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# Dynamically switchable porphyrin-based molecular tweezer for on–off fullerene recognition ☆

Zhimin Sun, Xin-Hui Guo, Yue Zhao, Qing-Yu Meng, Li-Juan Xing*, He-Lue Sun*

*College of Chemistry and Materials Science, Hebei Key Laboratory of Organic Functional Molecules, Hebei Normal University, Shijiazhuang 050024, China*

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ABSTRACT

Herein, a novel molecular tweezer based on 2,2′-bipyridine-bridged porphyrin subunits was constructed for efficient fullerenes recognition. The *syn* conformation of the molecule, which was obtained by Zn(II) coordination, gives rise to a proper cavity to interact with fullerene guests to form a stable 1:1 complex in toluene solution. It exhibits distinct binding selectivity towards $C_{60}$ over $C_{70}$. Moreover, the fullerene recognition capacity can be adequately suppressed by importing $H_2PO_4^-$ to competitively capture Zn(II) along with *syn-anti* conformational conversion. Subsequently, the molecular tweezer regenerated to bind the fullerene by introducing the $Ca^{2+}$ into the system. Significantly, the association-disassociation process can be switched reversibly and repeatedly.



Motivated by widespread biological switches such as protein tyrosine phosphatases (PTPs), protein tyrosine kinases (PTKs), and GTPases, the construction of artificial molecular switches (AMSs), displaying dynamic stimuli-responsive behaviors, has a long history of capturing the full attention of scientists [1–5]. From the characteristics of AMS, it is considered that molecular tweezers with specific on–off switching properties are excellent candidates for building AMSs [6]. During the last decades, various molecular tweezers [7–13] have been developed *via* non-covalent interactions such as $\pi$–$\pi$ interactions, metal–ligand coordination, ionic interactions and hydrogen bonding, which have shown fascinating applications in molecular recognition, optical switch and complex structure construction. As well known, precise regulation the conformation of the molecular tweezer is the key point in realizing their dynamic on–off switching properties. In this respect, the 2,2′-bipyridine ligand, whose conformation was able to interconvert between its *anti* and *syn* pattern when it coordinated with suitable metal ions, is an ideal allosteric center for constructing dynamic molecular tweezer [14,15].

Fullerenes are well-known carbon allotrope with defined numbers of $Csp^2$ atoms, which own plenty of potential applications in multiple research areas [16–19]. Molecular tweezers as fullerene receptors have been thoroughly investigated due to their potential applications in the field of material science [20,21], photosynthetic techniques [22–24], and biochemistry [25,26]. More importantly, the development of dynamic molecular tweezers with on–off fullerenes recognition ability will promote the research of fullerenes. Porphyrins and their derivatives have a wide range of applications and functions regarding molecular recognition [27,28], solar cells [29–31], radical-based materials [32,33], photochemical catalysis [34], and optoelectronic materials [35]. Attributing to the highly delocalized $\pi$–electron rich characteristic, porphyrins can bind electron–deficient fullerenes through $\pi$–$\pi$ interactions in the solid state [36]. Notably, when introducing porphyrin units to jaw- or pocket-like structures, the binding affinity to fullerenes can be dramatically enhanced as the result of the chelate effect for bidentate complexation [37,38]. The donor-acceptor property of the porphyrin-fullerene combination had numerous superiorities in establishing photovoltaic solar cells for mimicry of natural photosynthesis.

To develop porphyrins-based molecular tweezers as fullerenes receptors should take the following objectives into consideration: (1) high binding ability towards fullerenes, (2) distinct selectivity for either $C_{60}$ or $C_{70}$, and (3) dynamic performance with on–off switchable behavior. One of the possible approaches to endow the dynamic switchable property is introducing coordination units into molecular tweezers structures. Controlling the coordination and decoordination processes, their binding ability could be adjusted as the conformation fluctuated. To attain this objective, we anticipated that molecular tweezers with 2,2′-bipyridine ligand and porphyrin units could exhibit high affinity to fullerenes and dynamic switchable molecular recognition ability. When the 2,2′-bipyridine ligands coordinated with metal ions like Zn(II) and resulted in *syn*

☆ This paper is dedicated to Prof. Yu Liu on the occasion of his 70th birthday.

* Corresponding authors.
*E-mail addresses:* xinglijuan@hebtu.edu.cn (L.-J. Xing), heluesun@hebtu.edu.cn (H.-L. Sun).

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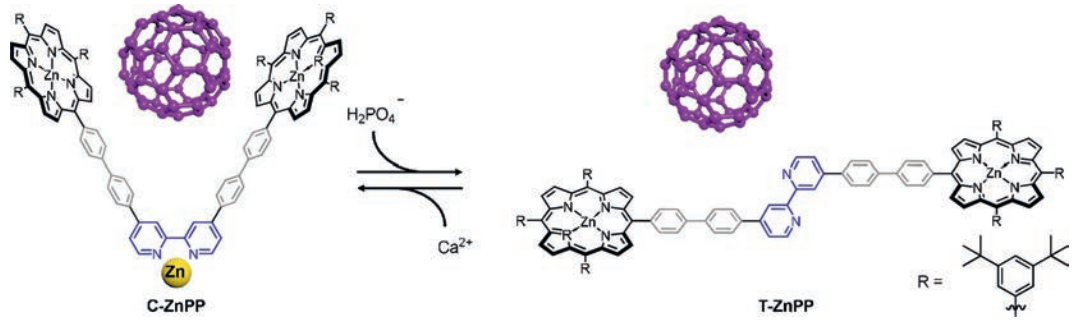
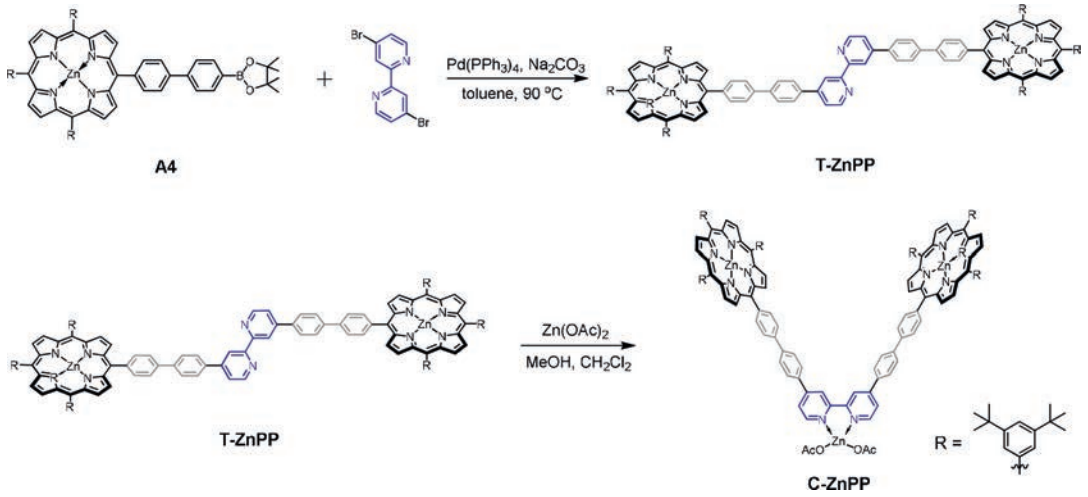



**Fig. 1.** Structures of C-ZnPP, and T-ZnPP and illustration of the molecular tweezer on and off fullerene recognition.




**Scheme 1.** Synthetic routes of T-ZnPP and C-ZnPP.

conformation, molecular tweezers were in the active model with an advisable cavity for fullerenes binding. After the treatment with certain chemicals (specific anion like $H_2PO_4^-$ could competitively bind with Zn(II)) that lead to the decoordination process, molecular tweezers could be unfolded into *anti*−conformation with the binding ability losing. Molecular tweezers could re-recognize with fullerenes after $H_2PO_4^-$ precipitated by $Ca^{2+}$ [39]. The dynamic porphyrin-based molecular tweezer's reversible recognition of fullerenes capability was adjusted by successively introducing $H_2PO_4^-$ and $Ca^{2+}$ (Fig. 1).

As shown in Scheme 1, *anti*-conformation **T-ZnPP** was designed and synthesized in good yield through a Suzuki coupling reaction between 4,4′-dibromo-2,2′-bipyridine and biphenyl-substituted zinc porphyrin, which was further characterized by $^1$H NMR, $^{13}$C NMR, and mass (HRMS) spectroscopy (Figs. S14-S16 in Supporting information). The molecular tweezer **C-ZnPP**, in *syn*-conformation, was easily prepared *in situ* by zinc acetate coordinating with **T-ZnPP** (Scheme 1). As for the complex **C-ZnPP**, downfield and upfield shifts of the signals corresponding to protons $H^3$ ($\Delta\delta = 0.04$ ppm), $H^5$ ($\Delta\delta = -0.31$ ppm), and $H^6$ ($\Delta\delta = -0.49$ ppm) of the 2,2′-bipyridine parts indicated the fully sufficient conversion from **T-ZnPP** to **C-ZnPP** (Fig. S19 in Supporting information).

With the molecular tweezer (**C-ZnPP**) in hand, a series of UV−vis and fluorescence titration experiments were performed to verify the recognition capability of **C-ZnPP** to the fullerenes. Compound **C-ZnPP** exhibited an intense Soret band at 427 nm and two less intense Q-bands in the 500−700 nm region. As shown in Fig. 2a, upon adding $C_{60}$ with progressive concentrations (0−70 equiv.) to $0.5 \times 10^{-6}$ mol/L solutions of **C-ZnPP** in toluene at 25 °C, the Soret band at 426 nm withstood a slight bathochromic shift accompany with an obvious isosbestic point (431 nm). Small

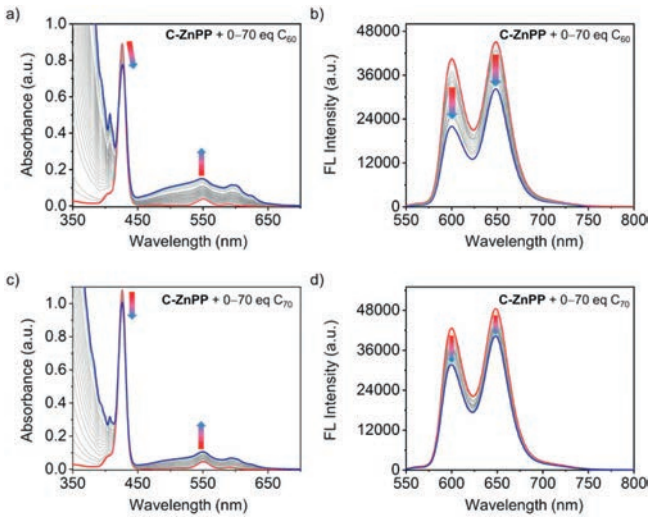



**Fig. 2.** UV−vis titration spectra of a toluene solution of C-ZnPP ($0.5 \times 10^{-6}$ L/mol) with addition of 0−70 equiv. of $C_{60}$ (a) and $C_{70}$ (c). Fluorescence titration spectra ($\lambda_{ex} = 427$ nm) of a toluene solution of C-ZnPP ($0.5 \times 10^{-6}$ L/mol) with addition of 0−70 equiv. of $C_{60}$ (b) and $C_{70}$ (d).

bathochromic shifts of the Soret bands of porphyrins were commonly observed on complexation of fullerenes, which here demonstrated typical $\pi-\pi$ interaction between **C-ZnPP** and $C_{60}$, lowering the energy of the porphyrin $\pi$ to $\pi*$ transition [40]. Moreover, intensities of Q-bands were gradually increased due to the charge transfer interactions between porphyrin units and fullerenes [41].

The Job's plot analysis revealed the stoichiometry of **C-ZnPP** and $C_{60}$ was 1:1 (Fig. S20a in Supporting information). The binding constant ($K_a$) for the formation of **C-ZnPP**⊃$C_{60}$ complex was $1.05 \times 10^4$ L/mol, which was evaluated (1:1 mode) spectrophotometrically by UV–vis titration data (Table S1 in Supporting information) [42]. The similar UV–vis titration experiments were conducted for **C-ZnPP** and $C_{70}$. As shown in Fig. 2c, the Soret band at 427 nm underwent barely shifts and a little hypochromicity with the addition of $C_{70}$ into the toluene solution of **C-ZnPP**, which suggested the binding ability was not as strong as that of **C-ZnPP** with $C_{60}$. The binding stoichiometry of **C-ZnPP** and $C_{70}$ was 1:1 (Fig. S20b in Supporting information) according to the corresponding Job's plot analysis (Fig. S20b), and the $K_a$ value for the formation of complex **C-ZnPP**⊃$C_{70}$ was $2.38 \times 10^3$ L/mol calculated from the UV–vis titration results using a 1:1 model (Table S1). Thus, the molecular tweezer **C-ZnPP** exhibited reasonable recognition capability towards to fullerenes ($C_{60}$ and $C_{70}$) and visible binding selectivity to $C_{60}$. The fluorescence titration experiments were also accomplished to estimate the binding ability of **C-ZnPP** to fullerenes. It is generally reported that porphyrin fluorescence is quenched when fullerenes approach, as a result of photoinduced electron transfer between $^1$S of the porphyrins and fullerenes [43]. Upon addition of $C_{60}$ and $C_{70}$ to the toluene solution of **C-ZnPP** respectively, conspicuous quenching effect occurred when exciting at 427 nm as a result of complex **C-ZnPP**⊃$C_{60}$ and **C-ZnPP**⊃$C_{70}$ formation (Figs. 2b and d). Notably, the fluorescence measurements showed a larger quenching of the excited state of **C-ZnPP** by $C_{60}$ than $C_{70}$, which also reveals the binding selectivity that was consistent with the UV–vis titration results as mentioned above.

To gain further insight into the mechanism of the molecular tweezer **C-ZnPP** specifically binding with fullerenes, similar UV−vis and fluorescence titration experiments for **T-ZnPP** (*anti*-conformation) and control molecule **2HPP** (no zinc in porphyrin units) were also performed. As for **T-ZnPP**, with the separate addition of fullerenes (0−50 equiv. of $C_{60}$ or $C_{70}$), the Soret band at 427 nm sustained no shifts and a slight lowering in intensity (Figs. S21a and c in Supporting information). Meanwhile, there was only a modest decrease in fluorescence emission intensity, which implied the fullerenes binding ability of **T-ZnPP** was relatively weak (Figs. S21b and d in Supporting information). Therefore, the UV–vis and fluorescence titration results affirmed that the *syn*-conformational structure of **C-ZnPP** played crucial roles in fullerenes recognition. **C-ZnPP**, which possesses the tweezer-like bis-porphyrin-based structure could tightly bind with fullerenes due to the appropriate cavity size between two porphyrin clefts for accepting fullerenes, while *anti*-conformational **T-ZnPP** does not. What is more, upon the separate addition of fullerenes (0−50 equiv. of $C_{60}$ or $C_{70}$) to a toluene solution of **2HPP**, there was no obvious change in the UV–vis and fluorescence titration data (Fig. S22 in Supporting information). It is well known that metalloporphyrins have a larger $\pi$−conjugated systems with 18 electrons, which are excellent components for binding with $\pi$−acceptor fullerenes [44]. **2HPP**, without metal insertion, basically has extremely weak fullerenes recognition capability.

Diffusion ordered spectroscopy (DOSY) experiments were also conducted to prove the formation of supramolecular complexes between **C-ZnPP** and fullerenes ($C_{60}$ or $C_{70}$). With the addition of 1 equiv. of $C_{60}$ to a $1.5 \times 10^{-3}$ mol/L toluene solution of **C-ZnPP**, signals of **C-ZnPP**⊃$C_{60}$ complex were detected with a diffusion coefficient of $2.15 \times 10^{-6}$ cm$^2$/s, which was same as signals ($D = 2.14 \times 10^{-6}$ cm$^2$/s) of free molecular tweezer **C-ZnPP** within error (Fig. S23 in Supporting information). It was proved that **C-ZnPP** can bind with $C_{60}$ to form a 1:1 complex and maintain its cavity size [8,45]. Furthermore, the diffusion coefficient of **C-ZnPP**⊃$C_{70}$ ($D = 1.86 \times 10^{-6}$ cm$^2$/s) was smaller than **C-ZnPP**⊃$C_{60}$ complex, which could be believed that **C-ZnPP** were expanded due to its insufficient cavity for $C_{70}$. The diffusion coefficient data of **C-ZnPP**⊃$C_{60}$ and **C-ZnPP**⊃$C_{70}$ complexes demonstrated that the cavity size of **C-ZnPP** was more suited for $C_{60}$ than $C_{70}$, explaining why it had superior selectivity towards $C_{60}$ as determined by the binding constant, UV–vis, and fluorescence titration results previously.

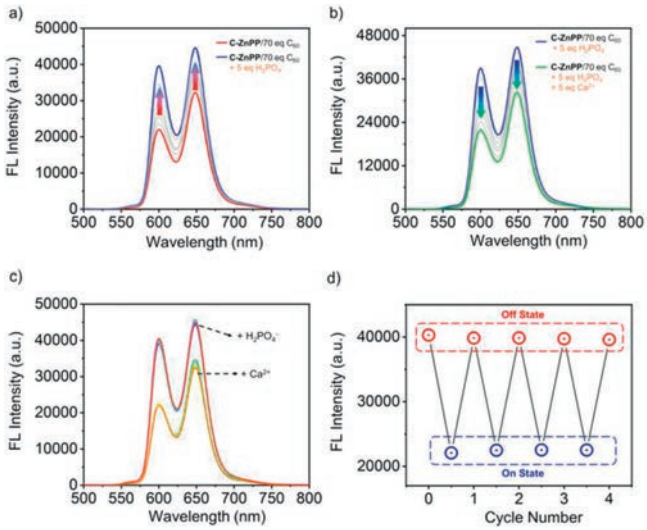



**Fig. 3.** Fluorescence spectra ($\lambda_{ex} = 427$ nm) of the mixture of C-ZnPP ($0.5 \times 10^{-6}$ mol/L) and 70 equiv. of $C_{60}$ in toluene with the addition of 0−5 equiv. of $H_2PO_4^-$ (a), and the mixture of C-ZnPP ($0.5 \times 10^{-6}$ mol/L), 70 equiv. of $C_{60}$, and 5 equiv. of $H_2PO_4^-$ with the addition of 0−5 equiv. of $Ca^{2+}$ (b). (c) Fluorescence spectra ($\lambda_{ex} = 427$ nm) of the mixture of C-ZnPP ($0.5 \times 10^{-6}$ mol/L) and 70 equiv. of $C_{60}$ with the sequentially cyclical addition of 5 equiv. of $H_2PO_4^-$ and $Ca^{2+}$. (d) Reversible cycling of the fluorescence emission intensity at 576 nm of four consecutive association-dissociation processed by successively adding $H_2PO_4^-$ and $Ca^{2+}$.

Thereafter, a series of ion-controlled coordination/decoordination experiments were carried out to investigate the switchable fullerene recognition ability of molecular tweezer **C-ZnPP.** As mentioned above, the significant fluorescence quenching phenomenon occurred when excessive $C_{60}$ added into the toluene solution of **C-ZnPP**. As shown in Fig. 3a, following the introduction of 0−5 equiv. $H_2PO_4^-$ (tetrabutylammonium salt) to a toluene solution of **C-ZnPP**/70.0 equiv. $C_{60}$ mixture, it was found that the quenched fluorescence emission of **C-ZnPP** by $C_{60}$ was reasonably restored. It is inferred that Zn cation, which coordinated with the 2,2′-bipyridine components of **C-ZnPP**, was gradually competitively replaced by $H_2PO_4^-$, and **C-ZnPP** was transformed to **T-ZnPP** bearing *anti*-conformation accompany with the binding ability to $C_{60}$ restraining. Therefore, the fluorescence emission intensity of the mixture solution was recovered. When 5 equiv. of $H_2PO_4^-$ was added, the emission intensity was able to return to its original state and kept the stated steady as up to 10 equiv. of $H_2PO_4^-$ was introduced (Fig. S24 in Supporting information). Moreover, the $C_{60}$ recognition on−off behaviors of **C-ZnPP** were further monitored by $^1$H NMR spectroscopy in toluene-$d_8$ (Fig. 4a). Upfield shifts ($\Delta\delta = 0.051$ ppm) of the signals corresponding to the $\beta$ protons of porphyrin units of **C-ZnPP**⊃$C_{60}$ complex by virtue of shielding effect which compared to free **C-ZnPP** indicated that $\pi$–$\pi$ stacking interactions between **C-ZnPP** and $C_{60}$. When adding $H_2PO_4^-$ to the **C-ZnPP**⊃$C_{60}$ complex solution, it was found that signals for $\beta$ protons suffered downfield shifts ($\Delta\delta = 0.046$ ppm) and came back to the same position as the **C-ZnPP** itself, within error, due to the structural transformations of the molecular tweezer from *syn* to *anti* conformation. $^{13}$C NMR spectroscopy is also very practical for disclosing the details of fullerenes complexation, since $C_{60}$ has a unique signal. Obvious upfield shifts ($\Delta\delta = 0.15$ ppm) of the signals ascribing to $C_{60}$

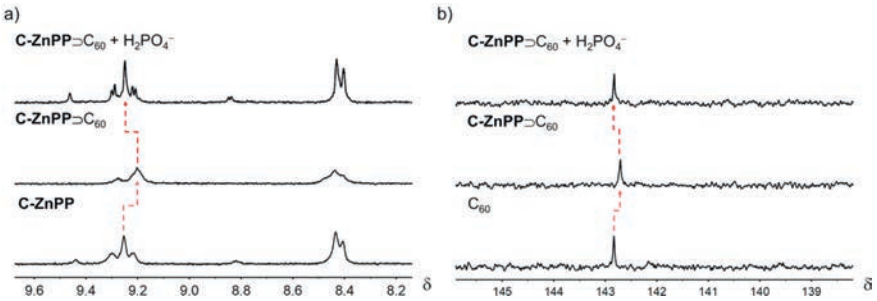



**Fig. 4.** (a) Stacked $^{1}$H NMR spectra (400 MHz, toluene-$d_8$, 298 K) of a $1 \times 10^{-3}$ mol/L solution of C-ZnPP, C-ZnPP with 1 equiv. of $C_{60}$, and 1:1 mixture of C-ZnPP and $C_{60}$ with 1 equiv. of $H_2PO_4^-$. (b) Stacked $^{13}$C NMR spectra (100 MHz, toluene-$d_8$, 298 K) of a $1 \times 10^{-3}$ mol/L solution of C-ZnPP with 1 equiv. of $C_{60}$, $C_{60}$, and 1:1 mixture of C-ZnPP and $C_{60}$ with 1 equiv. of $H_2PO_4^-$.

belonging to **C-ZnPP**⊃$C_{60}$ complex were found compared to free $C_{60}$ because of ring current effects of porphyrins (Fig. 4b). Thus, the upfield shift should be the evidence for effective complexation between **C-ZnPP** and $C_{60}$ [24,37]. Subsequently, upon the addition of $H_2PO_4^-$ to **C-ZnPP**⊃$C_{60}$ complex solution, signals of $C_{60}$ moved to downfield and returned to the free $C_{60}$ state due to interactions between **C-ZnPP** and $C_{60}$ diminishing. In general, $^{1}$H and $^{13}$C NMR spectroscopy results were consistent with the switchable ion-controlled fullerene recognition process as assumed and in line with the conclusion obtained by UV−vis and fluorescence spectroscopy experiments.

In light of the relatively poor solubility of $Ca(H_2PO_4)_2$ in toluene, $Ca^{2+}$ was employed to capture $H_2PO_4^-$ and reproduce the *syn* conformational **C-ZnPP** with the $C_{60}$ binding ability recovery. The fluorescence titration experiments showed that upon 0−5 equiv. of $Ca(ClO_4)_2$ were progressively added to a toluene solution of **C-ZnPP**/70 equiv. $C_{60}$/5 equiv. $H_2PO_4^-$ mixture, a distinct quenching effect took place, which suggested the regenerate of **C-ZnPP** (Fig. 3b). It is worth to mention that $H_2PO_4^-$ or $Ca^{2+}$ have no interactions with the zinc ions from the porphyrin units based on the UV–vis and fluorescence spectra of **2HPP**, **T-ZnPP**, and **T-ZnPP** with separate addition of $H_2PO_4^-$ or $Ca^{2+}$ (Fig. S25 in Supporting information). With the addition of $H_2PO_4^-$ or $Ca^{2+}$ to **T-ZnPP** solution, it exhibited characteristic two Q-bands of metalloporphyrins in the 500−700 nm region, whereas porphyrins, like **2HPP**, exhibited four Q-bands (Fig. S25a). What's more, when introducing $H_2PO_4^-$ or $Ca^{2+}$ to **T-ZnPP** solution, the emission $\lambda_{max}$ of around 597 nm was still the same (Fig. S25b). Generally, the whole dissociation−association of $C_{60}$ by **C-ZnPP** process controlled through alternately adding $H_2PO_4^-$ and $Ca^{2+}$ was identified to be effectively repeatable with the excellent reversibility (Figs. 3c and d).

In conclusion, we designed and synthesized a novel molecular tweezer based on the 2,2′-bipyridine-bridged zinc-porphyrin in *syn*-conformation, which has the appropriate cavity to adequately bind the fullerene forming 1:1 supramolecular complex. It exhibited distinct binding selectivity towards $C_{60}$ ($K = 1.05 \times 10^4$ L/mol) over $C_{70}$ ($K = 2.38 \times 10^3$ L/mol). Furthermore, the fullerene recognition capacity of the molecular tweezer was able to be switched by introducing $H_2PO_4^-$ and $Ca^{2+}$ successively. Upon the addition of $H_2PO_4^-$, the molecular tweezer could undergo a *syn*−*anti* conformational conversion with the binding ability vanishing. After adding $Ca^{2+}$ to competitively capture $H_2PO_4^-$, the molecular tweezer regenerated and the fullerene binding ability was fully recovered. More importantly, the molecular tweezer underwent conformational conversion between its *syn*- and *anti*-form accompany with the association-dissociation process reversibly and repeatably, which were demonstrated by $^{1}$H NMR, $^{13}$C NMR, UV−vis, and fluorescence spectroscopy analyses. This dynamically switchable molecular tweezer with on−off fullerene recognition ability is expected to be used in the field of fullerene separation and donor−acceptor photovoltaic solar cells.

## 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.

## Acknowledgments

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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.2023.109162.

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