Review

Dynamic materials fabricated from water soluble pillar[n]arenes bearing triethylene oxide groups

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ABSTRACT

Pillar[n]arenes are a new kind of supramolecular macrocyclic hosts which have developed rapidly due to their unique topology and high functionality, giving rise to many applications in the construction of interesting and functional materials. Among them, water-soluble pillar[n]arenes bearing triethylene oxide (TEO) chains have drawn increasing research interest due to their advantageous properties. In this review, we summarized the recent progress of dynamic materials fabricated from water soluble pillar[n]arenes bearing TEO groups, including thermoresponsive materials with lower critical solution temperature (LCST) behavior, cyclic host liquids, and smart windows. It is anticipated that more and more 'smart' supramolecular materials based on modified pillar[n]arenes will be developed in this burgeoning area of research.

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1. Introduction

Host–guest interactions based on a macrocyclic host and a guest molecule have played an important role in supramolecular chemistry due to their various dynamic properties [1–6]. Incorporation of host–guest interactions can endow materials with interesting properties, such as stimuli responsiveness, self-healing, and adaptability. Macrocyclic host molecules, such as crown ethers, calixarenes, cyclodextrins and cucurbiturils, are typical host molecules which are widely used to achieve so-called 'smart' materials [7,8]. Pillar[n]arenes, as a new kind of macrocyclic host in supramolecular chemistry, have developed rapidly and attracted much attention in creating various supramolecular assemblies with application as supramolecular materials [9–18]. In contrast to typical host molecules, pillar[n]arenes display many useful features. Where this highly symmetrical pillar shaped architecture display similarities to the pumpkin–shaped cucurbiturils [19,20], the repeating units of pillar[n]arenes are also similar to calixarenes [21–23]. Furthermore, pillar[n]arenes have numerous phenolic oxygen units on both upper and lower rims, making

them analogous to the highly functionalized cyclodextrins [24–26]. Pillar[n]arenes are easy to prepare, facile to modify, possess a unique shape and display solubility in a range of solvents – many of the properties required from a desirable macrocyclic host. In particular, their symmetrical pillar structure and convenient modification endows them with the ability to bind a diverse range of guest molecules – neutral guests, anionic guests, cationic guests, rigid guests, flexible chain guests, hydrophobic guests and hydrophilic guests can all be accommodated with pillar[n]arenes [11,27].

Stimuli-responsive materials that can respond to external stimuli, such as temperature, light, redox reactions and pH, have also received much attention in the field of smart materials due to their various practical applications. Among these materials, water-soluble thermoresponsive materials exhibiting lower critical solution temperature (LCST) behavior are some of the most fascinating materials due to their important potential biomedical applications. At temperatures higher than the LCST, these materials display decreased solubility, leading to a fall in solution transmittance and an observable cloud point. Generally, most investigations on LCST phase behavior are focused on polymers, such as poly(*N*-isopropylacrylamide), however, in recent years LCST behavior has gradually been reported for host–guest systems, focusing mainly on pillar[n]arene systems. Compared with polymers, these pillar[n]arene based materials not only possess LCST behavior but

also show additional functions, such as controllable host–guest behavior. Interestingly, pillar[n]arene derivatives bearing triethylene oxide (TEO) chains exhibit LCST behavior in aqueous solution, which seems to be a common feature of LCST behavior of ethylene oxide dendronized polymers [28]. In contrast, pillar[n]arenes with short ethylene oxide chains have been found to be insoluble in water. Moreover, LCST behavior is not observed for model compounds without the pillar[n]arene backbone, suggesting that the pillar-shaped hydrophobic macrocyclic architecture containing a number of hydrophilic TEO groups is required for LCST behavior [29]. It is also worth noting that the ionic nature of some water-soluble pillar[n]arenes can affect electrolyte behavior [30,31], but those pillar[n]arenes bearing TEO chains are non-ionic and retain their water-solubility overcoming any such limitations to responsive LCST behavior. In addition, pillar[n]arenes bearing TEO chains exhibit many other interesting properties. For example, they can serve as cyclic host liquids for highly efficient construction of mechanically interlocked molecules [32]. In this mini-review, we will describe the recent advances in dynamic materials constructed from pillar[n]arenes bearing TEO chains.

2. Materials with LCST behavior

As mentioned above, pillar[n]arenes exhibit lower LCST behavior due to modification of multiple TEO groups. In 2012, Ogoshi *et al.* reported the first example of pillar[5]arene bearing ten TEO chains that exhibited LCST behavior [29]. As we can see in Fig. 1, the clear aqueous solution of **P5** at room temperature became turbid on heating before becoming transparent again upon cooling. At temperatures above the cloud point (T_{cloud}), the interaction of the hydrophobic groups causes **P5** to aggregate and then separate from water. The T_{cloud} was determined to be 42 °C in 2 mmol/L aqueous solution of **P5**. Notably, T_{cloud} was dependent upon the concentration of **P5**. As the concentration of **P5** increased, T_{cloud} decreased. Interestingly, the T_{cloud} of **P5** can be readily tuned from 42 °C to 60 °C upon addition of didecylviologen dibromide (**C10Vio**) as a guest. When **C10Vio** was added, the turbid solution of **P5** became clear at 55 °C (Fig. 1b). Furthermore, if cucurbit[7]uril was used as a competitive host to exclude **C10Vio** from the cavity of **P5**, T_{cloud} was recovered to that of pristine **P5**, followed by the observation of a turbid solution. This chemically

responsive material may have potential use for cloud point extraction.

Controlling supramolecular assemblies on the nanoscale by external stimuli has drawn much attention in nanoscience. Among various stimuli, light is especially useful due to its rapid and non-invasive nature. Azobenzene derivatives have proven to be especially advantageous due to their controllable conformational changes between the *trans* and *cis* forms with UV/visible light irradiation, accompanied by variations in their molecular properties. In a follow-up investigation, Ogoshi *et al.* demonstrated the photoreversible switching of LCST behavior in a photoresponsive host–guest system, constructed from pillar[6]arene with TEO substituents and an azobenzene derivative [33]. They first synthesized a new water-soluble thermoresponsive pillar[6]arene with 12 TEO groups (**P6**, Fig. 2a). Similar to **P5**, **P6** also exhibits LCST behavior in aqueous solution. Host–guest studies showed that the *trans* form of an azobenzene guest (**G1**, Fig. 2a) formed a stable 1:1 complex with **P6**. In a 2 mmol/L aqueous solution of **P6**, complexation increased the T_{cloud} from 41 °C to 57 °C. Irradiation with UV light induced a conformational change for **G1** from the *trans* to *cis* form, and dethreading occurred due to a size mismatch between the *cis* form of **G1** and the pillar[6]arene cavity. Subsequently, this dethreading decreased the T_{cloud} from 57 °C to 41 °C. This process was reversible, and the cloud point could be switched by alternating irradiation with UV/visible light. In this work, the photocontrollable threading/dethreading resulted in photoreversible switching of LCST behavior, exhibiting their potential application for drug delivery.

In 2014, Xue *et al.* reported a new water-soluble thermoresponsive pillar[10]arene with TEO groups, the cloud point of which could be reversibly controlled based on a chemical-responsive host–guest system (Fig. 3) [34]. By etherification of per-hydroxylated pillar[10]arene, **P10** was obtained as a light yellow liquid. Like pillar[5,6]arenes with TEO groups described above, **P10** also showed LCST behavior in water. The clear aqueous solution of **P10** (2.00 mmol/L) became turbid upon heating to 50 °C. It became clear again as the temperature decreased. Formation of a stable host–guest complex based on **P10** and **G2** was evidenced by ^{1}H NMR spectroscopy, UV–vis absorption spectroscopy and isothermal titration calorimetry. The authors demonstrated that the T_{cloud} of **P10** increased upon the gradual addition of **G2**, resulting in a
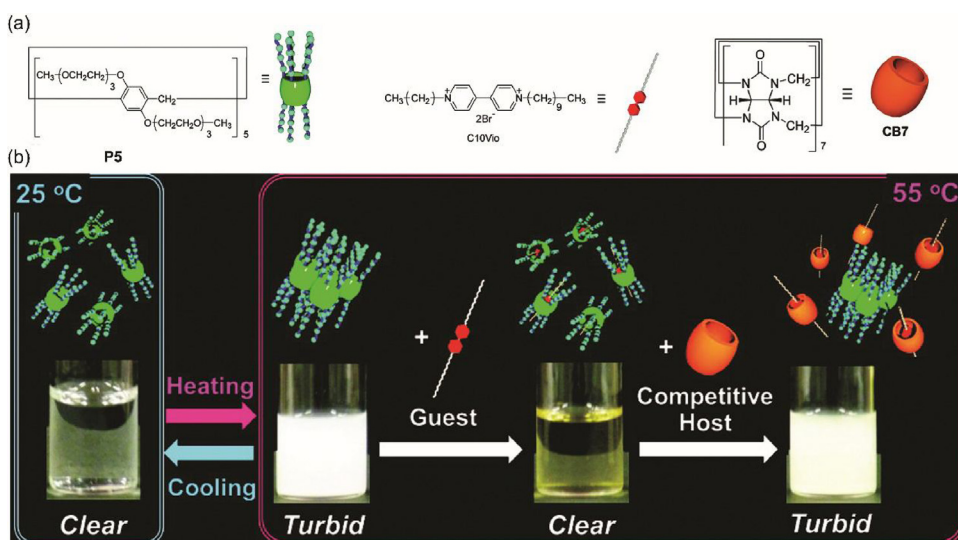

Fig. 1. (a) Chemical structures of pillar[5]arene modified with triethylene oxide groups, viologen derivatives, and cucurbit[7]uril. (b) Illustration of the reversibly tunable LCST utilizing host–guest complexation of **P5**. Reproduced with permission [29]. Copyright 2012, American Chemical Society.
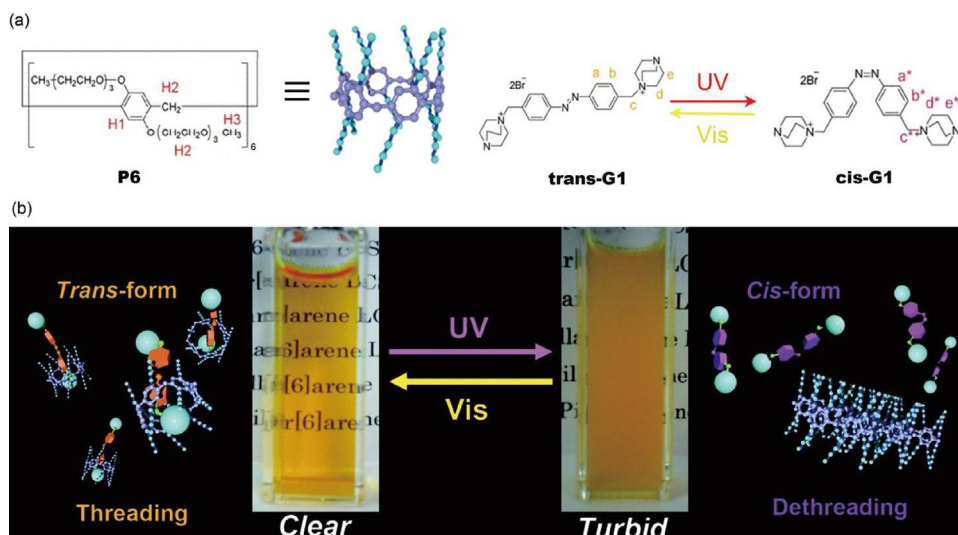
Fig. 2. (a) Chemical structures of pillar[6]arene modified with triethylene oxide groups (**P6**) and an azobenzene guest (**G1**). (b) Illustration of the photoreversible switching of LCST utilizing host–guest complexation of **P6**. Reproduced with permission [33]. Copyright 2012, American Chemical Society.

clear solution (Fig. 3b). When excess diethylamine (DEA) was added to the above solution, the yellow solution turned dark green, indicating dissociation of the complex. Then the complex formed again when trifluoroacetic acid (TFA) was added to neutralize DEA. Concomitantly, the dark green solution gradually reverted to yellow. This process could be repeated several times (Fig. 3c). The
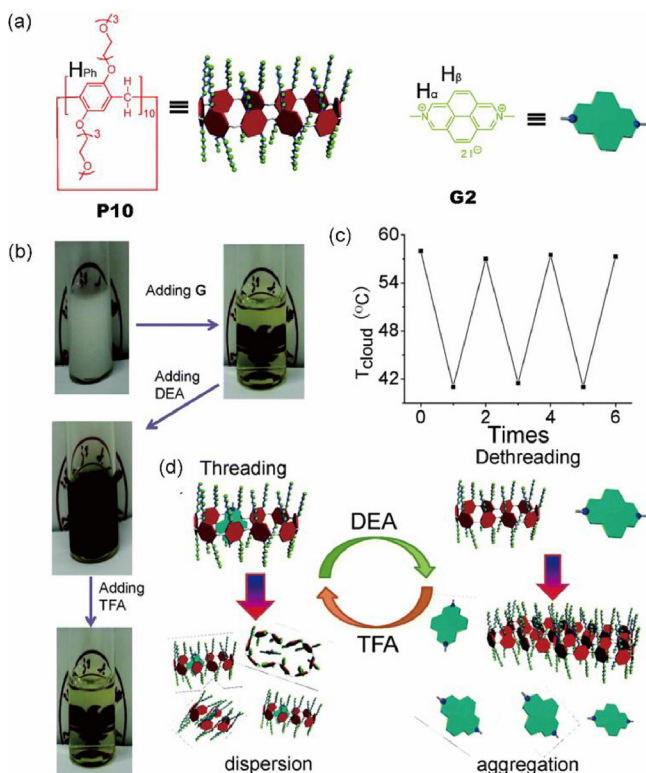

Fig. 3. (a) Chemical structures of **P10** and **G2**. (b) Photos of a mixture of **P10** (2.00 mmol/L) and **G2** (3.00 mmol/L) in water at 50 °C after adding DEA or TFA. (c) Changes in T_{cloud} upon addition of DEA or TFA. (d) Illustration of chemical-responsive control of the LCST behavior by pillar[10]arene-based host–guest interactions. Reproduced with permission [34]. Copyright 2014, the Royal Society of Chemistry.
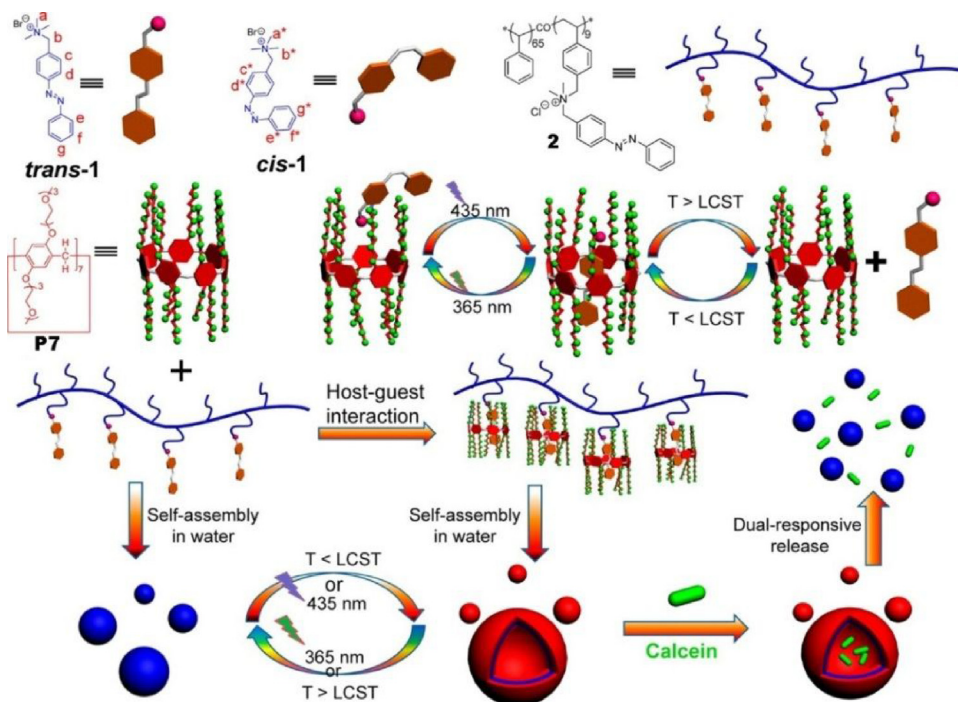
authors suggest that this thermoresponsive material has potential use in many bio-relevant fields, such as smart bioactive surfaces, phase separation immuno-assays, hyperthermia induced drug delivery and tissue engineering due to the biocompatible nature of the TEO moieties.

Macromolecular supra-amphiphiles refer to a kind of macro-molecular amphiphile whose hydrophobic and hydrophilic parts are linked by supramolecular forces. In 2015, Huang *et al.* reported a new host–guest recognition motif between a thermoresponsive water-soluble pillar[7]arene (**P7**) and an azobenzene derivative (Fig. 4) [35]. **P7** was functionalized with 14 TEO groups and also showed LCST behavior in water. The authors used this recognition motif to fabricate a pillararene-based supra-amphiphilic poly-pseudorotaxane, which can further self-assemble into vesicles in water. This new host–guest system in water has dual photo- and thermo-responsiveness. The host–guest complexation between **P7** and the model compound **1** was first investigated by ^{1}H NMR spectroscopy and ITC. The stoichiometry and binding constant between **P7** and *trans*-**1** was determined to be 1:1 and $(1.97 \pm 0.24) \times 10^4$ L/mol. The photocontrollable threading/dethreading between **P7** and **1** could be controlled by alternative irradiation with light at 435 nm and 365 nm. Moreover, based on the LCST behavior of **P7**, the disassembly and assembly process of the complex **P7**⊃*trans*-**1** could also be switched by the heating/cooling treatment. Subsequently, the self-assembly of random copolymer **2** and **P7** was studied by a combination of techniques, such as NOESY, DLS and TEM. As a result, the reversible transformations between nanoparticles based on the self-assembly of **2** and vesicles based on the self-assembly of **P7**⊃**2** were achieved by controlling the temperature or UV/vis light irradiation. Furthermore, the vesicles were successfully employed in the controlled release of calcein dye molecules. This example provided a new approach to combine polymer science with pillararene chemistry to fabricate functional supramolecular materials.

In a follow-up investigation, Huang *et al.* developed a dual-thermoresponsive gemini-type supra-amphiphilic macromolecu-lar [3]pseudorotaxane based on pillar[10]arene /paraquat cooper-ative complexation [36]. They chose pillar[10]arene with TEO groups (**P10**) as the host, which can bind two paraquat moieties to form a gemini-type supra-amphiphilic [3]pseudorotaxane (**P10**⊃**G3**$_2$) (Fig. 5). The guest molecule **G3** is paraquat-containing

Fig. 4. Chemical structures of **P7**, **1**, and polymer **2**, and illustration of the dual-responsive controlled assembly and disassembly of **P7/2** supramolecular vesicles and the process of dual-responsive release of calcein molecules. Reproduced with permission [35]. Copyright 2015, American Chemical Society.

poly(*N*-isopropylacrylamide) (PNIPAM) polymers. Both **P10** and **G3** exhibits LCST behavior in water. Generally, T_{cloud} of the host was affected by the addition of guest molecules. The effect of the added **P10** on the thermoresponsive behavior of **G3** was explored by

turbidity experiments. A solution **P10⊃G3**$_2$ was prepared in water at 20 °C, and then gradually warmed to 37 °C (above the LCST of PNIPAM), leading to the formation of vesicles evidenced by TEM and DLS. This is because the PNIPAM was changed from hydrophilic to hydrophobic upon increasing the temperature. Concomitantly, the water-soluble [3]pseudorotaxane was turned into an amphiphilic gemini-type supra-amphiphile. When a solution of vesicles was cooled to 25 °C, the hydrophobic part of **P10⊃G3**$_2$ became hydrophilic, and the average diameter of the assemblies decreased from 142 nm to 6.8 nm, indicating the fragmentation of vesicles at 25 °C. By contrast, when the temperature was increased to 60 °C, the average size of the aggregates increased from 142 nm to 1484 nm. This was due to the fact that phase separation had occurred when the temperature exceeded the LCST of **P10**, leading to irregular precipitates being observed by TEM. Finally, the vesicles were further applied in the controlled release of hydrophilic dye calcein and hydrophobic anticancer drug DOX.

Macrocyclic receptors are a major cornerstone in supramolecular chemistry. Recently, Jiang *et al.* reported several naphthol-based molecular receptors, of which perbutyloxatub[4]arene (**TA4**) was particularly interesting [37–40]. Although **TA4** does not belong to the pillar[*n*]arene family, it is of value to discuss **TA4** in the context of LCST behavior for this review. In recent work, the same group reported the synthesis and host–guest properties of a
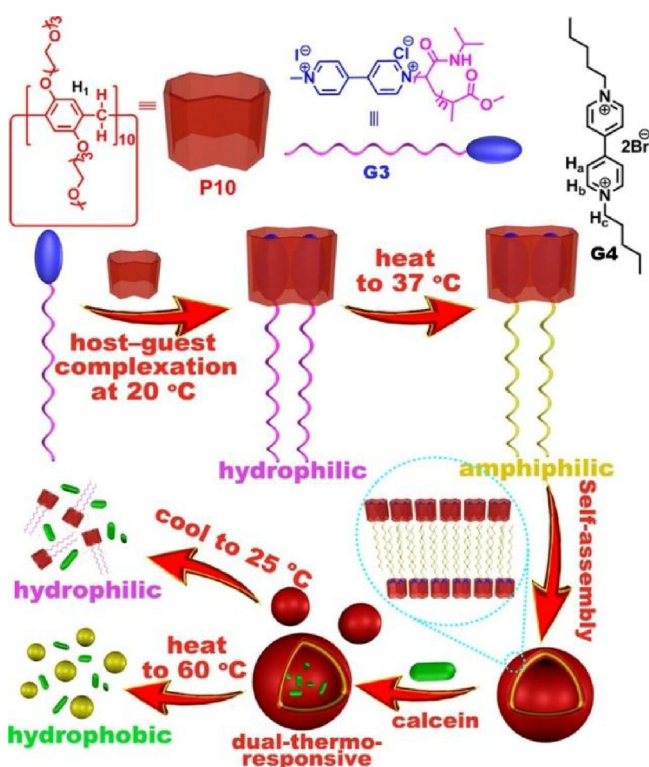

Fig. 5. Chemical structures of **P10**, **G4** and polymer **G3** and schematic illustration of preparation of polymeric vesicles and their application in dual-thermoresponsive release of calcein molecules Reproduced with permission [36]. Copyright 2016, American Chemical Society.

Fig. 6. Photos of **WTA4** (1.0 mmol/L) in water before and after addition of **7**$^+$ (2.0 mmol/L) followed by NaHS (30.0 mmol/L) in sequence at 43 °C. Reproduced with permission [41]. Copyright 2017, American Chemical Society.
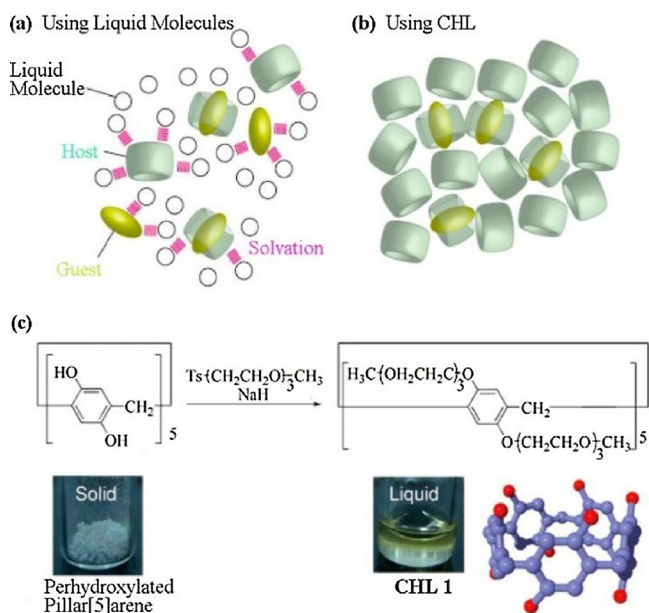
Fig. 7. Concept and synthesis of CHLs. (a) Host–guest complexation using liquid molecules as solvents. (b) Host–guest complexation in CHLs. (c) Synthesis and physical appearance of pillar[5]arene-based **CHL1**. Reproduced with permission [32]. Copyright 2012, American Chemical Society.

water-soluble oxatub[4]arene (**WTA4**) (Fig. 6) that showed H$_2$S-responsive LCST behavior [41]. **WTA4** exhibits high solubility in aqueous solution at 25 °C and displayed reversible LCST behavior. The T_{cloud} of **WTA4** (1 mmol/L) was determined to be 35.3 °C. By careful screening, it was determined that guest **7$^+$** could bind to **WTA4** in water via a 1:1 binding stoichiometry. The effect of host–guest complexation on the T_{cloud} of these systems was also studied. As the concentration of **7$^+$** increased from 0 to 3 mmol/L, the T_{cloud}

of **WTA4** gradually increased from 35.3 °C to 55.0 °C. On the basis of this host–guest system, chemically responsive turbid-to-clear and clear-to-turbid transitions can be achieved (Fig. 6). When a solution of **WTA4** was heated to 43 °C (above the T_{cloud} of **WTA4** (35.3 °C)), the solution became milky. Addition of **7$^+$** to the above solution at 43 °C (below the T_{cloud} of **WTA4⊃7$^+$** (55 °C)) caused the solution to become transparent. The solution reverted to turbidity upon the addition of NaHS at 43 °C. This is thought to be due to the production of H$_2$S from NaHS and subsequent reduction of the nitro group of **7$^+$**, leading to the destruction of the host–guest association between **7$^+$** and **WTA4**. This work is particularly important as the LCST behavior of this new host–guest system responds to the occurrence of the important physiological gasotransmitter H$_2$S.

3. Cyclic host liquids

Liquid media are commonly utilized for organic synthetic chemistry. By contrast to solid compounds, molecules in the liquid phase have more freedom and can react more easily with other molecules. To this end, Ogoshi et al. developed cyclic host liquids (CHLs) by transforming host molecules from solids to liquids at room temperature by modification with TEO groups [32]. This unique pillar[5]arene could not only play the role of host but also solvent when reacting with guest molecules and led to a highly efficient synthesis of mechanically interlocked molecules. As shown in Fig. 7, guest molecules could be directly surrounded by an excess of CHLs. While perhydroxylated pillar[5]arene is a solid, the pillar[5]arene modified with TEO groups (**CHL1**) is a liquid at room temperature. Introduction of flexible TEO chains reduces the crystallinity of pillar[5]arene and leads to a liquid state. **CHL1** has good solubility both in water and numerous organic solvents, indicating that **CHL1** could be employed as a solvent for a diverse range of compounds. DSC measurements showed that **CHL1** remained as a liquid even at −50 °C. In contrast to a typical
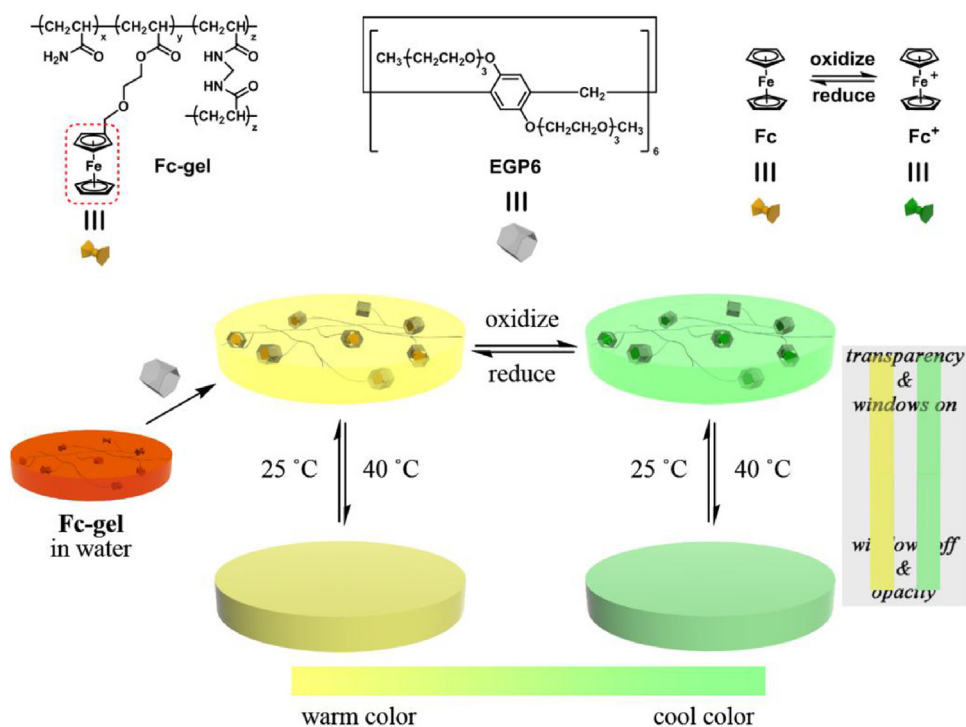

Fig. 8. Chemical structures and schematic illustration of the warm/cool tone switchable thermochromic material for smart windows. Copied with permission [43]. Copyright 2018, Nature Publishing Group.

complexation system, the CHL system is extremely efficient for maintaining host–guest complexation due to no addition of solvent being required. In this work, they demonstrated facile synthesis of [2]rotaxanes in a high yield by using **CHL1** even with unfavorable statistical combinations of host–guest complexes.

By combining the advantages of polymer science and mechanically interlocked molecules, polyrotaxanes are fascinating supramolecular architectures, in which many macrocyclic molecules can be threaded onto a linear polymeric chain. In a follow-up study, Ogoshi *et al.* reported an elegant polyrotaxane, capable of forming a topological gel by covalently linking alkene moieties on pillar[5]arene macrocycles [42]. Specifically, the pillar[5]arene macrocycles could function as CHLs. First, the authors utilised **CHL1** wheels and polytetrahydrofuran as a polymer chain to prepare pillar[5]arene-based non-ionic polyrotaxanes. Then, by changing the terminal $-CH_3$ groups on the TEO chains to cross-linkable $-CH=CH_2$ groups, topological gels were obtained after inter-molecular crosslinking using olefin metathesis. This case provides a new perspective to fabricate covalently cross-linked gels.

4. Smart window materials

Due to the thermo-responsiveness of TEO-modified pillar[6]arene (**EGP6**) and the redox-induced reversible color switching of ferrocene/ferrocenium, Wang *et al.* developed a novel hydrogel based smart window this year [43], Advanced functional materials play a pivotal role in the construction of smart windows, and can provide a more comfortable indoor environment for humans to enjoy a better lifestyle. Based on their previous research on host–guest interactions between pillar[6]arene and ferrocene [44–48], the authors first prepared a well-swollen and highly transparent hydrogel (**Fc-gel·EGP6**) which was obtained by immersing dry **Fc-gel** in an aqueous solution of **EGP6** (Fig. 8). Due to the formation of hydrophilic inclusion complexes between **EGP6** and **Fc-gel**, the water absorption capacity of the generated **Fc-gel·EGP6** hydrogel is dramatically improved, leading to drastically improved swelling behavior and an enhanced transparency. Furthermore, because of the thermo-responsiveness exhibited by **EGP6** and the reversible transformation between the orange and green colors of the ferrocene/ferrocenium group under redox-conditions, they have orthogonally integrated these functional properties in the design of a warm/cool tone-switchable thermochromic material. The authors are confident that such materials are highly desirable for the fabrication of smart windows with dual functionality–both for regulating the input of solar energy and for improving the mood and emotions of the inhabitants of a room.

5. Conclusions and outlook

In conclusion, recent advances in the construction of dynamic functional materials based on water soluble pillar[n]arenes with TEO groups are summarized in this review. Pillar[n]arene with TEO substituents is an extremely versatile host in the pillar[n]arene family, and exhibits numerous advantageous properties, such as water solubility, thermoresponsivity and liquidity at room temperature. Thus the application of the many pillar[n]arenes with TEO groups described in this review includes thermoresponsive materials with LCST behavior, cyclic host liquids and smart windows. In the future, we believe that the properties of such pillar[n]arenes will be further explored and many more fascinating dynamic materials fabricated from TEO substituted pillar[n]arenes will be reported.

Acknowledgments

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