Pillar[n]arene–calix[m]arene hybrid macrocyclic structures

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To reserve planar chirality, enhance molecular recognition, and build advanced self-assemblies, hybrid macrocyclic hosts containing rigid pillar[n]arene and flexible calix[m]arene were designed, prepared and investigated for interesting applications. This review summarizes and discusses different synthetic strategies for constructing hybrid macrocyclic structures. Pillar[n]arene dimer with rigid aromatic double bridges provided the possibility of introducing calix[m]arene cavities, where the planar chirality was reserved in the structure of pillararene. The capacity for molecular recognition was enhanced by hybrid macrocyclic cavities. Interestingly, the obtained pillar[n]arene–calix[m]arene could self-assemble into “channels” and “honeycomb” in both the solid state and solution phase as well as donate the molecular architecture as the wheel for the formation of mechanically interlocked molecules, such as rotaxane. In addition, the pillar[n]arene and calix[m]arene could also be coupled together to produce pillar[n]arene embedded 1,3-alternate and cone conformational calix[m]arene derivatives, which could catalyze the oxidative polymerization of aniline in aqueous solutions. Except for building hybrid cyclophanes by covalent bonds, weak supramolecular interactions were used to prepare pillar[n]arene–calix[m]arene analogous composites with other pillar-like pillar[n]pyridiniums and calix-like calix[m]pyrroles, exhibiting reasonable performances in enhancing molecular recognition and trapping solvent molecules.

1 Introduction

To mimic natural and biological processes,1–3 such as protein–glucose interactions,4–8 macrocycles9–11 were designed and prepared as specialized host molecules12,13 for molecular recognition14 by weak supramolecular interactions.15 To date, various macrocycles16–18 with various physiochemical characteristics have been reported. Among them, cyclophanes19–23 with repeated aromatic moieties, such as pillar[n]arene22–28 and calix [m]arene29–34 (Scheme 1), have attracted considerable academic and practical attention.25 For example, owing to the presence of multivalent modification sites,26 the structural skeletons of both pillar[n]arene and calix[m]arene could be directly used as the backbone for embedding valuable functional groups for various applications to water-soluble37 self-assembled amphiphiles,38–41
advanced hierarchical architectures,\textsuperscript{45,43} such as metal organic frameworks,\textsuperscript{44–48} as well as energy storage and batteries.\textsuperscript{49,50}

Actually, owing to the presence of similar aromatic oligomeric structures, the development of novel structures and applications of pillar[n]arene\textsuperscript{51} has always resembled those of calix[m]arene.\textsuperscript{52} However, because of different substitution sites on aromatic rings, pillar[n]arene and calix[m]arene\textsuperscript{53} always exhibit different topologies and morphologies in molecular conformation and configuration,\textsuperscript{34} as well as diverse behaviors in particular physiochemical characteristics.\textsuperscript{55} For example, from the molecular geometry and shape perspective, the flexible structures of calix[m]arene could result in an adaptable cavity for diverse guests,\textsuperscript{56} while the rigid conformational structures of pillar[n]arene can produce unique planar chirality.\textsuperscript{57}

It is important to compare these two attractive analogues of aromatic macrocycles in terms of their structural and physiochemical behaviors.\textsuperscript{58} Several investigation methods have been developed for this purpose; for example, computational studies\textsuperscript{59} such as density functional theory (DFT)\textsuperscript{60} have been employed in investigating the diverse physiochemical properties of pillar[n]arene and calix[m]arene for different performances and applications, such as host–guest complexation.\textsuperscript{60} Furthermore, a direct comparison between these two cyclophanes in practical experiments was also carried out.\textsuperscript{61,62} Pillar[n]arene and calix[m]arene demonstrated their advantages. Thus, it aroused another academic interest in combining the molecular structure and characteristics of pillar[n]arene and calix[m]arene with the art of organic synthesis.\textsuperscript{63} The integration of pillar[n]arene and calix[m]arene into hybrid host molecules not only enriches the family of hybrid macrocycles but also

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combines their features, such as flexible architectures and planar chirality.\textsuperscript{64-67}

In this review, the preparation and application of pillar[n]arene–calix[m]arene hybrid macrocyclic compounds are summarized and discussed. Different classic organic synthesis methods\textsuperscript{68} were employed during the preparation, producing pillar[n]arene dimer with aromatic double bridges, coupled with pillar[n]arene–calix[m]arene hybrid hosts, and noncovalent

**Fig. 1** Single crystal structures of P2 in stick representation indicate (a) a column-like tubular supramolecular self-assembly with (b) noncovalent interactions, as highlighted in green, as well as (c) honeycombed self-assembly owing to the possession of $\pi-\pi$ stacking interactions and C–H–\(\cdots\)\(\pi\) hydrogen bonds, where hydrogen atoms were omitted for clarity.\textsuperscript{61}

**Scheme 3** Two synthesis routes from the starting molecule P4 with the assistance of F1 to prepare diverse oxacalix[4]arene-bridged pillar[5]arene dimers, such as PC1, PC2 and PC3, with different planar chirality. P5 is an important intermediate compound in one synthesis strategy.\textsuperscript{65}
interaction-based pillar[\(n\)]arene-calix[\(m\)]arene resembling systems. Pillar[\(n\)]arene dimer with rigid aromatic double bridges possessing resembled calix[\(m\)]arene cavities could reserve the planar chirality and include dumbbell guests\(^{69,70}\) for fabricating mechanically interlocked molecules.\(^{71,72}\) In addition, the pillar[\(n\)]arene and calix[\(m\)]arene could also be covalently coupled to produce pillar[\(n\)]arene-containing 1,3-alternate and cone conformational calix[\(m\)]arene derivatives for catalyzing the oxidative polymerization of aniline.\(^{73}\) Furthermore, weak supramolecular interactions have been used to prepare pillar[\(n\)]arene–calix[\(m\)]arene analogous composites using pillar[\(n\)]pyridiniums\(^{74}\)/calixarene and calix[\(m\)]pyrrole\(^{75,76}\)/pillararene to enhance molecular recognition and trapping solvent molecules.\(^{77}\) Finally, we consider possible scientific and technical issues and new challenges, as well as provide brief and primary suggestions for future studies to be conducted on pillar[\(n\)]arene–calix[\(m\)]arene hybrid macrocycles.

2 Preparation of pillar[\(n\)]arene–calix[\(m\)]arene hybrid macrocyclic structures

2.1 Merging structures of pillar[\(n\)]arene and calix[\(m\)]arene

Based on the art of organic synthesis,\(^{78}\) both the direct modification of pillar[\(n\)]arene with calix[\(m\)]arene and the
functionalization of calix[m]arene with pillar[n]arene were challenging because of several organic experimental issues, such as the diverse solubilities of the two cyclophanes in reaction solutions,\(^7\) as well as the multivalent reactive sites\(^8\) embedded in their structures. Thus, the current rigid skeleton of pillar[n]arene was initially considered the structural basis, and heteroatoms, such as oxygen-atom on the hydroquinone moieties of pillar[n]arene, were further used as a bridge to incorporate extra aromatic subunits via meta positions, producing an additional calix-like cyclophane cavity. Such hybrid macrocycles have particular structural characteristics, e.g., “fused” hybrid macrocyclic cavities “double-bridged”\(^9\) by aromatic rigid linkers with unique planar chirality.\(^{92}\) Furthermore, it provides the opportunity to raise another supramolecular issue, i.e., the fabrication of mechanically interlocked molecules.\(^{92}\)

For example,\(^{93}\) 1,4-dimethoxypillar[4]arene[1]hydroquinone (P1) (Scheme 2) was used as the previous piece to react with the “bridging group”—1,5-difluoro-2,4-dinitrobenzene (F1) (Scheme 2) in the presence of triethylamine as the catalyst in THF solutions under N\(_2\) atmosphere to produce 2,4-dinitro-5-fluorophenyl difunctionalized pillar[5]arene (P2) (Scheme 2) using the classic nucleophilic aromatic substitution (S\(_{\text{NAr}}\)) yielding 90%. Then, P2 was dissolved in DMF to promote its solubility under K\(_2\)CO\(_3\) condition and prepare another nitrophenyl difunctionalized pillar[5]arene (P3) (Scheme 2), yielding 99%. Interestingly, the vapor diffusion method was applied to synthesize the single crystal structures of P2 and P3 using hexane, heptane and dichloromethane, revealing that tubular and honeycombed self-assemblies\(^{94}\) were formed in the solid state with solvent molecules encapsulated inside these structures (Fig. 1).\(^{93}\)

Considering the above organic experiment of preparing significant previous pieces of conformationally locked A1/A2 difunctionalized pillar[5]arene (P2 and P3) (Scheme 2), the pillar[5]arene/calix[4]arene-containing fused hybrid macrocyclic compound—oxacalix[4]arene-bridged pillar[5]arene dimers (PC1–PC3) with diverse planar chirality (Scheme 3) were further obtained using two different synthesis strategies, such as the
one-pot reaction between pillar\([4]\)arene\([1]\)hydroquinone \(P_4\) and \(F_1\) (Scheme 3) in DMSO in the presence of \(K_2CO_3\) at room temperature and two-step approach with \(P_5\) (Scheme 3) as the intermediate compound in relatively improved yields. Interestingly, \(PC_1\) and \(PC_2\) were a pair of enantiomers, as confirmed by chiral high performance liquid chromatography (HPLC), and \(PC_3\) (Fig. 2) was the achiral \(meso\) compound of \(PC_1/PC_2\) as revealed by \(^1\)H NMR and electrospray ionization high resolution mass spectrometry (ESI-HRMS).

Fused hybrid macrocyclic hosts are good candidates for preparing advanced molecular architectures, such as mechanically interlocked molecules. Conformationally fixed double-bridged pillar\([5]\)arene–calix\([4]\)arene hybrid structures were further used as the “wheel” subunit for constructing planar chirality-containing \([2]\)rotaxanes, such as \(PC_4, PC_5, PC_8\) and \(PC_9\) (Scheme 4), as well as \([3]\)rotaxanes, such as \(PC_6, PC_7\) and \(PC_10\) (Scheme 4), using the “stopper” moieties‐possessing functional molecules \(F_2\) and \(F_3\) (Scheme 4) to form the “axle” subunit via the threading/stoppering method (Fig. 3).

2.2 Coupling independent structures of pillar\([n]\)arene and calix\([m]\)arene

Except for expanding the current structural skeleton of pillar\([n]\)arene to build extra calix-like cavities, two independent structures of pillar\([n]\)arene and calix\([m]\)arene can be coupled via classic organic reactions. For example, tetraacyl chlorides-

Scheme 5  Synthesis route for producing \(PC_{11}\) and \(PC_{12}\) by coupling thiacalix\([4]\)arene \(C_1\) and pillar\([5]\)arene \(P_6\).

Scheme 6  Synthesis of \(PC_{13}\) using \(P_7\) and \(C_2\).
containing \( p \)-tert-butylthiacalix[4]arene \( C1 \) (Scheme 5) could be integrated with amino monofunctionalized pillar[5]arene derivative \( P6 \) (Scheme 5) in dichloromethane in the presence of Hüngel's base under the argon atmosphere through acylation reaction, preparing pillar[5]arene–calix[4]arene hybrid macrocycles, such as 1,3-alternate conformational calixarene-containing \( PC11 \) (Scheme 5) and cone conformational calixarene-containing \( PC12 \) (Scheme 5), yielding 58% and 65%, respectively. Interestingly, the combination of pillar[5]arene and thiacalix[4]arene into a hybrid macrocyclic system not only allowed the integration of the diverse capacities of molecular recognition towards valuable guest molecules, such as aniline and aniline/\( p \)-toluenesulfonic acid, but also promoted the oxidative polymerization of aniline in aqueous \( p \)-toluenesulfonic acid solutions by increasing the yield and molecular weight of emeraldine.

2.3 Integrating pillar-like and calix-like aromatic cyclic oligomers

In addition to the direct coupling of pillar[\( n \)]arene and calix[\( m \)]arene into hybrid macrocyclic structures, other pillar-like and calix-like macrocycles, such as heteroatom-containing ones, were employed to resemble fused cyclophanes. Owing to the introduction of heteroatoms, the capacity for molecular recognition generated from fused macrocycles was enhanced.

For example, carboxylate\(^{89,90} \) difunctionalized pillar[5]arene \( P7 \) (Scheme 6) was reacted with the calix-like nitrogen-rich macrocycle—calix[4]pyrrole \( C2 \) (Scheme 6) via an esterification reaction in the presence of catalytic amounts of 1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride.
(EDCI) and 4-dimethylaminopyridine (DMAP), leading to the formation of pillar[5]arene–calix[4]pyrrole PC13 (Scheme 6) with two calix[4]pyrrole subunits on both ends yielding 50%. Owing to the possession of nitrogen-rich calix[4]pyrrole structures and conformational fixed pillar[5]arene moieties, PC13 exhibited enhanced inclusion ability for chiral anion salts, such as chiral (R/S)-mandelate anionic guests as tetramethylammonium salts, by a series of weak supramolecular interactions, including hydrogen bonding, cation–π, π–π stacking and C–H⋯π interactions. The association constants (Kₐ) were determined in the range of 5.15 × 10⁴ to 2.25 × 10⁵ M⁻¹.

Particularly, the introduction of heteroatoms embedded in the macrocyclic structures provided the possibility of combining pillar-like and calix-like cyclic aromatic oligomers via noncovalent interactions, i.e., the heteroatom-containing macrocycles acting as the guest molecule to be included by the other cavity via supramolecular interactions, such as electrostatic forces. For example, cationic pillar[n]pyridiniums, such as the rigid square-shaped tetramer P8 and the flexible roughly hexagonal hexamer P9 (Scheme 7), were encapsulated by anionic p-sulfonatocalix[4]arene C3 (Scheme 7) in the solid state and aqueous solution phase by electrostatic attractions, and anion⋯π⁺, anion⋯π⁺⋯anion and π⋯π⁺ interactions, leading to the co-assemblies of oppositely charged hybrid macrocycles (Fig. 4 and 5).

### 3 Overview and outlook

In conclusion, the design, preparation and application of pillar [n]arene–calix[m]arene hybrid macrocyclic structures were discussed and summarized in this review. To overcome the challenges of the art of organic synthesis, various strategies were employed to fabricate hybrid cyclophanes. First, the structural skeletons of pillar[n]arene and calix[m]arene were fused via the formation of a pillararene dimer with double aromatic bridges by incorporating the architectures of calixarene under the S₈Ar reaction conditions, leading to the self-assembly of hybrid macrocycles in the solid and solution phases, the reservation of planar chirality of pillararene, and the enhancement of molecular recognition by hybrid cavities. Interestingly, double-
bridged pillar[n]arene dimers further provided the pillar[n]arene cavity as the wheel to integrate with “dumb-bell”-like linear molecules, affording mechanically interlocked molecules, such as [2][3]rotaxanes, with planar chirality. Additionally, classic organic synthetic methods, such as acylation reaction, were further carried out to directly combine independent units of pillar[n]arene and calix[m]arene, producing two types of pillar[5]arene–calix[4]arene hybrid macrocycles with 1,3-alternate and cone conformations, which were observed to have the capacity to catalyze the oxidative polymerization of aniline in aqueous solutions. Furthermore, pillar-like cyclophanes, such as pillar[n]pyridiniums, and calix-like cyclophanes, such as calix[m]pyrrole, were also used for the fabrication of hybrid macrocyclic systems with calixarene and pillarene, respectively, to resemble pillar[n]arene–calix[m]arene composites, where both covalent bonds and noncovalent interactions acted as the driving forces. Particularly, these analogous systems exhibited reasonable performances in enhancing molecular recognition and trapping solvent molecules in the solution and solid phases.

Numerous studies on the fabrication and application of pillarene–calixarene hybrid macrocycles are still attractive for future researchers, needing attention from related scientists in synthesis chemistry, physical chemistry and material science.

First, the efficient integration of pillarene and calixarene together as well as the effective separation of enantiopure hybrid macrocycles are still challenges in organic synthesis chemistry. In particular, owing to the possession of pillarene and the introduction of larger functional groups, the planar chirality of pillarene granted future applications for hybrid macrocycles but made the separation and purification of such compounds more difficult.

Second, other sized pillare and calix are macrocycles should be employed in the fabrication of such hybrid cyclophanes to enrich the family of pillarene/calixarene-based fused cyclophanes. Larger cavities of fused hybrid macrocycles not only enlarge the possibilities of complexing diverse functional guest molecules, but also provide significant backbones and building blocks for constructing advanced functional structures, such as self-assemblies and mechanically interlocked molecules. Future applications should be further explored based on functional architectures with covalent bonds, mechanical forces and supramolecular interactions.

Third, a computational study is highly recommended for proposing and investigating pillarene–calixarene hybrid macrocycle-based supramolecular materials, such as the study of different behaviors of host-guest inclusions, as well as the competition and cooperation between the two hybrid macrocycles.

### Abbreviations

| Abbreviation | Description |
|-------------|-------------|
| EDCI | 1-(3-Dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride |
| ESI-HRMS | Electrospray ionization high resolution mass spectrometry |
| DMAP | 4-Dimethylaminopyridine |
| HPLC | High performance liquid chromatography |
| $K_a$ | Association constants |
| $SNAr$ | Nucleophilic aromatic substitution |

### Conflicts of interest

There are no conflicts to declare.

### Acknowledgements

H. Z. and Z. L. would like to thank Dr Nathan L. Strutt for his kind help and patient guide to pillarene chemistry during their academic visit to Northwestern University. H. Z. and Z. L. acknowledge the financial support from the “Young Talent Support Plan” (No. 050700-71240000000046) of Xi’an Jiaotong University, Xi’an Peihua University, Natural Science Foundation of Shaanxi Province (No. 2021JM-006 and 2021JQ-863).

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