Fig. 6) [113]. This novel macrocyclic architecture enabled the isolation of both enantiomers and a meso–compound, providing a rare example of stereoisomerism in fused multi-macrocyclic systems. Through comprehensive structural characterization and photophysical studies, the researchers elucidated the distinct solid-state packing modes of these isomers, which significantly influenced their chiroptical properties. This work not only advances the synthetic strategies for multicyclic chiral macrocycle but also deepens the understanding of how molecular topology influences stereochemistry and functional properties in the solid state.

2.3. Coordinated multi-macrocycles

Metal-coordinated multi-macrocyclic compounds refer to supramolecular systems formed through the dynamic linkage of individual macrocycle *via* metal coordination [114]. In these architectures, metal ions or clusters serve as "nodes" that connect two or more macrocycle through coordinate bonds, generating well-defined two- or three-dimensional structures [115]. This linkage strategy offers dynamic reversibility and structural tunability, enabling system reconfiguration through modulation of metal species, ligand design, and external conditions [116].

In 2020, Huang and co-workers synthesized four optically active metallacycles (**7a**–**d**, Fig. 7) with planar chirality by employing 60° and 90° Pt(II) acceptors and planar chiral pillar[5]arene ligands [117]. These tetra-nuclear Pt-coordinated macrocycle exhibited distinct chiroptical properties: the *pS* enantiomers displayed negative Cotton effects, while the *pR* enantiomers showed positive Cotton effects in CD spectra. Notably, all metallacycles demonstrated CPL activity, suggesting their potential for optoelectronic applications. The work established a novel strategy for constructing planar chiral metal-organic macrocycle (MOCs), advancing the study of chiral supramolecular self-assembly and structure-property relationships.

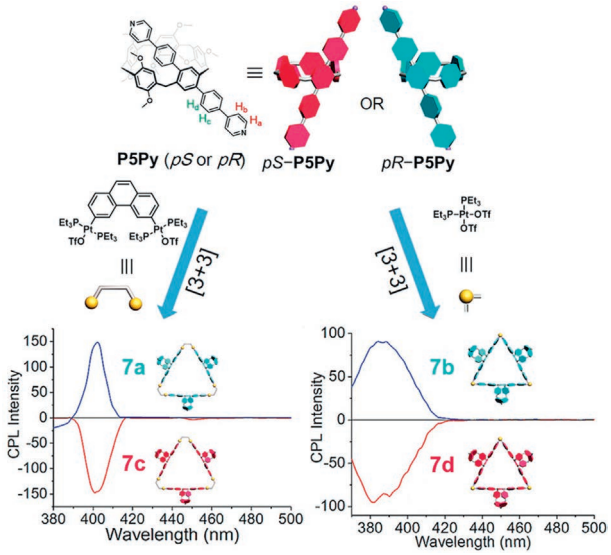


Fig. 7. Synthetic route to planar chiral tetra-nuclear metallacycles **7a**–**7d** and corresponding CPL spectra demonstrating chiroptical activity. Copied with permission [117]. Copyright 2020, American Chemical Society.

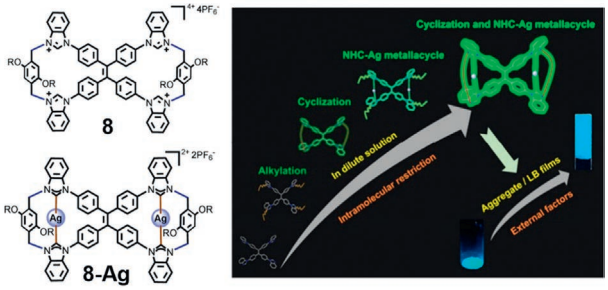


Fig. 8. Molecular structures and photoluminescence enhancement mechanism of bi-macrocycles **8** and binuclear **8-Ag** complex. Copied with permission [118]. Copyright 2024, American Chemical Society.

In 2024, the Qian team reported an amphiphilic TPE-based bi-macrocyclic hosts **8** featuring benzimidazolium salt moieties, along with its binuclear silver-coordinated counterpart **8-Ag** (Fig. 8) [118]. Both macrocycle exhibited aggregation-induced emission (AIE) characteristics in solution, aggregated states, and thin films. The work not only provides a strategy for designing amphiphilic AIE-active macrocycle but also highlights the synergistic role of metal coordination and supramolecular assembly in modulating luminescence properties. The system holds promise for applications in stimuli-responsive smart materials and interfacial nanoscience.

Very recently, Deng *et al.* developed a series of copper(I)-based mononuclear metallacycles 9 featuring [2]catenane ligands through a combined strategy of mechanical interlocking and covalent modification (Fig. 9) [119]. These complexes demonstrated exceptional performance in the electrocatalytic nitrate reduction reaction (NO_3RR), achieving highly selective ammonia (NH_3) generation. This work successfully addressed the long-standing challenge of balancing activity, selectivity, and durability in non-precious metal catalysts, offering new perspectives for sustainable nitrogen cycling.

2.4. Mechanically interlocked macrocycle

Mechanically interlocked macrocyclic compounds refer to supramolecular architectures formed through the dynamic inter-

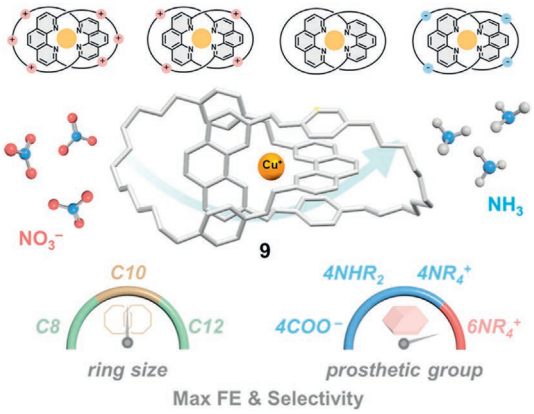


Fig. 9. Molecular structure of [2]catenane-ligated Cu(I) mononuclear bi-macrocycles **9**. Copied with permission [119]. Copyright 2025, American Chemical Society.

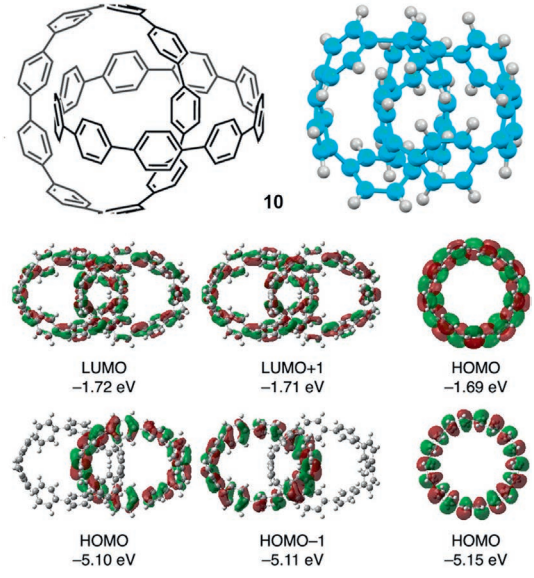


Fig. 10. Structure, crystallographic data, and frontier orbital analysis of [9]CPP-based [2]catenane **10**. Copied with permission [124]. Copyright 2024, American Chemical Society.

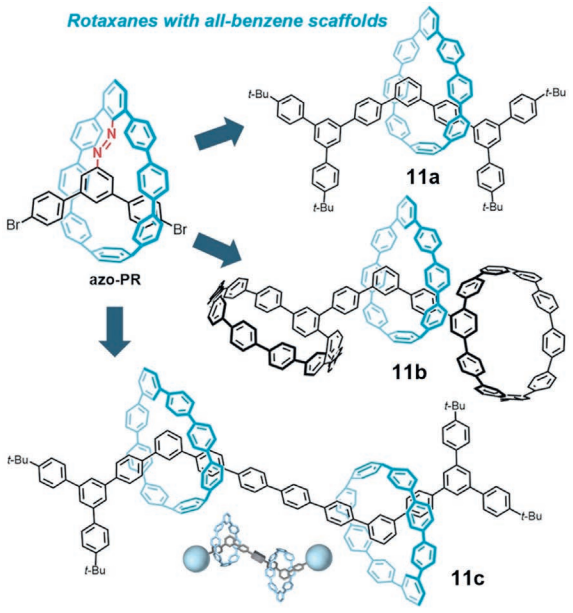


Fig. 11. The structures of pseudorotaxane azo-PR, all-benzene skeleton [2]- and [3]rotaxane **11a–c**. Copied with permission [125]. Copyright 2024, WILEY-VCH.

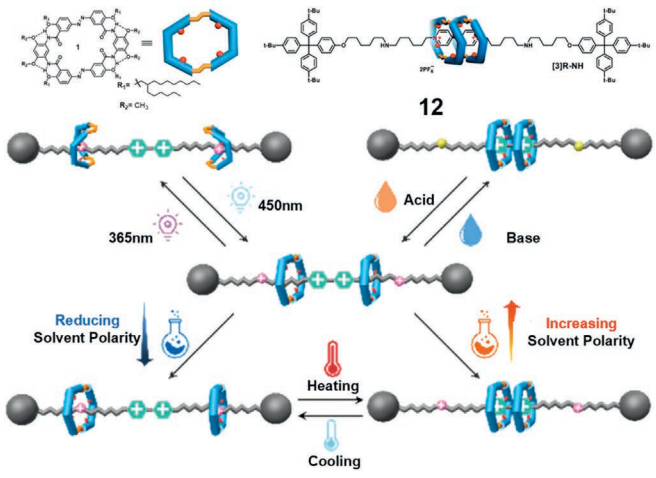


Fig. 12. The structure of [3]rotaxane compound **12** and the schematic diagram of multiple stimulus response. Copied with permission [126]. Copyright 2025, Elsevier.

twining or threading of individual macrocycle *via* non-covalent interactions (rotaxanes, catenanes [120–122]. Their defining characteristic is the absence of covalent bonds between constituent macrocycle, with stability achieved instead through physical interlocking or mechanical bonding [123].

In 2024, Kenichiro Itami and co-workers reported the synthesis of a [9]cycloparaphenylene ([9]CPP)-based bi-macrocycles [2]catenane **10** (Fig. 10) [124]. Single-crystal X-ray diffraction analysis unambiguously confirmed its interlocked architecture. While the UV–vis absorption spectrum showed remarkable similarity to the monomeric precursor, the fluorescence emission exhibited a **10** nm bathochromic shift accompanied by a decrease in quantum yield from 0.73 to 0.54 and a reduced lifetime of 11.3 ns. The observed photophysical modifications suggest substantial inter-macrocycle electronic communication in the excited state, providing new insights into through-space conjugated systems. This advancement opens new avenues for designing functional materials with tailored optoelectronic properties.

In 2024, Cong and co-workers designed and synthesized a separable azo-linked pseudorotaxane azo-PR, and used azo-PR as the core template to construct the all-benzene skeleton [2]- and [3]rotaxane **11a–c** (Fig. 11), which not only enriched the supramolecular topology of the all-benzene interlocking structure, but also demonstrated the practicability of the azo-traceless template in the construction of mechanical bonds [125]. The synthesis of all-phenylene rotaxane and its related controllable motion properties are expected to provide new ideas for the design and synthesis of molecular switches and nanomachines in the future.

Very recently, Yuan's team reported a multi-stimuli-responsive [3]rotaxane compound **12** based on hydrogen-bonded aromatic amide azo macrocycle (Fig. 12) [126]. The molecule realizes the dynamic shuttle of macrocyclic components under acid-base, temperature, solvent polarity and light conditions through the double recognition sites of secondary ammonium cation and 4,4′-bipyridinium. This system pioneered the application of hydrogen-bonded aromatic amide macrocycle in higher-order rotaxanes and realized the precise regulation of mechanical interlocking molecular motion through multi-mode stimulation response mechanism, providing new ideas for the development of intelligent molecular devices and adaptive materials.

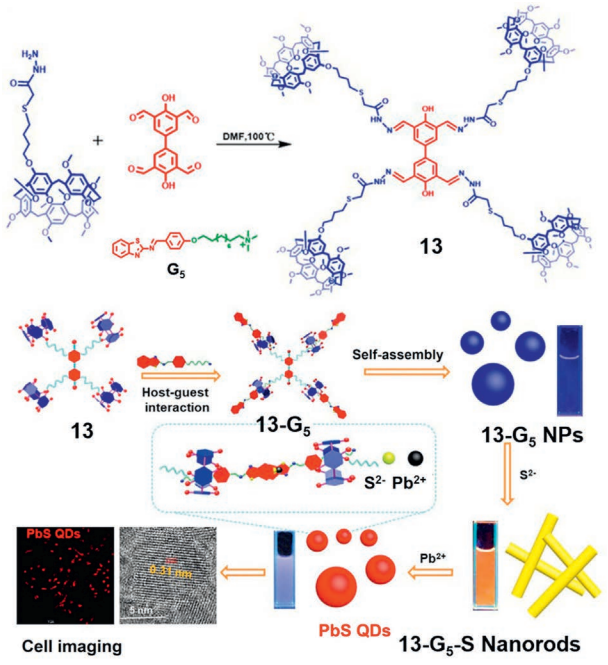


Fig. 13. Synthetic route of the tetra-pillar[5]arene macrocyclic compound **13** and schematic illustration of **13** and $\mathbf{G_5}$ self-assembling to form **13-G$_5$** NPs; subsequent addition of S^{2-} driven crosslinking to form **13-G$_5$-S** Nanorods; further introduction of Pb^{2+} leads to *in situ* generation of PbS quantum dots and their application in cellular imaging. Copied with permission [128]. Copyright 2022, American Chemical Society.

3. Application of multi-macrocyclic supramolecular functional materials

3.1. Recognition materials

The development of supramolecular chemistry promotes the study of functional molecules. As special molecular structures, macrocyclic compounds have attracted much attention due to their specific size and shape of cavities and host-guest recognition characteristics. Its cavity can interact with guest molecules to realize the detection of target molecules. Under the demand for high sensitivity and high selectivity sensors in the sensing field, the characteristics of multi-macrocyclic compounds provide the possibility to meet this demand [127].

In 2022, our group synthesized a novel tetra-pillar[5]arene macrocyclic compound **13**. Using an amphiphilic molecule $\mathbf{G_5}$ as the guest, host-guest interactions between **13** and G enabled their assembly into NPs **13-G$_5$** in a DMSO/H_2O mixed solution (Fig. 13) [128]. Subsequently, upon adding S^{2-} to the **13-G$_5$** supramolecular system, the NPs transformed into nanorods. Further introduction of Pb^{2+} into the system led to the *in-situ* formation of PbS quantum dots. After quantum dot formation, both fluorescence intensity and quantum yield significantly improved. This method of fluorescence enhancement *via in situ* generated PbS quantum dots enables the detection of Pb^{2+} in aqueous solutions and cells, with a detection limit of 4.52×10^{-7} mol/L. Therefore, this work provides an easy method for the construction of fluorescent nanomaterials based on a supramolecular system and a new approach for the *in situ* recognition of ions in cells by the *in situ* formation of nanomaterials such as QDs.

In early 2024, Cao *et al.* synthesized a 3D bi-macrocyclic compound **14** (Fig. 14) featuring dual hydrophobic cavities [129]. This architecturally unique host forms an exceptional 1:4 host-guest complex with d(GpC) dinucleotides through multivalent interactions. Therefore, this work not only shows a new artificial host system to capture biomimetic hydrogen-bonded G·C·G·C quadruplex for understanding the complex assembled substructure of DNA or RNA, but also offers a novel chiroptical sensor for monitoring and detecting different hydrogen-bonded nucleotide assemblies based on their different conformation chirality.

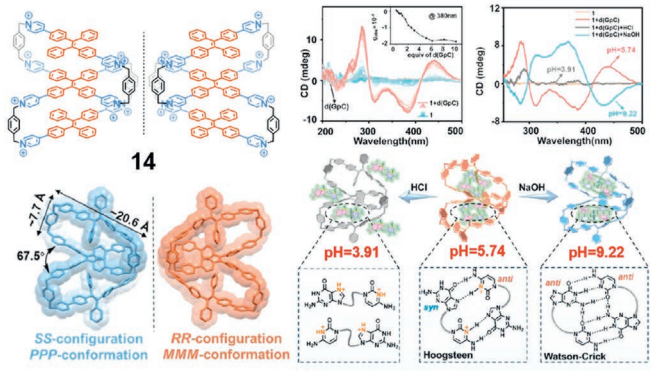


Fig. 14. Molecular architecture of bi-macrocycles **14** and its recognition mechanism for d(GpC) dinucleotides. Copied with permission [129]. Copyright 2024, WILEY-VCH.

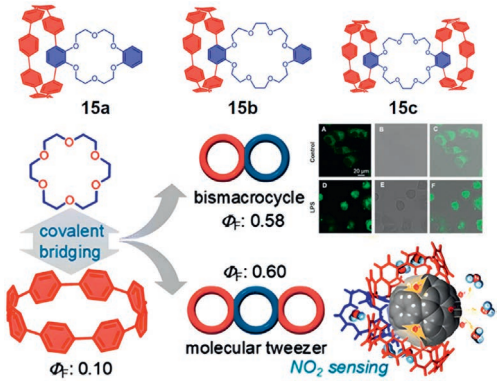


Fig. 15. Synthesis and multifunctional applications of crown ether-cycloparaphenylene hybrid multi-macrocyclic molecules **15a-c** in supramolecular sensing and biomedicine. Copied with permission [130]. Copyright 2025, Royal Society of Chemistry.

Very recently, Xu and co-workers successfully synthesized a series of crown ether-cycloparaphenylene bi-macrocyclic and tri-macrocyclic hybrid molecules **15a-c** (Fig. 15) *via* Suzuki-Miyaura cross-coupling reactions [130]. These molecules not only exhibit excellent photophysical properties but also demonstrate diverse host-guest interaction characteristics. The study revealed that the tri-macrocyclic hybrid molecules can form unique molecular tweezer-like structures, showing significantly enhanced binding affinity for fullerenes. The resulting complexes exhibit both high sensitivity and good cycling stability in NO_2 detection. Meanwhile, the bi-macrocyclic hybrid molecules display dual functionality: demonstrating significant cytotoxicity against cancer cell lines at low concentrations while also being capable of detecting inflammatory responses through fluorescence signals, highlighting their potential in biosensing applications. These important findings fully demonstrate the multifunctional nature of crown ether-cycloparaphenylene hybrid multi-macrocyclic molecules in supramolecular sensing and biomedical research, providing new research directions for environmental monitoring and disease diagnosis.

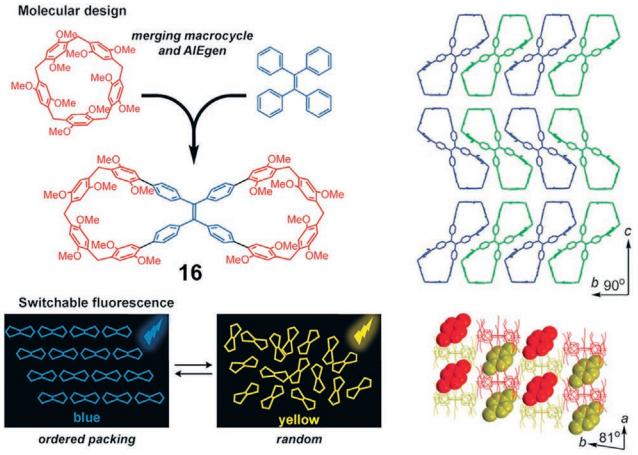


Fig. 16. Molecule single crystal structure, and fluorescence variation of the bow-tie aromatic hydrocarbon **16**. Copied with permission [135]. Copyright 2019, WILEY-VCH.

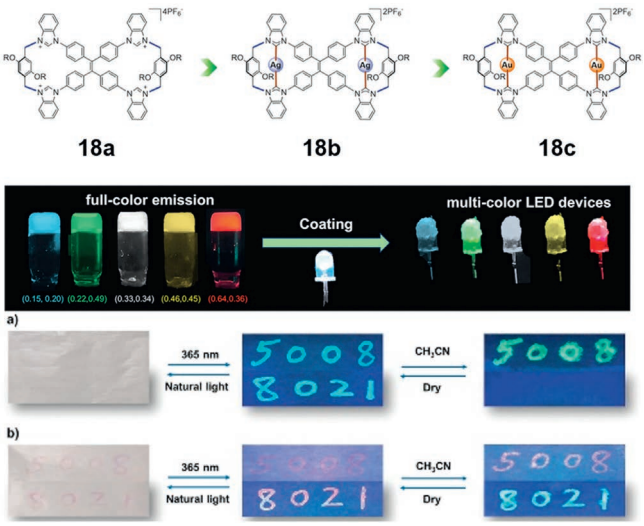


Fig. 18. Bi-macrocyclic molecule **18a** and bi-metallacycle **18b/18c**: molecular structures, full-color emission, and information anti-counterfeiting applications. Copied with permission [137]. Copyright 2024, WILEY-VCH.

3.2. Luminescent materials

With the advancement of science and technology, the demand for luminescent materials continues to grow across various fields, including displays, lighting, and bioimaging. Traditional materials, limited by their luminescent efficiency, stability, and color tunability, struggle to meet these diverse requirements [131,132]. Multi-macrocyclic compounds have emerged as a focal point in the development of novel luminescent materials due to their unique structural and electronic properties [133]. The progress in supramolecular chemistry has provided both theoretical and technical support for their design and synthesis, enabling the precise modulation of structures to achieve desired luminescent performance [134].

Later in 2019, Cong and co-workers reported a new type of fluorescent bi-macrocycles **16** (bowtie aromatics) based on pillar[5]arene (Fig. 16) [135]. It is worth noting that this molecular design will utilize the AIE properties of the TPE core and the supramolecular properties of the double electron-rich cavity. In addition, this new macrocycle also exhibits multi-stimuli-responsive convertible fluorescence from blue to yellow, which is closely related to the accumulation of molecules in the macrocycle. The work provides important design concepts and inspiration for the development of novel intelligent fluorescent materials, molecular sensors, and information encryption materials.

In 2020, the Stang team prepared a hexagonal Pt(II) metal multi-macrocycles **17** decorated with six-pillar[5]arene (Fig. 17) to provide a platform [136]. The AIE effect is used to confirm this trend. As mentioned above, it was found that the metal macrocyclic promoted the aggregation of the guest in the acetone/water mixture through the host-guest interaction based on the metal macrocycle. As a result, **17** showed strong fluorescence emission in acetone/water solution (5:95), which was 166 times higher than that in acetone. It is expected that these findings will provide a basis for the design and synthesis of host-based metallacycles/metallacages and may enable the development of a range of new host-containing materials.

Later in 2024, Tu *et al.* reported two nitrogen-containing carbometallacycles **18a-M** (M = Ag, Au), featuring TPE as the core (Fig. 18) [137]. This work utilized both covalent and coordination

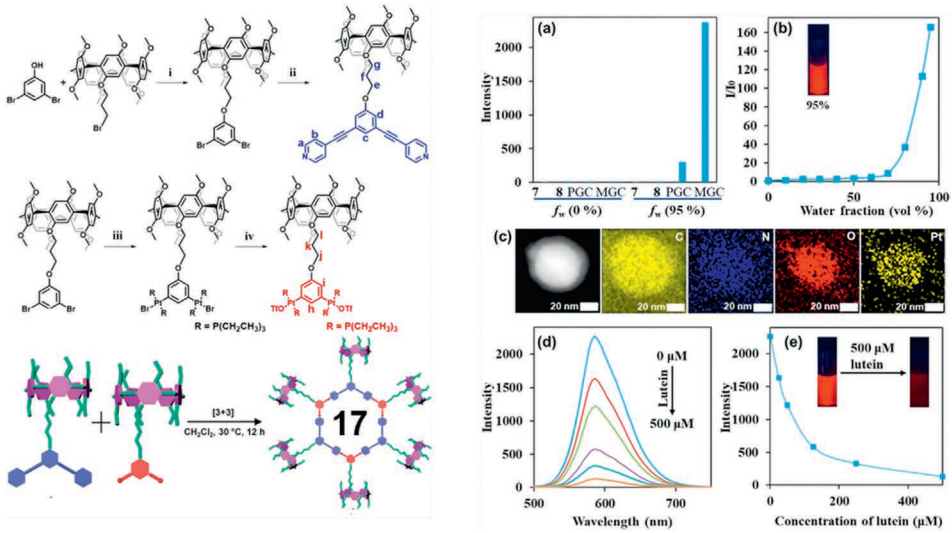


Fig. 17. The synthetic route of Pt(II) metal multi-macrocycles **17** and its fluorescence spectra in the acetone/water mixed system. Copied with permission [136]. Copyright 2020, American Chemical Society.

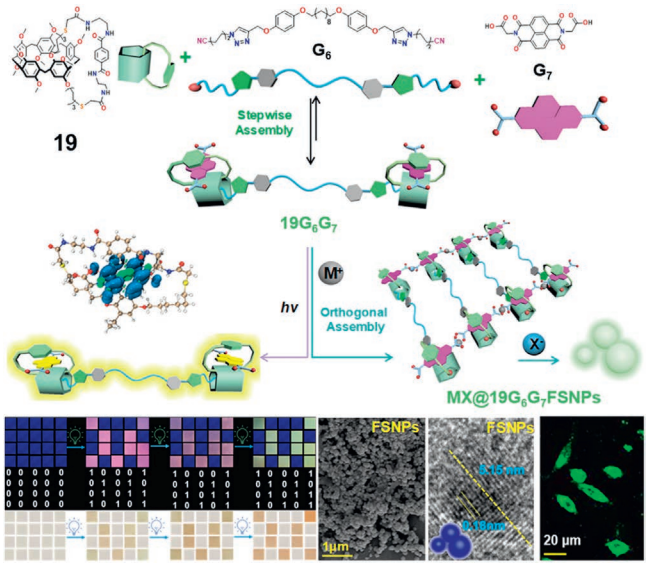


Fig. 19. Schematic illustration of the stepwise and orthogonal assembly between the fused bi-macrocyclic compound **19** and guest molecules **G6** and **G7** to construct multi-functional supramolecular NPs (FSNPs). Copied with permission [138]. Copyright 2025, Elsevier.

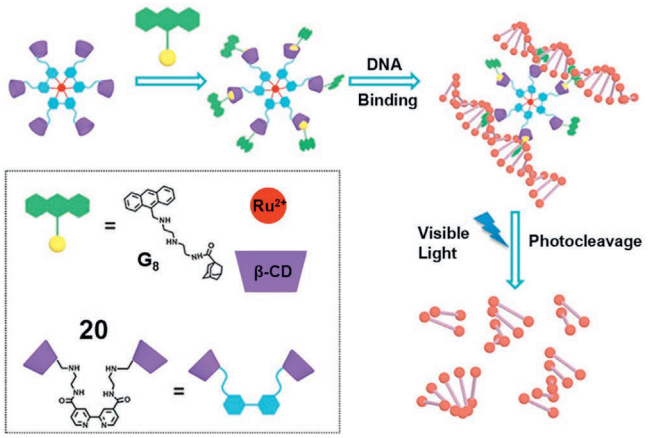


Fig. 20. Schematic illustration of a supramolecular polymer based on β-cyclodextrin bi-macrocycles **20** for light-driven cancer therapy. Copied with permission [146]. Copyright 2018, American Chemical Society.

bonds to restrict rotation within the bi-metallacyclic, thereby enhancing their quantum yields. By adjusting the CH_3CN/H_2O solvent ratio, the luminescence of **18b** could be tuned from blue to green. Further diversification of the emission spectrum, including high-quality white light (CIE coordinates 0.33, 0.34), was achieved through fluorescence resonance energy transfer (FRET). The compatibility of **18b** with agarose gel enabled its application in information encryption and anti-counterfeiting. These results not only demonstrate the feasibility of achieving full-color emission using a dual-fluorophore system but also highlight its potential applications in information encryption, data storage, and anti-counterfeiting technologies.

Very recently, our group designed and synthesized a novel fused bi-macrocyclic compound **19**, and proposed a stepwise orthogonal assembly strategy based on this compound for constructing multifunctional supramolecular materials (Fig. 19) [138]. Compound **19** possesses two cavities with distinct sizes and electronic structures, enabling selective recognition of the cyanoalkyl chain-containing guest $\mathbf{G_6}$ and the naphthalene diimide (NDI)-functionalized guest $\mathbf{G_7}$, respectively, forming a stable $\mathbf{19G_6G_7}$ supramolecular system. This assembly significantly enhances the luminescent performance of the light-induced NDI radical through steric hindrance and encapsulation effects, increasing its quantum yield to three times that of $\mathbf{G_7}$ alone, and demonstrating excellent time-dependent fluorescence properties suitable for advanced information encryption. Furthermore, $\mathbf{19G_6G_7}$ can undergo further orthogonal assembly with metal ions such as Ag^+, Pb^{2+}, and Zn^{2+}, as well as anions like I^- and S^{2-}, to construct a series of fluorescent supramolecular NPs (FSNPs). These NPs exhibit excellent biocompatibility and remarkable fluorescence properties, and have been successfully applied in cell imaging, demonstrating great potential in biomedical detection and imaging.

3.3. Biomedical materials

Macrocyclic supramolecular functional materials have emerged as "star materials" in the biomedical field due to their designable molecular recognition capabilities, dynamic self-assembly properties, and excellent biocompatibility. They demonstrate unique advantages in drug delivery, antibacterial/antiviral applications, biosensing, tissue engineering, and other areas, providing innovative solutions to overcome the limitations of traditional therapies [139–145].

In 2018, Liu and co-workers successfully constructed a bi-(β-cyclodextrin)macrocyclic compound **20** (Fig. 20) using bipyridine as the bridging unit [146]. Studies revealed that this bi-cyclodextrin hosts molecule could form a 3:1 coordination complex with a Ru(II) metal center. Furthermore, in an aqueous system, this complex efficiently assembled into a supramolecular architecture through host-guest interactions with an anthracene derivative functionalized by an adamantane group $\mathbf{G_8}$. Notably, this supramolecular system exhibited remarkable photo-responsive properties under visible light excitation: it could specifically accumulate in the nuclei of cancer cells and induce the highly efficient generation of reactive oxygen species (ROS), thereby demonstrating excellent antitumor activity. The work, based on the host-guest assembly strategy between a β-cyclodextrin-based bi-macrocyclic host and an anthracene-derived guest, provided an innovative approach for light-driven cancer therapy.

Later in 2022, Guo's team designed and synthesized a sulfonate-modified azocalix[4]arene dimer **21** (Fig. 21) [147]. The **21** can not only bind functional guest molecules for the preparation of host-responsive supramolecular polymers but also accommodate stimuli-responsive guests, thereby enabling the facile construction of multi-stimuli-responsive supramolecular polymers. As proof of concept, the team employed hypoxia-responsive **21** as the hosts molecule and a glutathione (GSH)-responsive camptothecin dimer ($\mathbf{G_9}$) linked by disulfide bonds as the guest molecule. When mixed at a 1:1 molar ratio in water, these components spontaneously assembled into a unique host-guest dual-responsive supramolecular polymer. The polymer chains further self-assembled into NPs (DSPNs), which were subsequently applied for tumor therapy. This work provides a general new method for the construction of 'host-responsive' supramolecular polymers, which can also be extended to other functional hosts.

3.4. Catalytic materials

Multi-macrocyclic hosts molecular catalysts represent a class of structurally defined macrocyclic compounds that employ host-guest interactions, including hydrogen bonding, van der Waals forces, and π-π stacking, to selectively bind substrates or metal ions. This specific molecular recognition enables precise modulation of catalytic reactivity and selectivity, offering unique advan-

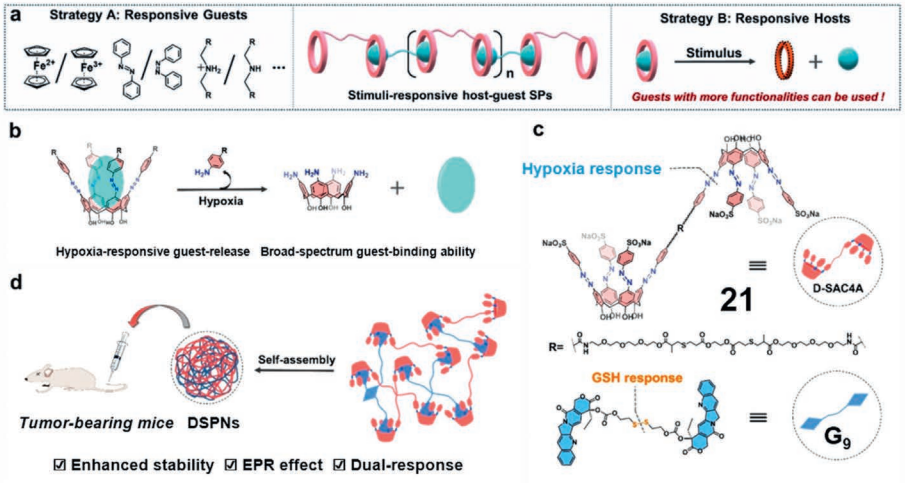


Fig. 21. Chemical structure of bi-macrocycles **21** and schematic design of host-guest dual-stimuli-responsive supramolecular polymer. Copied with permission [147]. Copyright 2022, WILEY-VCH.

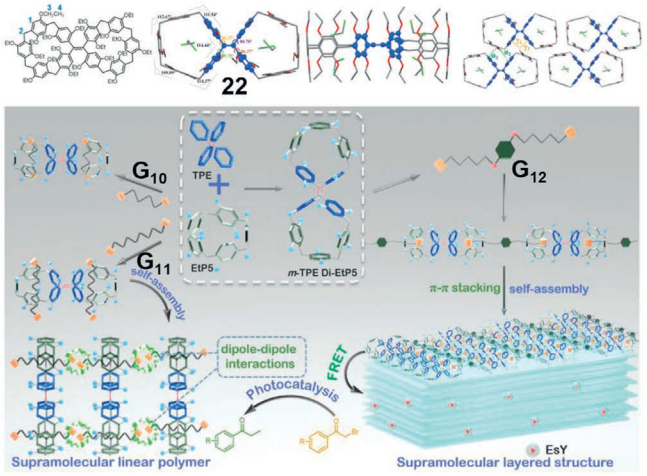


Fig. 22. The molecular and single crystal structures of bi-pillar[5]arene **22** and the schematic diagram of supramolecular polymers constructed with different guests. Copied with permission [149]. Copyright 2023, WILEY-VCH.

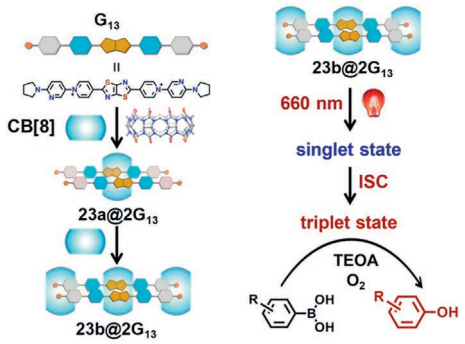


Fig. 23. The two-step host-guest complexation reaction of $\mathbf{G_{13}}$ with CB[8] and the photocatalytic oxidation of boric acid under red light (660 nm) irradiation. Copied with permission [150]. Copyright 2024, Royal Society of Chemistry.

tages in stereochemical control and reaction pathway regulation [148].

In early 2023, Hu *et al.* connected bi-pillar[5]arene by C–C double bond to obtain a bi-pillar[5]arene **22** containing a TPE unit (Fig. 22) [149]. Host-guest studies have shown that guest molecules, adiponitrile (**G10**) or sebaconitrile (**G11**), can enter the pillar[5]arene cavity pseudorotaxane structure with **22** in a ratio of 2:2, and **22** and **G11** can form a linear supramolecular polymer. In addition, **22** forms a supramolecular layered polymer with a long chain dinitrile guest molecule containing a phenyl group (**G12**), and the polymer can be used as a photocatalyst to catalyze the dehalogenation reaction. The work successfully combines fluorescence properties with host-guest chemistry through ingenious molecular design and fine supramolecular regulation, and realizes multi-level functional integration from molecular recognition to material construction to photocatalytic application. It is an important milestone in the development of supramolecular chemistry to functionalization and systematization.

In 2024, Zhang and co-workers developed a red-light supramolecular photocatalytic system by preparing cucurbit[8]uril-mediated mono-macrocyclic $\mathbf{23a@2G_{13}}$ and bi-macrocyclic complex $\mathbf{23b@2G_{13}}$ (Fig. 23) [150]. The long-axis molecule 4,4′-(thiazole[5,4-*d*]thiazole-2,5-diyl)bi-(6(pyrrolidine-1-yl)-[1,3-bipyridyl]−1-ium)chloride ($\mathbf{G_{13}}$) can undergo two-step host-guest complexation with cucurbit[8]uril (CB[8]) to form two host-guest complexes $\mathbf{23a@2G_{13}}$ and $\mathbf{23b@2G_{13}}$ in turn. The formation of $\mathbf{23a@2G_{13}}$ and $\mathbf{23b@2G_{13}}$ leads to increasing intermolecular interaction between TPPs. Finally, $\mathbf{23b@2G_{13}}$ exhibits strong red light absorption capacity and long-lived triplet state. In addition, the electron transfer and energy transfer between $\mathbf{23b@2G_{13}}$ and oxygen are relatively smooth, so $\mathbf{23b@2G_{13}}$ can be used as an efficient red-light catalyst for the oxidation of boric acid. In this work, the host-guest complexation was used to achieve precise regulation of intermolecular interactions, thereby creating a novel red-light responsive photocatalyst with a long-lived excited state.

3.5. Chiral materials

Multi-macrocyclic hosts molecular chiral materials represent a class of intelligent functional materials that integrate the cavity recognition advantages of macrocyclic structures with the selective properties of chiral characteristic molecules. The core design strategy thereof resides in modulating the intrinsic chirality, induced chirality, or supramolecular chirality of macrocycle, thereby achieving highly selective recognition of chiral enantiomers and function-oriented transformation [151–153].

In 2021, Yang's group reported a case of temperature-overprotection functionalized molecular universal joint **24a–c** based on pillar[*n*]arene (Fig. 24) [154]. The strategy involved inte-

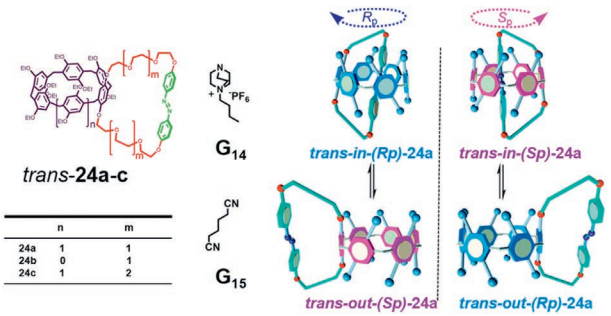


Fig. 24. Molecular structure of the pillar[n]arene-based bi-macrocyclic pseudorotaxane and schematic diagram of its chiral inversion. Copied with permission [154]. Copyright 2021, Springer Nature.

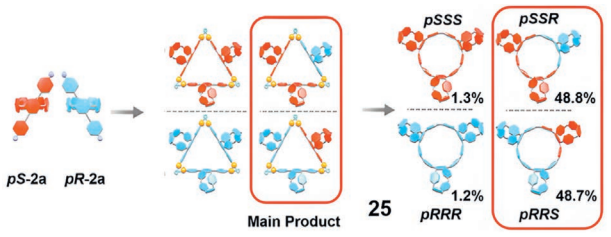


Fig. 25. Schematic representation of the social chiral self-sorting synthesis of *pSSR*- and *pRRS*-**25**. Copied with permission [155]. Copyright 2025, WILEY-VCH.

grating thermally responsive and photo-responsive functionalities into a pillar[*n*]arene-based bicyclic pseudorotaxane. The *cis/trans* photoisomerization of the azobenzene unit in **24a** could induce an in/out conformational switching of the azobenzene-bearing side ring, resulting in the inversion of planar chirality in **24a**. Simultaneously, temperature variations also triggered conformational chirality inversion due to significant entropy changes during the ring-flipping process. Consequently, when the temperature exceeded the upper threshold, the photo-induced switching could be inhibited, demonstrating an intelligent molecular photoswitch with temperature-overprotection functionality. This contrasts sharply with the commonly observed low-temperature gating effect. This study achieved the challenging high-temperature gating effect at the molecular level, marking a significant step toward constructing intelligent molecular devices capable of performing complex functions.

Very recently, Huang and co-workers propose an innovative social chirality self-sorting strategy to address stereoselectivity challenges in synthesizing multi-cavity pillararene macrocycle. Using triangular gold complexes, they achieve stereoselective synthesis of [9]cycloparaphenylene-pillar[5]arene trimers **25**, overcoming traditional statistical mixtures *via* gold-bond reorganization and intermolecular forces (Fig. 25) [155]. The resulting materials exhibit excellent CPL properties and enable construction of supramolecular polymer networks with nanofiber morphology and high luminescence retention. This work not only solves a major problem in the synthesis of multi-macrocycles, but also provides new ideas and methods for the development of chiral functional materials.

4. Conclusion

Multi-macrocyclic hosts molecules have emerged as a versatile platform for constructing functional supramolecular systems benefited from their distinctive multi-cavity architectures, multiple recognition sites, and multi-dimensionality assembly properties. Recent advances in supramolecular materials based on these hosts have demonstrated significant breakthroughs across diverse domains, including molecular recognition, programmable self-assembly, chiral materials, stimuli-responsive systems, catalytic transformations, and biomedical applications, highlighting their considerable potential for addressing contemporary challenges in supramolecular and material chemistry. Despite these achievements, there are still many challenges in this field. (1) How to design multi-macrocyclic hosts compounds to precisely recognize sophisticated target guest. (2) How to accurately realize the multi-component collaborative assembly to construct high performance supramolecular materials. (3) How to realize specific applications based on these high-performance multi-macrocyclic supramolecular systems by targeting areas such as molecular recognition, functional optical materials, chiral and biomedical materials, thus harnessing their potential and generating tangible value. In summary, compared to mono-macrocyclic, multi-macrocyclic supramolecular materials offer us a diverse range of functional systems with satisfactory properties. It can be anticipated that multi-macrocyclic supramolecular materials hold unlimited promise in the near future. With the multi-macrocyclic systems, more unprecedented functionalities and applications will definitely emerge under the incessant effort contributed by researchers.

Declaration of competing interest

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

CRediT authorship contribution statement

Xing Han: Writing – original draft, Investigation. **Xinsheng Lu:** Writing – review & editing. **Qi Lin:** Writing – review & editing, Supervision, Resources, Funding acquisition, Conceptualization.

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