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Emission enhancement induced by the supramolecular assembly of leggero pillar[5]arenes for the detection and separation of silver ions

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ABSTRACT

A renewable fluorescent material (**G⊂CP5L**) has been constructed *via* supramolecular assembly between a new derivative of pillararene, namely leggero pillar[5]arene, as the host molecule (**CP5L**) and a tetraphenylethylene (TPE)-based ditopic guest (**G**). This new material can simultaneously perform efficient detection and separation of silver(I) from aqueous environments. Possessing an electron-rich cavity and two cytosine groups modified on both rims, **CP5L** functions as the host-guest binding site for **G** and offers exclusive coordination sites for further interaction with Ag^+. Adding Ag^+ to the system undergoes dramatic fluorescence enhancement due to the mechanism of supramolecular assembly-induced enhanced emission (SAIEE). This fluorescence enhancement allows for efficient and visualized detection following a "light-up" pattern, achieving a limit of detection (LOD) of 1.3×10^{-7} mol/L, which is fully in line with the World Health Organization's drinking water standard of 9×10^{-7} mol/L. In addition, **G⊂CP5L** also shows strong anti-interference capability against other cationic species. For the separation of Ag^+ from aqueous systems, **G⊂CP5L** displays exceptional adsorption efficiency (97%) and reliable recovery performance, demonstrating excellent recyclability after five experimental cycles without compromising its adsorption activity

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Supramolecular assembling processes can lead to the generation of various stimuli-responsive materials *via* noncovalent interaction [1-3], such as hydrogen bonding [4,5], van der Waals force [6], metal-ligand coordination [7-9], electrostatic interactions [10], anion-π interactions, cation-π interactions [11], and halogen bonding interactions [12], host-guest recognitions [13], which endow these materials with dynamic and reversible properties. Pillar[*n*]arenes, as a novel category of synthetic macrocycles and a unique type of building blocks in the realm of supramolecular chemistry [14-16], play significant roles in fabricating functionalized supramolecular materials due to their rigid macrocycle skeletons [17,18], electron-rich cavities [19], versatile functionalizations [20,21], and superior host-guest properties [22]. Various supramolecular materials based on pillar[*n*]arene have been designed and applied in multiple research fields [23-25], such as drug delivery [26,27], gas adsorption and separation [28-30], artificial ion channels [31], and sensing and detection [32-35], thanking to their highly tunable properties and superior guest selectivity. In the meantime, the design and synthesis of pillar[*n*]arene derivatives with distinctive characteristics have been important pursuits for researchers. For instance, our group reported the syntheses of leaning pillar[6]arenes and geminiarenes with nanometer-sized adaptive cavities, demonstrating capabilities of separating aromatics and cyclic aliphatics [17,36,37]. Chen *et al.* fabricated a pagoda[5]arene with a large electron-rich cavity, which forms stable complexes with pyridinium or bipyridinium salts *via* host-guest interaction and exhibits acid/base-responsiveness in solution [38]. Notably, we developed a new version of pillararenes denoted as leggero pillar[5]arene (P[5]L), which possesses four alkoxyl-modified phenyl groups and one bare benzene ring in its molecular skeleton [39]. Its water-soluble derivative and mono-quinone substituted derivative have also been reported [40]. Recently, a novel leggero pillar[4]arene[1]quinone containing one benzoquinone unit was reported, which shows high selectivity to *p*-xylene over other C8 aromatics [41]. Compared with traditional pillar[5]arene, P[5]L possesses enhanced adaptability in its cavity structure, while the inherent host-guest properties of pillararenes are nicely maintained. Therefore, we envision that the selective modifications on the portals of P[5]L would open up more possibilities in creating

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supramolecular materials and developing the practical applications of this new macrocycle in broader fields.

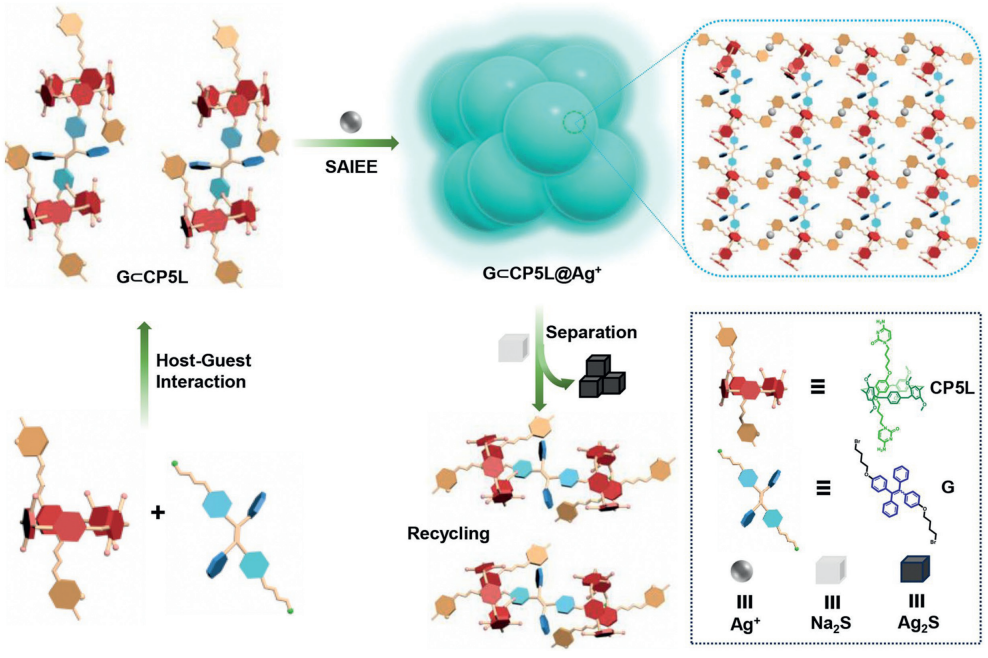
In 2001, Tang and colleagues proposed the mechanism of aggregation-induced emission (AIE) [42], where the AIE-active luminogens show weak fluorescence in diluted solutions yet become significantly more emissive upon aggregation due to the restriction of intramolecular motions (RIM) [43]. This groundbreaking photophysical mechanism thus effectively addressed the issue of aggregation-caused quenching (ACQ) observed in traditional organic luminophores and propelled the development of luminescent materials [44,45]. Over the years, researchers have invested a tremendous amount of effort in combining supramolecular self-assembling strategy with AIE luminogens [46], resulting in the creation of versatile fluorescent supramolecular materials with outstanding sensitivity and responsiveness. For instance, the integration of AIEgens with host-guest systems has been widely adopted in constructing artificial light-harvesting systems [47-51], fluorescence sensing and bioimaging agents [52,53], and other related fields [54]. It is worth noting that, building on earlier fundamental studies [55-58], a mechanism has been proposed for supramolecular assembly-induced enhanced emission (SAIEE) [59], it can be deemed a promising method in preparing substances with enhanced photoluminescence properties and enriched functionalities, thus closing the gap between supramolecular macrocycles and fluorescent materials [60-62].

Silver ions, as one of the heavy metal ions, impact the health of organisms by deactivating proteins [63]. The World Health Organization (WHO) stipulates strict regulations that the amount of Ag^+ in potable water must not exceed 0.9 μmol/L. Therefore, designing and developing a probe material that can simultaneously and efficiently detect and separate silver ions from aqueous environments is imperative. Drawing inspiration from the specific coordination between cytosine (C) and silver ion (C-Ag^+-C) [64,65], we herein report the design and preparation of a supramolecular assembly (**G⊂CP5L**) between cytosine-functionalized leggero pillar[5]arene (**CP5L**) as the host molecule, and a TPE-based ditopic guest (**G**) with AIE characteristics *via* host-guest interactions. With the addition of Ag^+ aqueous solution, a supramolecular polymer, *i.e.*, **G⊂CP5L**@Ag^+, can be constructed through C-Ag^+-C pairs. This construction process is accompanied by a phenomenon known as the SAIEE mechanism, which leads to a significant increase in fluorescence emission (Scheme 1). Hence, this supramolecular material (**G⊂CP5L**) can be used as a fluorescence probe to detect and separate silver ions with superior recycling capacity. We anticipate this strategy will broaden the real-world application scope of leggero pillararenes and provide a practical and efficient approach for detecting and separating silver ions pollution in water.

Detailed synthetic pathways of **CP5L**, **G**, and **G1** are shown in Figs. S1 and S9 (Supporting information). In short, we have synthesized cytosine-functionalized leggero pillar[5]arene (**CP5L**) for the first time with a yield of 65% through nucleophilic substitution reaction between the precursor P5L-$(Br)_2$ and cytosine. Tetraphenylethylene (TPE) modified by two bromoalkyl units (**G**) and a single bromo-substituted TPE (**G1**) were prepared according to literature reports [66]. All the substances reported for the first time were characterized using ^{1}H NMR, ^{13}C NMR, and high-resolution mass spectroscopy (HRMS) (Figs. S2-S12 in Supporting information).

Before carrying out investigations on the host-guest interactions between **G** and **CP5L**, we must exclude the possibilities of self-inclusion within **CP5L**, considering the flexible alkyl side chains modified on its rims and the adaptive cavity. 2D ROESY NMR experiments were performed to analyze the spatial structure relationship of **CP5L** (Fig. 1a). For the phenyl proton signal peaks on the macrocycle (H_c), observable cross-peaks can be found correlated with the methylene proton signals (H_f) and methoxy proton signals (H_g), respectively, attributing to π-π interactions between the benzene rings of **CP5L** and C-H···π interactions between the benzene rings and the methylene and methoxy groups at high concentrations. On the contrary, no correlation was observed among **CP5L** phenyl protons (H_c) and flexible alkyl side chains protons (H_e, H_i) or cytosine group protons (H_a, H_d). This phenomenon suggested that the cytosine groups on the side chain were not encircled by the cavity of **CP5L** itself, verifying the absence of self-inclusion structures within **CP5L**.

The host-guest complexation formed between **G** (4 mmol/L) and **CP5L** (8 mmol/L) was first studied by ^{1}H NMR in the mixed deuterated solvent of $CDCl_3$/DMSO-d_6 (v:v = 9:1). As shown in Fig. 1b, af-



Scheme 1. Illustration of the silver ions' detection and separation process using the recyclable supramolecular assembly **G⊂CP5L**.

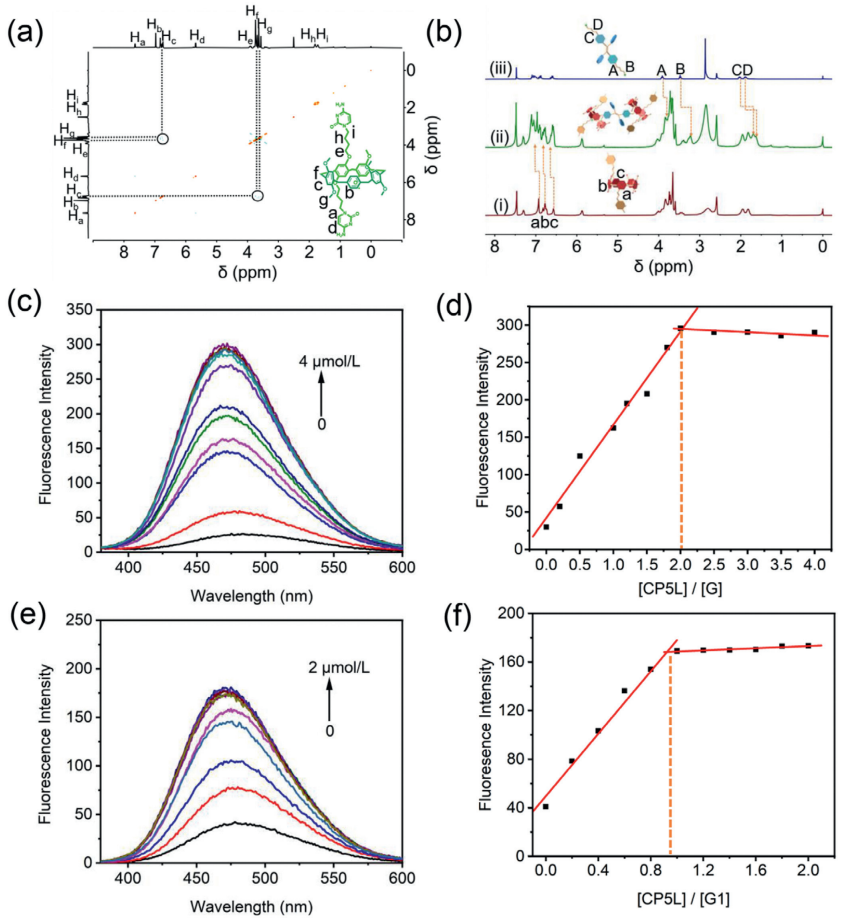


Fig. 1. (a) 2D ROESY analysis (600 MHz, DMSO-d_6, 298 K) of **CP5L** (20 mmol/L). (b) ^{1}H NMR spectra (400 MHz, 298 K, $CDCl_3$/DMSO-d_6 = 9/1, v/v) of (i) **CP5L**; (ii) **CP5L** + **G**; and (iii) **G** ([**G**] = 4 mmol/L, [**CP5L**] = 8 mmol/L). (c) Fluorescence spectra of **G** with different fractions of **CP5L**. (d) Molar ratio plot for **CP5L** and **G**, demonstrating 2:1 complexation stoichiometry ([**G**] = 1 μmol/L, [**CP5L**] = 0–4 μmol/L). (e) Fluorescence spectra of **G1** with different fractions of **CP5L**. (f) Molar ratio plot for **CP5L** and **G1** ([**G1**] = 1 μmol/L, [**CP5L**] = 0–2 μmol/L), demonstrating 1:1 complexation stoichiometry (λ_{ex} = 310 nm; excitation/emission slit widths = 3/3 nm; H_2O/DMSO = 9/1, v/v).

ter adding guest **G** to host **CP5L**, the proton signal peaks on the side chain of **G** showed a clear upfield shift, indicating that the side chain of **G** had been encapsulated by the cavity of **CP5L**. Moreover, a downfield shift of proton signals of the phenyl group on **CP5L** provided further confirmation of successful complexation between host and guest. The binding stoichiometry between **CP5L** and **G** was confirmed *via* fluorescence titration experiments. According to Figs. 1c and d, the stoichiometric ratio of **CP5L** and **G** was defined as 2:1. To verify the accuracy of this stoichiometry, we also synthesized a monobromo-substituted TPE as model guest (**G1**) and repeated the titration mentioned above. The results confirmed a 1:1 stoichiometric relationship between **G1** and **CP5L** (Figs. 1e and f), with the association constant (K_a) of (4.86 ± 0.2) × 10^7 L/mol, demonstrating high affinity between the guest entity and the cavity of **CP5L** (Fig. S13 in Supporting information). In addition, the SEM images showed that the host-guest complexes possessed a spherical morphology with regular shapes distinct from **G** (Fig. 2a), which also evidenced the successful preparation of **G⊂CP5L** (Fig. 2b).

The coordination effect of Ag^+ with the cytosine moieties of **CP5L** was investigated *via* UV–vis spectroscopy. As depicted in Fig. S14 (Supporting information), UV absorbance intensity steadily increased with the introduction of Ag^+. When above 1.0 equiv. of Ag^+ was added, UV absorbance of the mixture reached its maximal and maintained unchanged, indicating the formation of C-Ag^+-C pairs between **CP5L** and Ag^+. Moreover, ^{1}H NMR titration experiments further supported this conclusion (Fig. S15 in Supporting information). Upon adding Ag^+, cytosine proton signals of **CP5L** were shifted to the lower field. When 1.0 equiv. of Ag^+ was added, the proton signal peaks decreased to the minimum and almost disappeared, indicating the 1:1 coordination pattern between Ag^+ and **CP5L**.

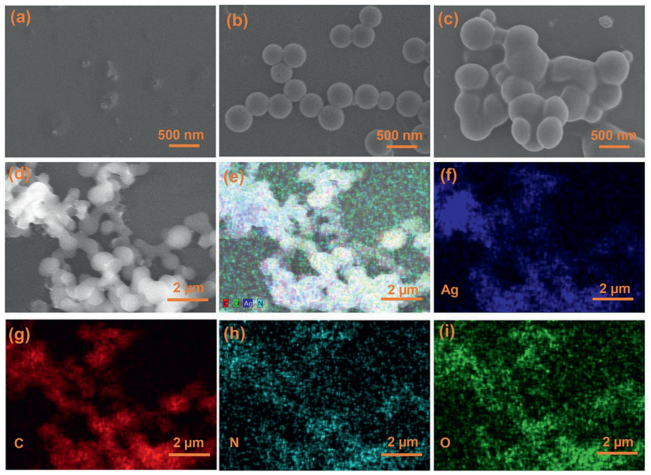


Fig. 2. SEM images of (a) **G**, (b) **G⊂CP5L**, (c, d) **G⊂CP5L**@Ag^+, and elemental mapping of **G⊂CP5L**@Ag^+, consisting of (e) the overlapped image and the distributions of (f) Ag, (g) C, (h) N, and (i) O, respectively.

Based on the above experiments, the supramolecular polymer, namely **G⊂CP5L**@Ag^+, was constructed *via* a synthetic method where the first step involved the preparation of host-guest complex **G⊂CP5L**, and the coordination with Ag^+ was conducted as the second step. Scanning electron microscopy (SEM) revealed that **G⊂CP5L**@Ag^+ exhibited a larger particle size than **G⊂CP5L** (Figs. 2c and d). Energy dispersive spectroscopy (EDS) results further indicated that the Ag element was distributed uniformly in the supramolecular assembly, proving the successful coordination of Ag^+ with **G⊂CP5L** (Figs. 2e-i). Additionally, further elucidation of the assembly process from **CP5L** to **G⊂CP5L** and subsequently to **G⊂CP5L**@Ag^+ was achieved through two-dimensional diffusion-ordered NMR spectroscopy (2D DOSY). As shown in Fig. S16 (Supporting information), the diffusion coefficients (D) for **CP5L**, **G⊂CP5L**, and **G⊂CP5L**@Ag^+ were 1.1×10^{-10}, 1.04×10^{-10}, and 9.03×10^{-11} m^2/s, respectively. The decrease in diffusion coefficients revealed the formation of **G⊂CP5L**@Ag^+ with higher molecular weights. We further confirmed the coordination between Ag^+ and **G⊂CP5L** using Raman spectroscopy. As shown in Fig. S17 (Supporting information), the characteristic peak at 1060 cm^{-1} was attributed to the C-N stretching vibration of cytosine, which shifted to 1070 cm^{-1} upon the addition of Ag^+. Additionally, the C=N and C=C stretching vibrations at 1572 cm^{-1} shifted significantly to 1625 cm^{-1}. The peak at 1321 cm^{-1} (C-H bending and C-N stretching) also shifted to 1304 cm^{-1}. Moreover, after adding Ag^+, the peak intensities at 953 cm^{-1} (C-C and C-N bending), 1209 cm^{-1} (C-N stretching) and 1418 cm^{-1} (C=C stretching) increased markedly. These results indicate the complexation of Ag^+ with **G⊂CP5L**.

Subsequently, the photophysical features of **G⊂CP5L**@Ag^+, **G⊂CP5L**, and **CP5L** were evaluated to determine whether SAIEE properties can be achieved upon the formation of supramolecular polymers. Regarding fluorescence intensity, **G** exhibited negligible fluorescence in dilute solutions owing to the unrestricted rotation of phenyl rings and conjoined nonradiative energy dissipation [67,68]. **G⊂CP5L** displayed slightly enhanced fluorescence emission, although the intensity remained rather weak. However, remarkably enhanced fluorescence emission was detected upon adding Ag^+ into the host-guest complexes, where the fluorescence intensification can be observed with naked eyes under UV light irradiation (Fig. 3a). Additionally, fluorescence experiments were performed to examine the fluorescence lifetime and quantum yield of **G⊂CP5L**@Ag^+. As shown in Fig. 3b, **G⊂CP5L**@Ag^+ demonstrated a fluorescence lifetime of 5.80 ns, surpassing the durability of **G** (4.02 ns) and **CP5L** (4.79 ns). Concurrently, the fluorescence quantum yield of **G⊂CP5L**@Ag^+ reached 31.51% (Fig. 3c), significantly exceeding **G** (4.73%) and **G⊂CP5L** (10.40%). Remarkably, **G⊂CP5L**@Ag^+ exhibited a higher radiative decay rate constant (K_r) of 5.42×10^7 s^{-1} coupled with a lower nonradiative decay rate constant (K_{nr}) of 1.18×10^8 s^{-1} compared with that of **G** (1.18×10^7 s^{-1} and 2.37×10^8 s^{-1}, respectively) and **G⊂CP5L** (2.17×10^7 s^{-1} and 1.87×10^8 s^{-1}, respectively) (Fig. 3d and Table S1 in Supporting information). In short, fluorescent supramolecular polymers involving host-guest complexation and silver(I) coordination were successfully fabricated and exhibited exceptional optical properties relying on the SAIEE mechanism.

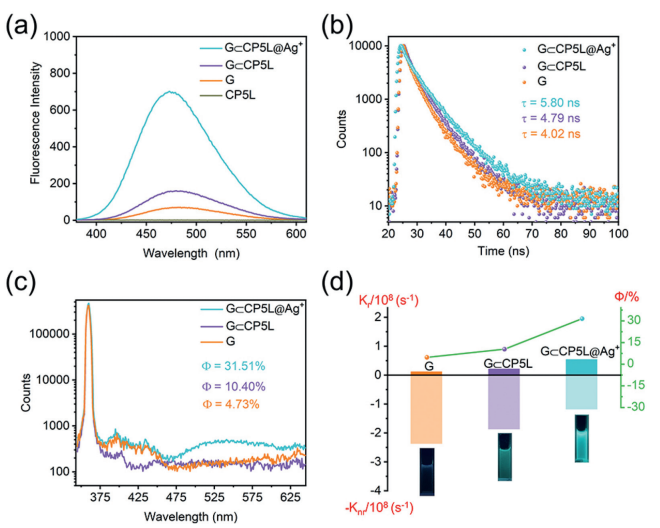


Fig. 3. (a) Fluorescence emission spectra of **G⊂CP5L**@Ag+, **G⊂CP5L**, **G** and **CP5L**. (b) Fluorescence decay profile and (c) quantum yield of **G⊂CP5L**@Ag^+, **G⊂CP5L**, **G**. (d) Comparison of the radiative/nonradiative decay rate constants and quantum yields of **G⊂CP5L**@Ag^+, **G⊂CP5L**, **G**. Inset: digital photos captured under UV light irradiation at 365 nm for **G**, **G⊂CP5L**, and **G⊂CP5L**@Ag^+.

Silver ion titration experiments were conducted to elucidate the importance of host-guest complexes **G⊂CP5L** in forming **G⊂CP5L**@Ag^+. As depicted in Fig. S18 (Supporting information), no discernible changes were spotted in the fluorescence intensity of either **G** or **CP5L** after introducing Ag^+. Nevertheless, a marked enhancement in the fluorescence emission of **G⊂CP5L** was observed upon continuous titration of Ag^+, reaching a maximal value as 1 equiv. of Ag^+ was introduced into the system. These findings demonstrated the significance of **G⊂CP5L** in the development of the fluorescence-enhanced **G⊂CP5L**@Ag^+.

In this section, titration experiments of Ag^+ into **G⊂CP5L** were performed, where the aqueous solutions of Ag^+ at different concentrations were added to **G⊂CP5L** for fluorescence testing. As depicted in Fig. 4a, the fluorescence emission exhibited a linear

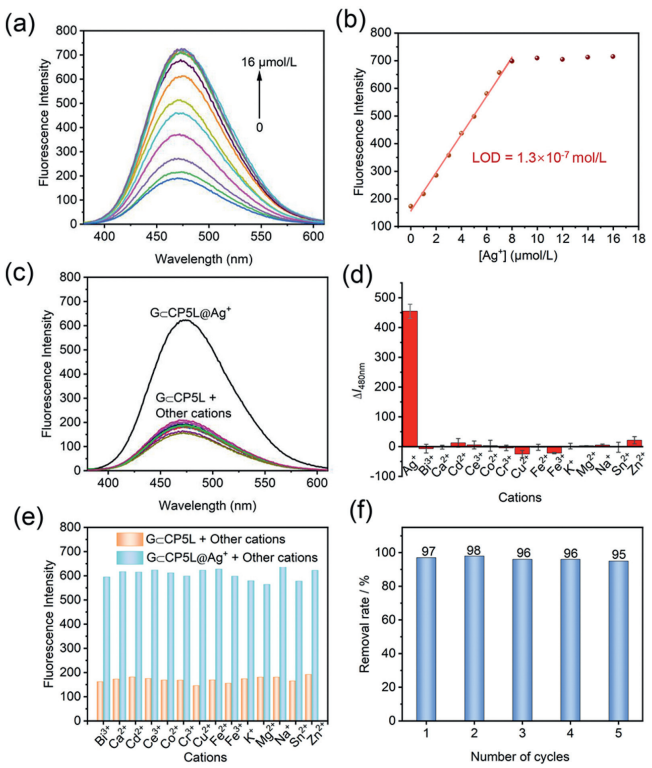


Fig. 4. (a) Fluorescence emission spectra of **G⊂CP5L** (8 μmol/L in DMSO) with different Ag^+ concentrations (0–16 μmol/L) in water. (b) Fluorescence intensity of **G⊂CP5L** at 480 nm *versus* Ag^+ concentrations under $\lambda_{ex} = 310$ nm; excitation/emission slit widths = 3/3 nm; H_2O/DMSO = 9/ 1, v/v. (c) Fluorescence spectra and (d) fluorescence changes of **G⊂CP5L** upon adding aqueous solutions of various metal cations (1.0 equiv. of each). (e) Degrees of fluorescence enhancement at 480 nm derived from **G⊂CP5L** with Ag^+ and other cations species (1 equiv.). (f) Recycling performance of **G⊂CP5L** toward the separation of Ag^+.

enhancement upon increasing Ag^+ concentration at 0–8 μmol/L. The limit of detection (LOD) was determined to be 1.3×10^{-7} mol/L based on 3 δ/s (Fig. 4b), where δ represents the standard deviation of 20 blank tests, and s denotes the slope of the fluorescence emission standardization curve equation. This LOD value is below the standard concentration of Ag^+ for drinking water set by WHO (9×10^{-7} mol/L) [69], indicating that **G⊂CP5L** can potentially be used as a fluorescence probe to detect Ag^+ in aqueous systems.

Fluorescence experiments were employed to assess the selectivity of **G⊂CP5L** for Ag^+. Fluorescence spectra of **G⊂CP5L** were recorded upon the addition of 15 metal cations (1 equiv.), including K^+, Na^+, Ca^{2+}, Cd^{2+}, Co^{2+}, Cu^{2+}, Fe^{2+}, Mg^{2+}, Sn^{2+}, Zn^{2+}, Bi^{3+}, Ce^{3+}, Cr^{3+}, Fe^{3+}, and Ag^+, respectively. As depicted in Fig. 4c, only Ag^+ can initiate a significant fluorescence intensity increase under identical conditions, indicating that **G⊂CP5L** was capable of performing selective recognition and detection toward Ag^+ exclusively (Fig. 4d). Furthermore, we used nitrate as the corresponding anion for the selected metal ions. To eliminate the possibility of interference from anions, we selected different sodium salts with various anions for fluorescence testing and compared them with sodium nitrate. As shown in Fig. S19 (Supporting information), different anions have no effect on the probe. In particular, it is worth noting that a multiplicity of heavy metal cations is present simultaneously in real-world aqueous conditions, such as industrial wastewater. Therefore, anti-interference experiments were implemented, whereby the presence of other metal cations demonstrated a very slight impact on the fluorescence emission increase of Ag^+ (Fig. 4e). Additionally, we investigated the stability of **G⊂CP5L** in detecting silver ions using fluorescence spectroscopy under conditions ranging of 20–50 °C. As shown in Fig. S20 (Supporting information), the fluorescence intensity of **G⊂CP5L**@Ag^+ shows a slight decreasing trend with increasing temperature. However, the extent of this decrease is minimal, suggesting that the probe possessed outstanding stability in the detection of Ag^+.

In order to confirm the ability of **G⊂CP5L** to separate Ag^+ from aqueous media, we carried out Ag^+ separation experiments and recycling experiments (Fig. 5). First, an aqueous solution of Ag^+ was introduced into **G⊂CP5L**, and the solution immediately converted into a white suspension with bright fluorescence under 365 nm UV light irradiation, followed by centrifugation to get a white precipitate. This result indicated the strong coordination between cytosine moieties of **G⊂CP5L** and Ag^+. Inductively coupled plasma spectrometry (ICP) experiments were conducted to determine the residual Ag^+ concentration in the solution. The result showed that the remaining Ag^+ concentration was below 0.94 ppm, indicating that over 97% of Ag^+ could be rapidly separated *via* simple operations. Based on the excellent Ag^+ separation efficiency of **G⊂CP5L**, we proceeded to assess its recyclability by dissolving the white precipitate in DMSO and introducing excessive Na_2S into the mixture, which led to the generation of Ag_2S as black precipitates. Thus, **G⊂CP5L** can be recovered after centrifugation. The recycling experiments of five cycles demonstrated no compromise in its adsorption activity (Fig. 4f). Therefore, we have validated that **G⊂CP5L** can function as a fluorescence probe for both detecting and separating Ag^+ in water, offering a feasible strategy and a reproducible platform for practical remediation of Ag^+ contaminants.

In summary, we have developed a supramolecular assembly system (**G⊂CP5L**) by employing cytosine-functionalized leggero pillar[5]arene and tetraphenylethylene (TPE)-based ditopic guest. **G⊂CP5L** possessed superior responsiveness to silver ions in water, taking advantage of the formation of **G⊂CP5L**@Ag^+ assembled *via* the coordination effect between cytosine moieties of **CP5L** and Ag^+. **G⊂CP5L**@Ag^+ displayed intensified blue-green fluorescence on account of the SAIEE mechanism. High selectivity and anti-interference ability have been achieved in the concurrent detection and separation of silver ions from aqueous media, demonstrating a low LOD value and high separation efficiencies. Particularly, **G⊂CP5L** can be recovered with very slight performance degradation after five cycles of continuous separation experiments, making it a promising candidate for sustainable wastewater remediation. This research enriches the functionalization protocols of leggero pillar[5]arenes and offers a simple and effective method for optically sensing and removing excessive silver ions in water. In the future, these supramolecular materials can be further modified to broaden their detection and recognition range for metal ions and pollutants. This will ensure that they meet the demands of diverse real-world applications, paving the way for innovative solutions to contemporary challenges.

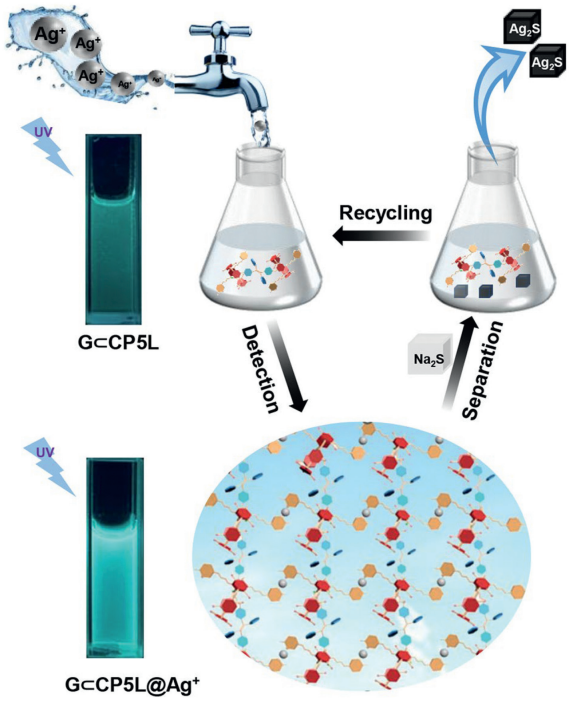


Fig. 5. Cartoon illustration of the recycling process of sensing and removal of Ag^+ by **G⊂CP5L**.

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

Kun Zhang: Writing – original draft, Methodology, Formal analysis, Data curation, Conceptualization. **Xin-Yue Lou:** Writing – review & editing, Validation, Methodology, Formal analysis. **Yan Wang:** Writing – review & editing, Validation, Formal analysis. **Weiwei Huan:** Writing – review & editing, Validation, Resources, Funding acquisition. **Ying-Wei Yang:** Writing – review & editing, Supervision, Resources, Project administration, Funding acquisition.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2024.110464.

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