



## Communication

# Metal-organic supramolecular nanoarchitectures by Ru(II) bis-(terpyridine)-bridged pillar[5]arene dimers with triphenylamine

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## ARTICLE INFO

## Article history:

Received 4 January 2020

Received in revised form 2 February 2020

Accepted 13 February 2020

Available online 14 February 2020

## Keywords:

Supramolecular chemistry

Pillar[5]arene

Self-assemble

Tunable PET

Host-guest complexation

## ABSTRACT

A metal-organic photoinduced electron transfer (PET) supramolecular nanoarchitecture comprised of Ru(II) bis(terpyridine)-modified pillar[5]arene (electron acceptor) and triazole triphenylamine amyl cyanide (electron donor) has been designed and constructed. Through the comparison of diverse solvents and acceptors, the two conditions, *i.e.*, modification of pillar[5]arene to donor and weak polar solvent are benefit for the occurrence of “efficient PET” because of shorter D-A distance in the presence of pillar[5]arene. Crucially, the fluorescence and PET process of the supramolecular assembly could be further modulated by solvent conversion and another competitive guest. The study provides a supramolecular method to design and construct tunable PET systems and PET-based smart materials.

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Self-assembly exists universally in chemistry, materials science and biology. Supramolecular self-assembly based on host-guest chemistry has become an important research topic in recent years due to its wide application in the construction of self-healing materials [1], fluorescence resonance energy transfer (FRET)-based light-harvesting devices [2], molecular machine [3], controlled release [4], photoinduced electron transfer (PET) systems [5] and so on. Since Ogoshi and co-workers first synthesized *para*-bridged symmetrical pillar[5]arenes in 2008 [6], pillararenes (especially pillar[5]arene and pillar[6]arene) as the fifth generation macrocyclic hosts after crown ether, cucurbituril, cyclodextrin and calixarene are booming. Pillararene has been usually utilized as a host media to construct host-guest complexes and supramolecular polymers [7]. Huang *et al.* developed a nanoparticle of pillararene-induced near-infrared emission enhancement through host-guest complexation, showing effective PH-responsive ability [8]. Wang *et al.* developed multiple-stimuli-responsive supramolecular vesicles based with pillar[6]arene and SAINT, achieving controllable release of an anticancer drug, *i.e.*, DOX [9]. Yang's group constructed a [2]biphenyl-extended pillar[6]arene/tetraphenylethylene-based supramolecular polymer, achieving assembly-induced emission enhancement for efficient mercury(II) detection and removal [10]. Meanwhile, the group also developed

a supramolecular polymer network based on a monofunctionalized leaning tower[6]arene, exhibiting tunable fluorescence characteristic [11].

On the other hand, PET reactions are prominent processes in nature and many areas of science and technology, among which are solar energy conversion, photocatalysis, synthetic organic photochemistry, super-resolution imaging, and photodynamic therapy [12]. Supramolecular systems based on PET attracted researchers' extensive attention for easy construction, stimuli-adjustment, conducive to tracking, and so on. Recently, Liu *et al.* reported a series of PET-based supramolecular architectures mediated by calixarene and cyclodextrin [13]. However, only a few of pillararene-mediated PET systems have been exploited. For instance, Stoddart *et al.* constructed porphyrinic supramolecular daisy chains incorporating pillar[5]arene and viologen, where PET process from porphyrin to viologen occurred [14]. Electron-rich triarylamine groups usually act as an electron donor, and their derivatives are usually used to construct organic photoelectric materials. Electron-deficient Ru(II) bis(terpyridine) derivatives are generally applied in metal-organic catalytic reaction. Despite Yao *et al.* reported a linear supramolecular polymer comprised of terpyridine monomodified pillar[5]arene with a guest through host-guest complexation and metal ( $Zn^{2+}$ ) coordination, achieving stimuli-responsive glue-sol transitions [15]. In the present work, a pure metal complex, *i.e.*, Ru(II) bis(terpyridine)-modified pillar[5]arenes (TPY-P5( $Ru^{2+}$ )) as host assembles with triazoletriphenylamine amyl cyanide (TTAC) as guest through host-guest

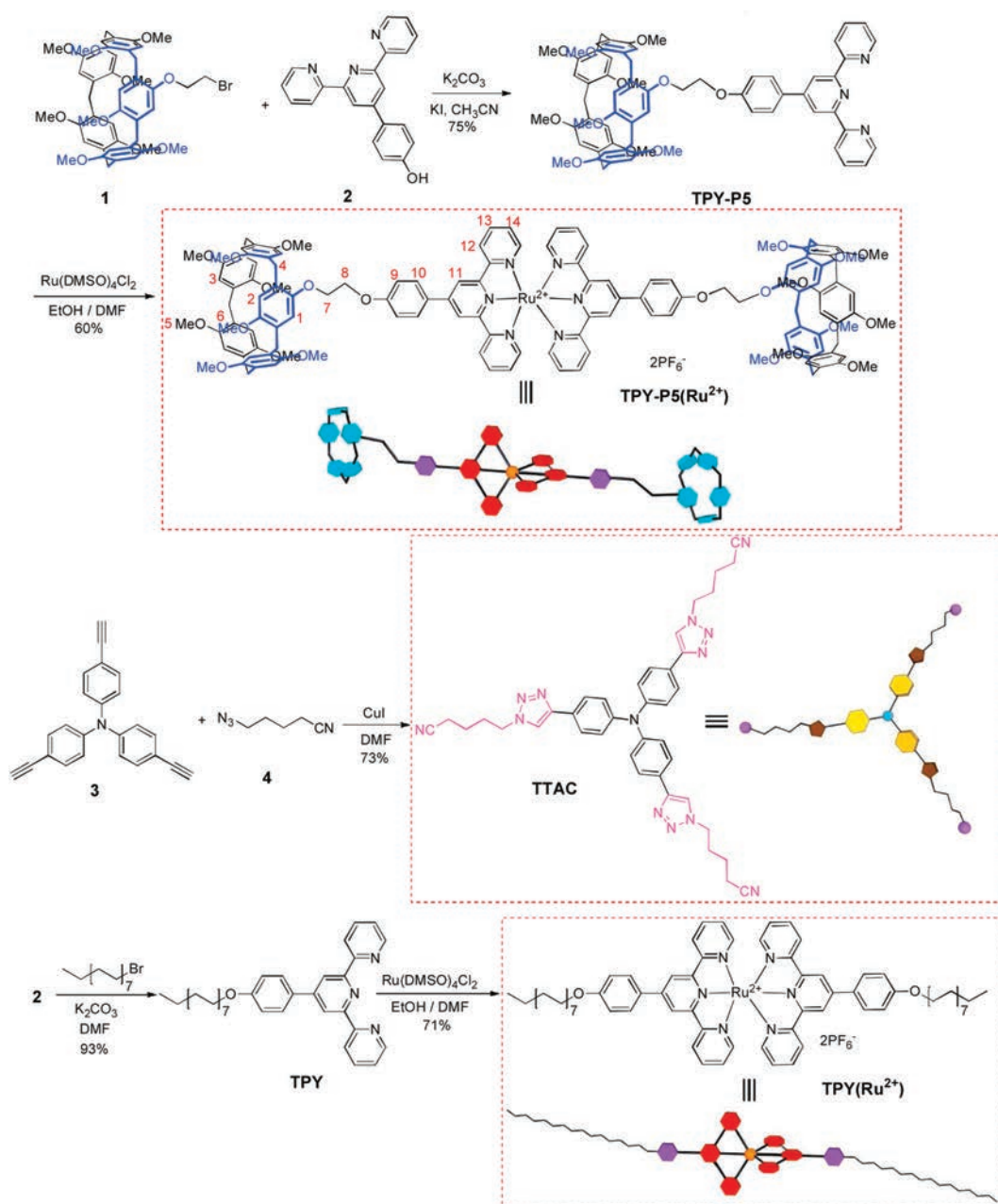
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interaction to fabricate a PET-based supramolecular system, achieving tunable fluorescence and PET, which is a new research and has not been exploited, to our knowledge.

Compounds **TPY-P5(Ru<sup>2+</sup>)** and **TTAC** were successfully synthesized, and their synthetic routes were shown in Scheme 1. The reaction of substance **1** [16] with **2** [17] obtained terpyridine-modified pillar[5]arene **TPY-P5** in 75% yield, and further complexed with Ru(DMSO)<sub>4</sub>Cl<sub>2</sub> to get **TPY-P5(Ru<sup>2+</sup>)** in 60% yield. Guest **TTAC** was prepared from alkyne **3** [18] and azide **4** [19] through “Click” reaction in 73% yield. Compound **2** reacted with *n*-tetradecyl bromide to obtain intermediate **TPY** in 93% yield, and further complexed with Ru(DMSO)<sub>4</sub>Cl<sub>2</sub> to prepare reference substance **TPY(Ru<sup>2+</sup>)** in 71% yield. Here, **TPY** was modified with the long alkyl chain (as a solubilizing agent) to increase the solubility of **TPY(Ru<sup>2+</sup>)**. The chemical structures of all the new compounds above were confirmed by <sup>1</sup>H NMR, <sup>13</sup>C NMR, and HR-MS, which were shown in Figs. S1–S15 (Supporting information).

The successful preparation of **TPY-P5(Ru<sup>2+</sup>)** and **TTAC** inspired us to investigate their assembling behavior. It is well known that large binding ability ( $K_a = 1.2 \times 10^4$  L/mol) exists between **P5** and the neutral guests bearing two or three short alkyl chains with a triazole site and a cyano site at either end in chloroform [20]. We assumed that **TPY-P5(Ru<sup>2+</sup>)** and **TTAC** might self-assemble to form a 3:2 (host/guest) supramolecular architectures. However, steric hindrance would become seemingly a key influencing factor for whether 3:2 host-guest binding pattern formed. Therefore, it is very necessary for us to examine whether the guest **TTAC** can complex with three **P5s**. From NMR titration spectroscopy (Fig. S16 in Supporting information), an apparent upfield shift of the resonance for methylene in the guest ( $H_{a-d}$ ) was observed, and their integral areas achieved unchanged with continued addition of 3 equiv. **P5**, indicating that one **TTAC** can assemble with three **P5s**. The complex stability constant ( $K_S$ ) was determined to be  $6.52 \times 10^8$  M<sup>-3</sup> using the <sup>1</sup>H NMR single point method [21].

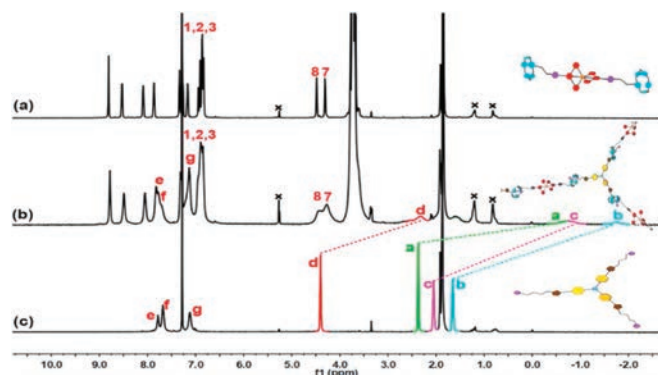


Scheme 1. The synthetic routes of host **TPY-P5(Ru<sup>2+</sup>)**, guest **TTAC** and reference compound **TPY(Ru<sup>2+</sup>)**.

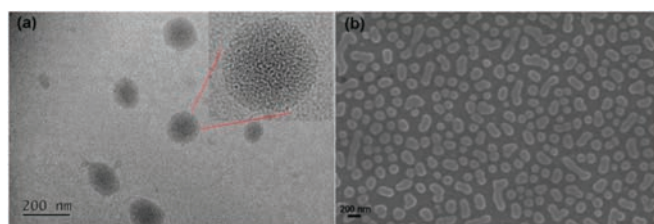
Furthermore, the binding stoichiometry of **TPY-P5(Ru<sup>2+</sup>)** with **TTAC** was verified by a Job plot, where a maximum peak at a molar ratio of 0.6 (Fig. S17 in Supporting information) was observed, revealing a 3:2 host–guest binding stoichiometry. Besides, a direct evidence came from the comparison of <sup>1</sup>H NMR spectroscopy. As illuminated in Fig. 1, the similar upfield shift for the resonance of methylene (H<sub>a-d</sub>) of the guest was presented, and all the signal peaks was changed to be passivated, implying that the occurrence of the assembling behavior and the formation of a new supramolecular polymer **TTAC<sub>2</sub>TPY-P5(Ru<sup>2+</sup>)**. The assembling pattern was further confirmed by NOSEY and ROSEY spectra (Figs. S18 and S19 in Supporting information) that the methylenes between cyano group and triazole in **TTAC** were located in the cavity of the **P5** in **TPY-P5(Ru<sup>2+</sup>)**.

Moreover, the intuitional proof on topology morphology formed automatically by the **TTAC<sub>2</sub>TPY-P5(Ru<sup>2+</sup>)** came from microscopic investigation. As shown in Fig. 2a, many nanoparticles were observed in the TEM image, which further verified by SEM and AFM images (Fig. 2b and Fig. S20 in Supporting information). Moreover, the hydrodynamic diameter of the assembly in CHCl<sub>3</sub> was detected as about 36.77 nm and 525.8 nm, indicating the formation of two types of nanoaggregates, i.e., oligomers and polymers in dilute solution. Meanwhile, the aforementioned observations also implied that oligomers formed by the assembly transformed gradually to a large size of polymers in solvent evaporation process (Fig. S21 in Supporting information). Above all, three jaw **TTAC** and bridged-bis-P5 **TPY-P5(Ru<sup>2+</sup>)** can self-organized to form nanoparticles in weak polar solvent. The possible mechanism for nanoparticle formation should be that the supramolecular assembly **TTAC<sub>2</sub>TPY-P5(Ru<sup>2+</sup>)** first formed twisted and random reticular nanostructures and then further intertwined to near spherical nanoparticles, as presented in Scheme 2.

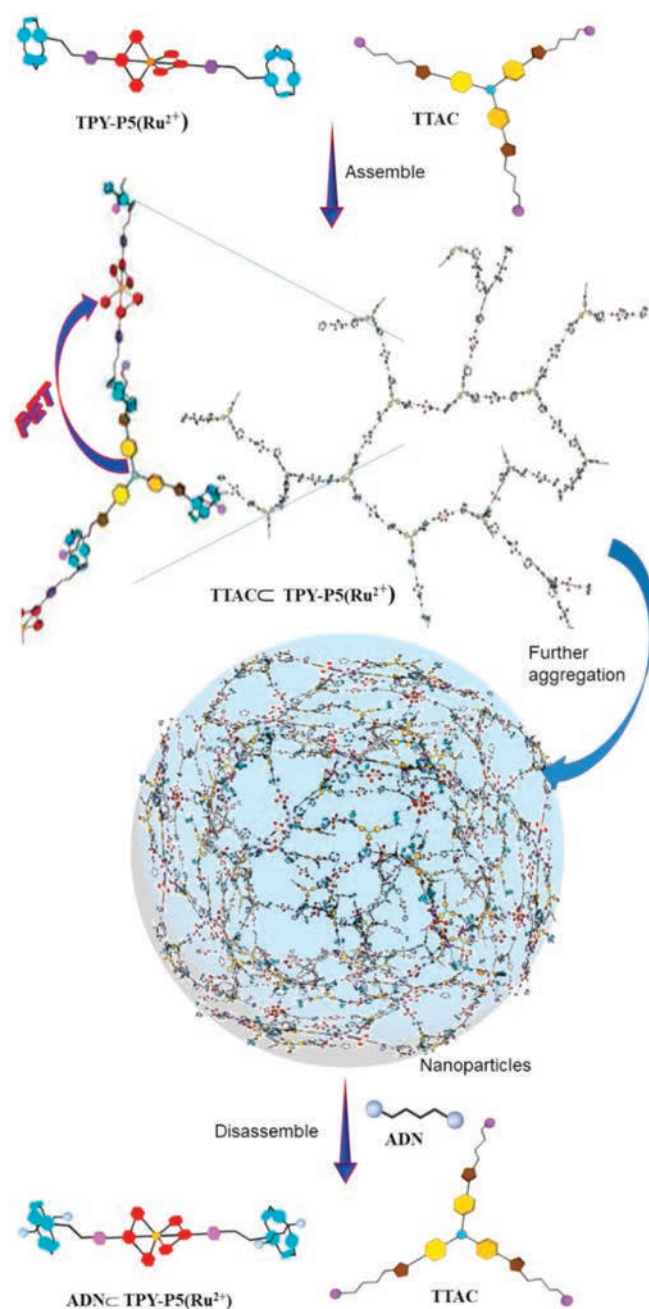
Viewing the structural feature of the host and guest, triphenylamine derivatives donated with lone pair electron usually act as electron donor and bis-terpyridyl ruthenium complexes with



**Fig. 1.** <sup>1</sup>H NMR (600 MHz, v(CD<sub>3</sub>CN): v(CDCl<sub>3</sub>) = 1:9) spectra of (a) host **TPY-P5(Ru<sup>2+</sup>)**, (b) assembly **TTAC<sub>2</sub>TPY-P5(Ru<sup>2+</sup>)** and (c) guest **TTAC**; [**TPY-P5(Ru<sup>2+</sup>)**] = 3 × 10<sup>-3</sup> mol/L, [**TTAC**] = 2 × 10<sup>-3</sup> mol/L.



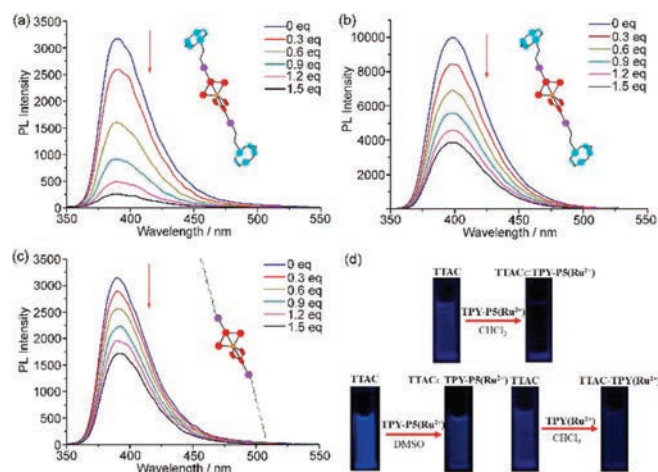
**Fig. 2.** (a) TEM image of assembly **TTAC<sub>2</sub>TPY-P5(Ru<sup>2+</sup>)**; (b) SEM image of assembly **TTAC<sub>2</sub>TPY-P5(Ru<sup>2+</sup>)**.



**Scheme 2.** The chemical structures of host **TPY-P5(Ru<sup>2+</sup>)**, guest **TTAC**, reference substance **TPY(Ru<sup>2+</sup>)** and **TTAC<sub>2</sub>TPY-P5(Ru<sup>2+</sup>)** and its schematic illustration of assembling and disassembling pattern.

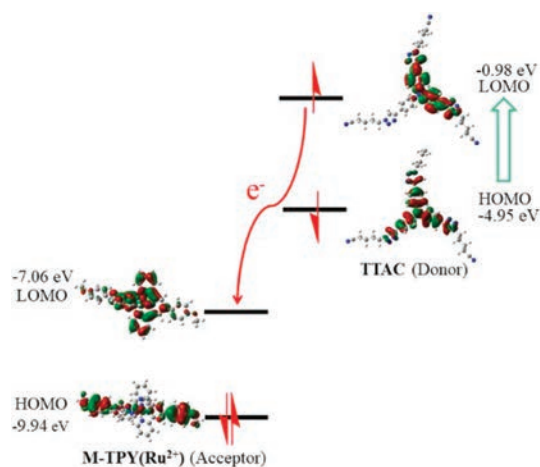
electron deficiency can play a role of electron acceptor. We supposed that the resultant supramolecular assembly might achieve PET between the host and guest originated from short enough intermolecular D–A distance. Subsequently, the photo-physical properties of the guest, host and assembly were performed. The UV–vis absorption spectra of them were carried out and displayed in Fig. S22 (Supporting information). As we predicted, when 1.5 equiv. **TPY-P5(Ru<sup>2+</sup>)** was gradually added to the chloroform solution containing **TTAC**, the fluorescence of the guest at 390 nm was dramatically quenched by approximately 94%, manifesting the occurrence of “efficient PET” from triphenylamine to bis-terpyridyl ruthenium (2+) complex (Fig. 3a) [22].

To demonstrate the important role of the PET process played by **P5**, some control experiments were performed. As illuminated in



**Fig. 3.** (a) The variation of fluorescent spectra of **TTAC** with continuous addition of **TPY-P5(Ru<sup>2+</sup>)** in chloroform; excitation at 338 nm (slit = 2.5, 2.5). (b) The variation of fluorescent spectra of **TTAC** with continuous addition of **TPY-P5(Ru<sup>2+</sup>)** in DMSO; excitation at 338 nm (slit = 2.5, 1). (c) The variation of fluorescent spectra of **TTAC** with continuous addition of **TPY(Ru<sup>2+</sup>)** in chloroform; excitation at 338 nm (slit = 2.5, 2.5). (d) The photographs of fluorescent quenching.  $[\text{TTAC}] = 1 \times 10^{-5}$  mol/L.

Fig. 3b, when chloroform solvent was changed to strong polar solvent such as DMSO, ~56% fluorescent intensity of **TTAC** at 398 nm was quenched in the presence of 1.5 equiv. **TPY-P5(Ru<sup>2+</sup>)**, which was much lower than that in chloroform. This result was attributed to weak binding ability of the guest and host in DMSO than in chloroform. As displayed in Fig. S23 (Supporting information), there were many unassembled host and guest in the system. Moreover, more quantitative evidence was provided by NMR titration experiment. From Fig. S24 (Supporting information), the  $K_S$  of the guest and **P5** in DMSO was determined to be  $1.53 \times 10^7 \text{ M}^{-3}$ , which was greatly lower than that in chloroform ( $6.52 \times 10^8 \text{ M}^{-3}$ ). Furthermore, we selected **TPY(Ru<sup>2+</sup>)** without **P5** as a reference compound to further verify our viewpoint, where the long alkyl chain was introduced to enhance the solubility of the metal-organic complex. Subsequently, fluorescent titration experiment was carried out. As shown in Fig. 3c, fluorescence intensity of **TTAC** at 390 nm was quenched by only 40% with continued addition of 1.5 equiv. **TPY(Ru<sup>2+</sup>)**, manifesting that “attenuated PET” occurred. Hence, in this PET system, **P5** played a key role in the “efficient PET” process *via* shortening the intermolecular electron D-A distance through supramolecular complexation. Interestingly, the guest and assembly showed great differences on the maximum fluorescence emission wavelength and intensity in  $\text{CHCl}_3$  and DMSO. As displayed in Figs. S25 and S26 (Supporting information), the maximum fluorescence manifested bathochromic-shift by 8 nm, when the solvent was changed from  $\text{CHCl}_3$  to DMSO. Simultaneously, the fluorescence intensity of the guest was enhanced by a factor of 23 in this process. More surprisingly, the fluorescence intensity of the assembly was strongly increased by a factor of 913. These variations were originated from that the guest and assembly formed different stack models in diverse solvent. Subsequently, the fluorescence spectra of different volume ratio of  $\text{CHCl}_3$ /DMSO solution containing the guest and assembly were performed. As displayed in Figs. S27 and S28 (Supporting information), the maximum emission of the guest and assembly both manifested obvious red-shift along the DMSO volume content from 0% to 30%, and the fluorescence intensity exhibited enhanced trend with the increased of the DMSO volume content from 40% to 90%. These experiments both revealed that the guest and assembly both possessed aggregation-induced emission enhancement (AIEE) property. Of an interest is that the supramolecular nano-assembly became a fluorescent switch triggered by solvent

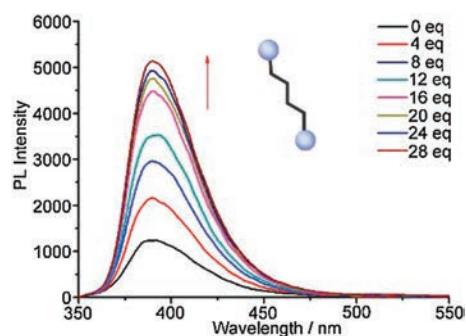


**Fig. 4.** The proposed photoinduced electron transfer mechanism of **M-TPY(Ru<sup>2+</sup>)** and **TTAC**.

conversion. More quantitatively, we selected the guest and assembly in DMSO to investigate the variation of their fluorescence quantum yield ( $\Phi_F$ ) to avoid test error because of very weak fluorescence intensity for them in  $\text{CHCl}_3$ . As manifested in Fig. S29 (Supporting information), the  $\Phi_F$  of **TTAC** was apparently declined from 31.98% to 6.92% with addition of 1.5 equiv. **TPY-P5(Ru<sup>2+</sup>)**, which further implied the occurrence of PET process.

To further demonstrate that the above-mentioned fluorescence quenching was indeed attributed to PET, the relevant optimized structures and HOMO-LOMO orbital energy was performed. Here, we selected 4-methoxytridipyridine (**M-TPY(Ru<sup>2+</sup>)**) instead of **TPY-P5(Ru<sup>2+</sup>)** with **TPY(Ru<sup>2+</sup>)** to calculate their HOMO and LOMO orbital energy. As shown in Table S1 (Supporting information) and Fig. 4, the LOMO energy level (-0.98 eV) of **TTAC** (electron donor) is greatly higher than **M-TPY(Ru<sup>2+</sup>)** as electron acceptor (-7.06 eV), which completely meets the conditions for the occurrence of PET. Therefore, a new PET system was witnessed.

Interestingly, the fluorescent intensity and intermolecular PET process of the resultant assembly were modulated by another competitive guest due to unsubstantial combination of the host and guest [23]. When excess adiponitrile (ADN) was gradually added in the solution containing the supramolecular assembly, the quenching fluorescent intensity of **TTAC**/**TPY-P5(Ru<sup>2+</sup>)** at 390 nm got partial recovery, implying that the supramolecular assembly was destroyed to some extent (Fig. 5). To prove the speculation, the NMR contrast experiment was subsequently performed. As illuminated in Fig. S30 (Supporting information), the <sup>1</sup>H NMR spectroscopy of free host **TPY-P5(Ru<sup>2+</sup>)** and guest **TTAC** was presented in the presence of excess ADN, manifesting that **TTAC**



**Fig. 5.** The variation of fluorescent spectrum of **TTAC**/**TPY-P5(Ru<sup>2+</sup>)** with continuous addition of ADN;  $[\text{TTAC}] = 1 \times 10^{-5}$  mol/L,  $[\text{TPY-P5(Ru<sup>2+</sup>)}] = 1.5 \times 10^{-5}$  mol/L; Excitation at 338 nm (slit = 5, 2.5).

was ejected from the cavity of **P5** in **TPY-P5(Ru<sup>2+</sup>)** by ADN. This showed that the resultant supramolecular assembly **TTAC**⊂**TPY-P5(Ru<sup>2+</sup>)** was completely dissociated and meanwhile, a new ternary supramolecular assembly **ADN**⊂**TPY-P5(Ru<sup>2+</sup>)** was produced, as illuminated in Scheme 2.

In conclusion, a multi-component metal-organic supramolecular nanoarchitectures **TTAC**⊂**TPY-P5(Ru<sup>2+</sup>)** comprised of **TPY-P5(Ru<sup>2+</sup>)** and **TTAC** had been constructed through host-guest complexation. The assembling pattern and formed topological morphology were demonstrated by NMR, NOSEY, ROSEY, SEM, TEM, AFM and DLS. Interestingly, the supramolecular assembly achieved “efficient PET” from **TTAC** to **TPY-P5(Ru<sup>2+</sup>)**, where the host-guest complexation played an important role in shortening the D-A distance. Subsequently, the occurrence of PET process was further proved by HOMO-LOMO orbital energy calculation. Crucially, the fluorescence and PET process of the assembly could be modulated by another competing guest to some extent. The present study provided a versatile strategy to develop smart metal-organic supramolecular materials based with PET for controllable photoelectric transforming.

### 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

We thank the National Natural Science Foundation of China (Nos. 21801063, 21672192, 21803059 and 21929101), China Postdoctoral Science Foundation (No. 2018M642767), Ph.D. Foundation of Henan University of Technology, China (No. 2017BS020), the Science and Technology Foundation of Henan Province (No. 192102210039), the Colleges and Universities Key Research Program Foundation of Henan Province (No. 19A150022), Foundation of Henan University of Technology (No. 2018QNJH14), and the Natural Science Foundation of Henan Province (No. 182300410255) for financial support.

### Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:<https://doi.org/10.1016/j.ccllet.2020.02.024>.

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