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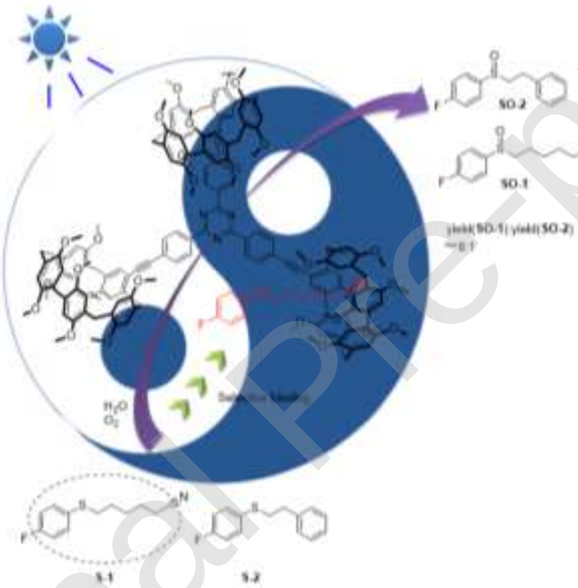
Communication

Pillar[5]arene based conjugated macrocycle polymers with unique photocatalytic selectivity

Hui Qiang[a, b], Tao Chen[b, c], Zhuo Wang[d], Wenqian Li[b, c], Yunzhe Guo[b, c], Jie Yang[d], Xueshun Jia[a], Hui Yang[b], Weibo Hu[b,*], Ke Wen[b, d,*]

[a] *College of Science, Shanghai University, Shanghai 200444, China*
[b] *Shanghai Advanced Research Institute, Chinese Academy of Sciences, Shanghai 201210, China*
[c] *University of Chinese Academy of Sciences, Beijing 100049, China*
[d] *School of Physical Science and Technology, ShanghaiTech University, Shanghai 201210, China*

Graphical Abstract



Enhanced catalytic photo-oxidation efficiency of the pillar[5]arene-contained conjugated organic polymers (**CMP-1** and **CMP-2**) toward their guest-like substrate **S-1** was demonstrated and a 'host-guest' interaction related mechanism was proposed. The conversion yield of **S-1** achieved near 18 folds over the non-guest –like substrate **S-2** in the case that **CMP-2** was used as a photocatalyst.

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ABSTRACT

The development of heterogeneous catalysts with substrate shape, size or electronic constitution selectivity is a huge challenge in photocatalysis. Reported herein is a host-guest interaction strategy to endow photocatalysts with special selectivity. By adjusting the precursors, conjugated macrocycle polymers (**CMPs**) with pillar[5]arene struts (**CMP-1** and **CMP-2**) and a corresponding non-pillar[5]arene-contained conjugated organic polymer (**COP-1**) were prepared

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⁎ Corresponding authors.
E-mail addresses: huwb@sari.ac.cn (W. Hu), wenk@sari.ac.cn (K. Wen)

and the photocatalytic activities toward sulfide derivatives were investigated. The sulfides showed similar conversions when **COP-1** was used as a photocatalyst, but exhibited significant differences when it turned to the **CMPs**. Remarkably, the conversion yield of **S-1** achieved near 18 folds over the one of **S-2** when **CMP-2** was used as a catalyst. Mechanism studies confirmed that the "host-guest" effect of pillar[5]arene struts in **CMPs** was the main cause of the difference. The present work establishes **CMPs** as novel heterogeneous photocatalysts with substrate selectivity, and such a method will inspire the researchers concerning preparation of heterogeneous catalysts with excellent selectivity.

Keywords:
Heterogeneous photocatalysis
Selectivity
Host-guest
Pillar[5]arene
Sulfide

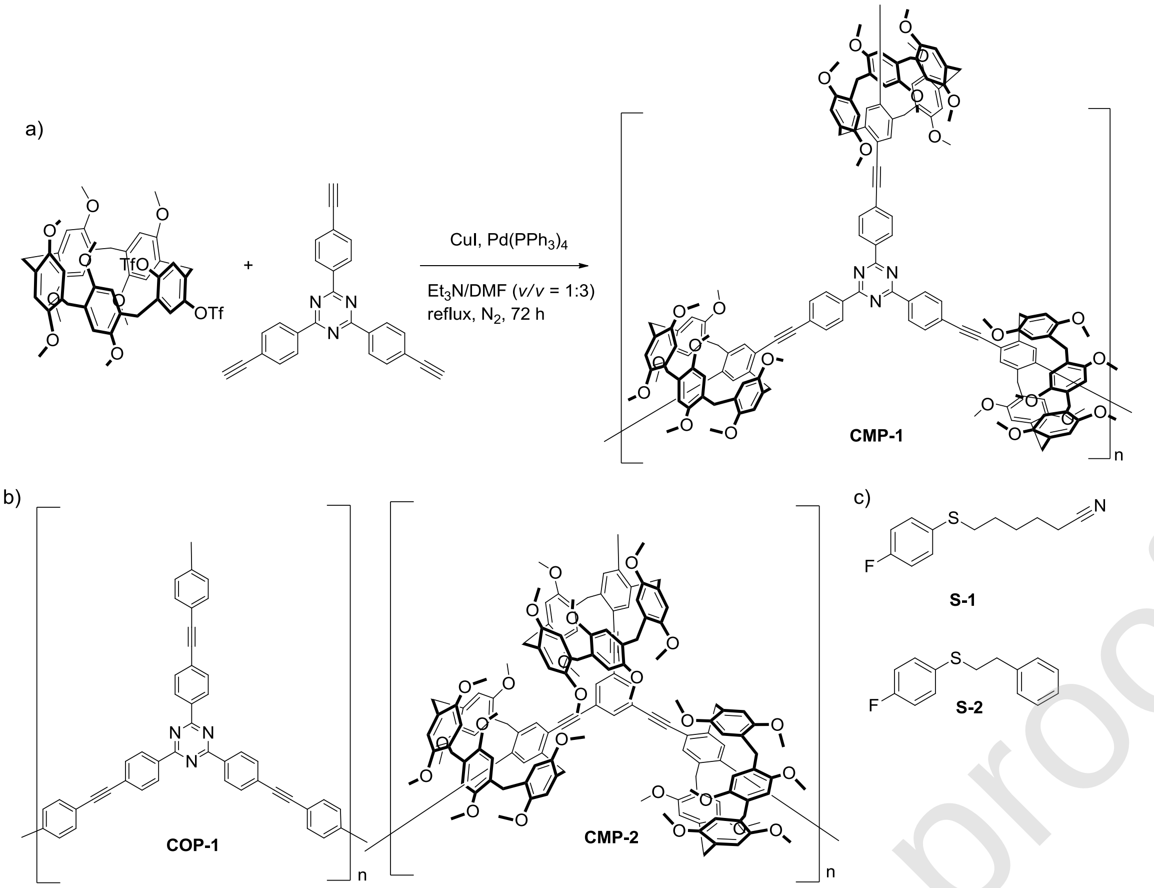
To alleviate energy crisis, design and preparation of photocatalysts which can efficiently convert solar energy into chemical energy have drawn increasing interests in recent years [1-7]. Various photocatalytic systems, including inorganic semiconductor, organic semiconductor [8,9], and metal-organic complex [10-12] have been intensely developed. Among them, organic semiconductors [13-18], such as conjugated organic polymers (COPs) [19-23], possessing advantages of facilely tailored functionality, thermal stability, recyclability and environmental friendliness, have gained much attention recently. Indeed, excellent photocatalytic activity has been achieved by some functional COPs [24-31], but substrate selective photocatalysis have rarely been reported. As such, strategies which enable photocatalysts with substrate shape, size or electronic constitution selectivity are very welcome.

Enzymatic reactions possess highly substrates selectivity, which is partially due to their binding specificity [28, 32-36]. Similar to enzymatic reactions, interactions between the reactant molecules and the catalysts is an important process for a heterogeneous photocatalysis. Hence, once selective adsorption occurred, substrates selective photocatalysis could be achieved. It is well-known that macrocyclic hosts could bind guest molecules selectively [37-42]. For example, pillar[5]arenes tend to include linear chain molecules into their cavities [43-46], and the binding constants vary with different functional groups of the linear chain molecules [47]. Recent studies have indicated that solid materials with macrocyclic host motifs could adsorb specific molecules [48-60]. Therefore, developing conjugated macrocycle polymer (CMPs) [61] photocatalysts, a class of organic semiconductors possessing macrocyclic host motifs, might be a feasible way to endow photocatalysts with special substrate selectivity.

Herein, we report a type of conjugated macrocycle polymers with pillar[5]arene struts (**CMP-1** [61] and **CMP-2**) for selective photocatalytic oxidation of sulfides. A corresponding non-pillar[5]arene-containing conjugated organic polymer (**COP-1**) was also prepared for comparison. Electron spin resonance (ESR) studies indicated that the **CMPs** and **COP-1** all possessed the abilities to convert O_2 or H_2O to active oxygen species (•OH or $^1O_2$) under visible light irradiation to initiate the oxidation of sulfides. **COP-1** showed similar photocatalytic activity toward the oxidation of sulfides with different substituents. **CMP-1** and **CMP-2** were found to exhibit much higher activity toward a guest-like sulfide than a non-guest-like one. The influence of the host-guest nature of the pillar[5]arene struts in the **CMPs** on the photocatalytic oxidation of the guest-like sulfide was confirmed by competitive experiments.

The **CMPs** and **COP-1** were prepared according to literature reports (details are deposited in Supporting information) [61,62]. The formation of the polymers was first characterized by Fourier transform infrared spectroscopy (FTIR). The disappearance of the C-H stretching band around 3250 cm^{-1} of the alkynes and the trifluoromethane sulfonate group at 931 cm^{-1} of pillar[4]arene[1]1,4-phenylenebistriflate indicated total consumption of the starting materials (Figs. S1a-c in Supporting information). ^{13}C cross-polarization magic-angle spinning (CP-MAS) NMR analysis provided further evidence for the formation of the polymers. Both **CMP-1** and **CMP-2** showed clear signals at ~30, ~55, ~90 and ~170 ppm, which could be assigned to the carbons of methoxy, methylene of the pillar[5]arene moiety, and carbons of the C≡C and triazine C=N units, respectively (Fig. 1d and Fig. S1d in Supporting information). Similarly, in the (CP-MAS) NMR spectra of **COP-1**, the signals at ~170 ppm assigned to triazine C=N units and ~90 ppm assigned to C≡C units were observable (Fig. 1d).

Scanning electron microscopy (SEM) was used to study the morphologies of the polymers. Irregular particles with different sizes for the as-synthesized conjugated polymers of **CMP-1** and **COP-1** could be observed in the SEM images, and the particles were inclined to be independent with each other (Figs. 1a-b). Interconnected particles could be found in **CMP-2** (Fig. S1e in Supporting information). Brunauer−Emmett−Teller (BET) surface areas were calculated from N_2 adsorption/desorption isotherm at 77 K. **CMP-1** and **CMP-2** exhibited type-II iso-thermal curve, and the BET areas were only 23.67 m^2/g and 8.56 m^2/g respectively. The low BET areas of **CMP-1** and **CMP-2** were speculated to be related the non-porous structure of pillar[5]arene, which could also be found in some previous reports [47]. **COP-1** exhibited type-IV isothermal curve with hysteresis loop at high absolute pressure value which indicated complex micro-mesoporous structure and the BET areas was 482.63 m^2/g. The pore volume of **COP-1** was 0.12 cm^3/g, while the ones of **CMP-1** and **CMP-2** were only 0.007 cm^3/g and 0.0012 cm^3/g respectively. The pore size distributions of polymers were calculated by nonlocal density functional theory (NLDFT) method (Fig. S2c-d in Supporting information). Thermogravimetric analysis (TGA) revealed that the polymers were stable at 300 °C (Fig. S2b in Supporting information). UV-vis diffuse reflectance (DRS) spectra showed that the absorption bands of the polymers extended to the visible light region (Fig. 1c), indicating the possible use of the polymers as photocatalysts under visible light irradiation.



Scheme 1. The synthetic route of the **CMP-1** (a); the chemical structures of **CMP-2** and **COP-1** (b); the chemical structures of **S-1** and **S-2** (c).

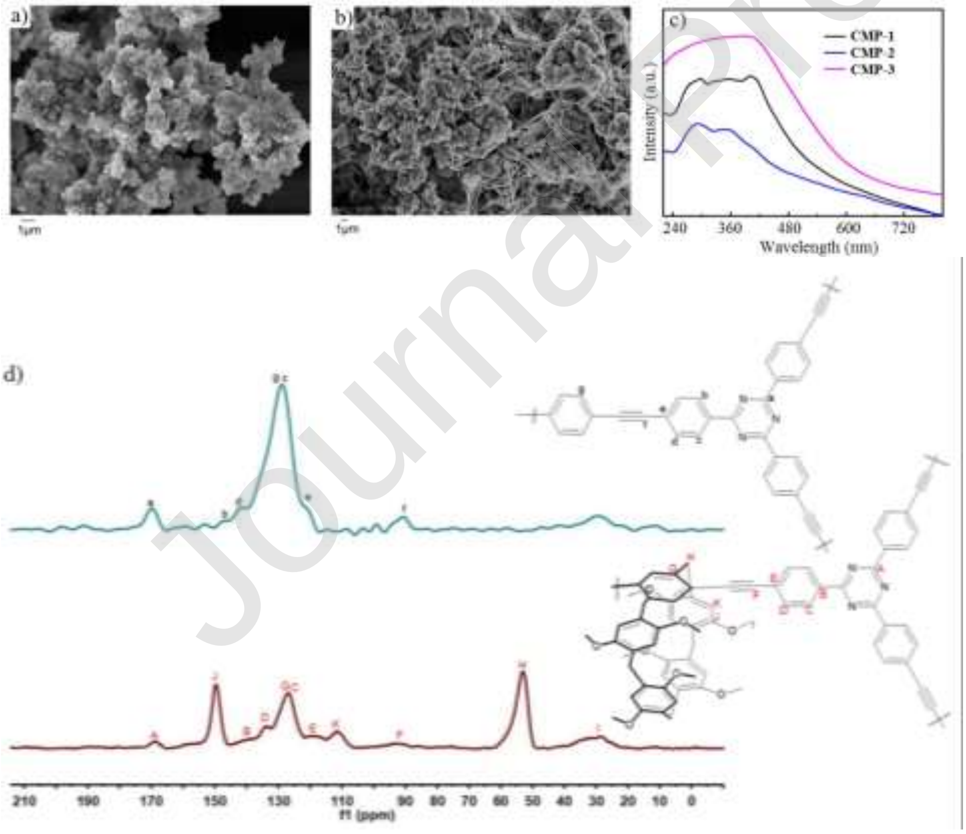
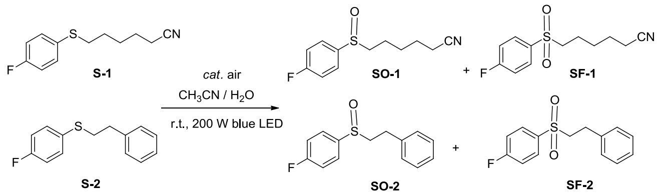


Fig. 1. SEM images of **COP-1** (a) and **CMP-1** (b); UV-vis diffuse reflectance (DRS) spectra of **CMPs** and **COP-1** (c); ^{13}C CP-MAS NMR of **COP-1** and **CMP-1** (d).

Photocatalytic performances of the polymers toward the oxidation of sulfides with different substituents were investigated. The experiments were carried out at room temperature, with a 200 W blue LED lamp (420 - 460 nm) as light source, using open vessels under air, with photo-irradiated time for 10 h, 20 h and 30 h. The conversion yields of the sulfides were calculated by a ^{1}H NMR spectroscopic method [63], using the formula of Conv.(%) (**SO-1** or **SO-2**) = Integration b/Integration [(a + b + c)] × 100% and Conv.(%) (**SF-1** or **SF-2**) = Integration c/Integration [(a + b + c)] × 100%, where “a”, “b”, “c” stand for the integrated peak area of characteristic H of sulfides, sulfoxides, and sulfones in the corresponding ^{1}H NMR spectra, respectively (details in Supporting information). The conversion yields of the sulfides with photo-irradiated time of 10 h, 20 h and 30 h were shown in Table 1. With the photo-irradiated time increased from 10 h to 30 h, the conversion yield of **S-2** to the corresponding sulfoxide (**SO-2**) catalyzed by both pillar[5]arene-based **CMPs** kept lower than 10%, while the conversion yield of **S-1** to **SO-1** increased with the increase of irradiation time and finally reached 80% in 30 h irradiation (Figs. S26-39 in Supporting information). The conversion yields of **S-2** and **S-1** to their corresponding sulfoxides catalyzed by **COP-1** increased with the irradiation time and reached 93% and 92%, respectively, in 30 h irradiation. The substrate selectivity was examined for pillar[5]arene-based **CMPs** catalysts as well as **COP-1**. It can be found that when **COP-1** was used as a catalyst, both sulfides (**S-1** and **S-2**) showed similar conversion yields after 30 h irradiation (92% and 93%). However, when catalyst **CMP-1** was used, the conversion yields of **S-1** and **S-2** exhibited significant difference. The conversion yield of **S-1** to **SO-1** was 80%, but **S-2** to **SO-2** was only 10%. The conversion yield ratio for **S-1**:**S-2** was 8:1. Similar phenomenon could be found in the case of catalyst **CMP-2**, with the conversion yields of **S-1** to **SO-1** and **S-2** to **SO-2** being 53% and 3%, respectively, and a conversion yield ratio for **S-1**:**S-2** was nearly 18 : 1. Prolonged irradiation time after 30 h did not increase the conversion yields of the products. **CMP-1** showed a higher photocatalytic activity than **CMP-2**, which might result from the triazine units of **CMP-1** that cause better electron-hole separation. Interestingly, in the cases of **CMP-1** and **CMP-2**, the conversion yields of **S-1** and **S-2** seemed to be proportional to the binding strength of pillar[5]arene toward the sulfides. As reported before, pillar[5]arenes can effectively bind linear dinitriles with the binding constant (*K*a) of 10^4 L/mol for hexanedinitrile in $CDCl_3$ [46-47, 64]. Phenyl group is too larger to be included by the pillar[5]arene cavity [43, 47].

Table 1

The photocatalyzed oxidization reaction of sulfides in the air.[a]



Entry	Catalyst	Sulfide	Conv. (%)					
			10 h		20 h		30 h	
			SO	**SF**	**SO**	**SF**	**SO**	**SF**
1	**CMP-1**	**S-1**	31	-	74	2	80	2
2	**CMP-1**	**S-2**	-	-	-	-	10	-
3	**CMP-2**	**S-1**	23	1	38	2	53	1
4	**CMP-2**	**S-2**	-	-	-	-	3	-
5	**COP-1**	**S-1**	36	-	79	4	92	4
6	**COP-1**	**S-2**	38	1	91	5	93	1
7[b]	-	**S-1**	-	-	-	-	-	-
8[b]	-	**S-2**	-	-	-	-	-	-

[a] Reaction conditions: sulfide (1.0 mmol), CMPs/COP (20.0 mg), in a mixed solvent of CH_3CN/H_2O (6.0 mL, *v/v* 2:1) under air at room temperature, irradiated with a 200 W blue LED lamp as the light source.

[b] Blank tests, irradiation time: 30 h, without catalysts but same reaction conditions, the conversion was hard to detect.

To confirm the presumption that the host-guest interaction induced catalytic difference in the **CMPs** photocatalytic systems, corresponding oligomers of the **CMPs** and **COP-1** (**O-CMP-1**, **O-CMP-2** and **O-COP-1**) were prepared and ^{1}H NMR experiments were conducted (Fig. 2a and Figs. S3a-b in Supporting information). Upon addition of **O-CMP-1** to the $CDCl_3$ solution of **S-1**, upfield shift and broadening for the proton signal of **S-1** was observed, suggesting the existence of host−guest interaction between **S-1** and the pillar[5]arene moieties of **O-CMP-1**. Such a change was not observed in the ^{1}H NMR spectrum of **S-1** upon addition of **O-COP-1**, indicating no interaction existed between **S-1** and **O-COP-1**. In the case of **S-2**, no change could be observed in its ^{1}H NMR spectrum upon addition of both **O-CMP-1** and **O-COP-1** (Fig. S3b), implying no interaction existed between **S-2** and **O-CMP-1** (**O-COP-1**). Similarly, host-guest interaction can be observed in the ^{1}H NMR spectrum of **S-1** and **O-CMP-2**, but not in the ^{1}H NMR spectrum of **S-2** and **O-CMP-2**.

Competitive experiments were used to evaluate the host-guest effect of pillar[5]arenes on the photocatalytic oxidation of **S-1** (Fig. 2b). Butanenitrile (**BN**), a well-known guest molecule for pillar[5]arenes [65,66], was used as a competitive guest molecule to bind the pillar[5]arene struts, weakening the interaction of the **CMPs** and **S-1**. As expected, by introducing excess **BN** into the system of **CMP-1** and **S-1**, a remarkable change was observed in the conversion yield of **S-1**, dropping from 80% to 18%. Similar phenomenon can be found in **CMP-2** involved photocatalytic system. However, under the same reaction conditions, no obvious change was observed in the conversion yield of **S-1** when **COP-1** was used as a photocatalyst. Therefore, we could concluded that the host-guest interaction of pillar[5]arene moieties in the **CMPs** with the substrate was vital to catalytic performance of the **CMPs**.

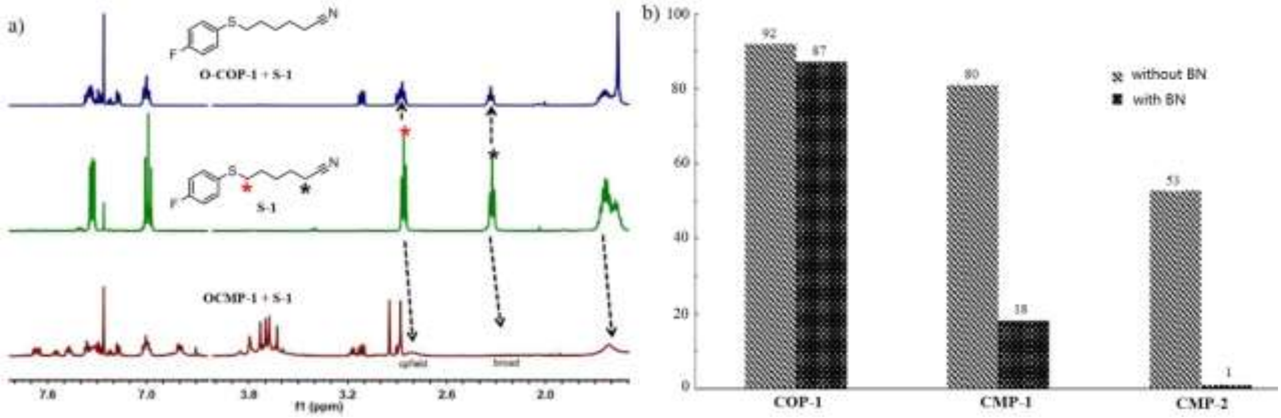
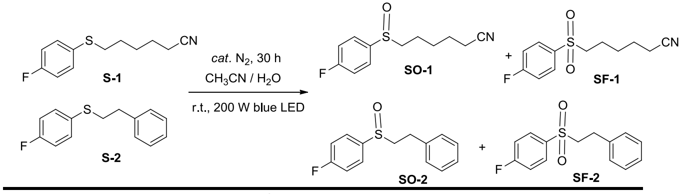


Fig. 2. Partial ^{1}H NMR ($CDCl_3$, 500MHz) spectra of **S-1** (5.0 mg), **S-1** (1.0 mg) + **O-COP-1** (5.0 mg) and **S-1** (1.0 mg) + **O-CMP-1** (5.0 mg) (a); The conversion yield of **S-1** to product **SO-1** with(out) the presence of excess butanenitrile (b).

Table 2

The photocatalyzed oxidization reaction of sulfides in the nitrogen with photo-irradiated time for 30 h.[a]



Entry	Catalyst	Sulfide	Conv. (%)	
			SO	SF
1	**CMP-1**	**S-1**	41	3
2	**CMP-1**	**S-2**	8	-
3	**CMP-2**	**S-1**	-	-
4	**CMP-2**	**S-2**	-	-
5	**COP-1**	**S-1**	88	3
6	**COP-1**	**S-2**	90	-

[a] Reaction conditions: sulfide (1.0 mmol), CMPs/COP (20.0 mg), with the solvent of CH_3CN/H_2O (6.0 mL, *v/v* 2:1), in the nitrogen at room temperature, irradiated with a 200 W blue LED lamp as the light source.

Electron spin resonance (ESR) was used to explore the photocatalytic mechanism of the polymers on oxidation of sulfides under visible light. In an O_2 saturated toluene solution, with 5,5-dimethyl-1-pyrroline N-oxide (**DMPO**) as the probe, the polymers all displayed clear four-line spectra of **DMPO-•OH** adducts after blue light irradiation, and the corresponding signal intensity increased with the irradiation time, indicating that all the polymers could generate •OH under visible light irradiation. By changing the spin trap matter from **DMPO** to 2,2,6,6-tetramethylpiperidinyloxyl (**TEMPO**), three-line spectra, which were the characteristic signal for the spin adduct formed by **TEMPO** and $^1O_2$, were observed in the systems (Figs. S4a-d in Supporting information), indicating that the polymers could generate $^1O_2$ under visible light irradiation. •OH and $^1O_2$ are all reactive oxygen species which can attack the S group in the oxidation reaction of sulfides.

As •OH either originated from the solvent H_2O or desolved O_2, ESR spectra in N_2 saturated ACN/H_2O were examined. Except the **CMP-2/DMPO** system, clear four-line spectra of **DMPO–•OH** adducts were detected in the systems of **COP-1/DMPO** and **CMP-1/DMPO** after blue light irradiation, indicating that **COP-1** and **CMP-1** could efficiently convert H_2O to •OH under visible light irradiation (Figs. S4e-g in Supporting information). Photocatalytic performance of all the polymers in the oxidation of **S-1** and **S-2** under N_2 atmosphere were assessed (Table 2). The result showed that except **CMP-2**, **CMP-1** and **COP-1** possessed certain catalytic activity under N_2 atmosphere toward the oxidation of sulfides, but the conversion yields were relatively lower than the ones in the air. The results were consistent with the ESR, indicating **COP-1** and **CMP-1** could photocatalytically convert H_2O to •OH to oxidize the sulfides to sulfoxides.

Based on the above observations, a mechanism for **CMP-1** (**COP-1**) involved photocatalytic oxidation reaction was proposed. Under light irradiation, electron-hole pairs are generated in the polymers which activate H_2O or solvated O_2 to produce active oxygen species, the active oxygen species can subsequently oxidize the sulfides to their corresponding sulfoxides. Accessibility of the active oxygen species to the sulfide substrates is crucial to catalytic efficiency. In the system of **CMP-1**, pillar[5]arene motifs tend to bind guest-like sulfide substrates and increase the availability of activated oxygen species to the bonded sulfides. Consequently, the oxidation rate of guest-like sulfide (**S-1**) is accelerated. In the system of **COP-1**, no selective binding exists, and therefore similar photocatalytic activities of **COP-1** toward both sulfides were observed.

In summary, we have demonstrated a host-guest interaction strategy to endow photocatalysts with special selectivity. By adopting conjugated macrocycle polymers with pillar[5]arene struts (**CMP-1** and **CMP-2**), enhanced oxidation efficiency of the guest-like substrate (**S-1**) over non-guest-like substrate (**S-2**) was observed. Mechanism studies confirmed that the "host-guest" nature of pillar[5]arene struts in the **CMPs** was the main cause of the difference. The present work will open up new opportunities in design of highly efficient organic semiconductor catalysts with reactant selectivity. Related work is ongoing in our lab.

Declaration of interests

√ 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.

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