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Luminescent cyclic trinuclear coinage metal  
complexes with aggregation-induced emission  
(AIE) performance†Yuan Tian,<sup>a</sup> Zhao-Yang Wang,<sup>\*a</sup> Shuang-Quan Zang,<sup>id</sup> <sup>\*a</sup> Dan Li<sup>id</sup> <sup>\*b</sup> and  
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A series of AIE active cyclic trinuclear complexes (Au<sub>3</sub>, Ag<sub>3</sub>, and Cu<sub>3</sub>) have been successfully prepared and elucidated by X-ray crystallography. These complexes showed excellent AIE properties and their quantum yields (QYs) were significantly increased compared to those of the free ligands. The remarkable solution induced AIE activity in atom-precise coinage metal complexes is still scarce, and this work opens a promising avenue for the development of easily prepared metal-based AIE luminogens with high emission efficiency.

Luminogens with aggregation-induced emission (AIE) properties have emerged as a new class of luminescent materials which have shown great possibility to be widely utilized as optoelectronic devices, sensory systems, *etc.*<sup>1</sup> The AIE effect is considered constructive because the photoemission of the propeller-like molecules could be significantly enhanced after aggregation, which greatly differs from the aggregation-caused quenching (ACQ) observed in many conventional chromophores that possess planar and well-conjugated structures.<sup>2</sup> In general, conventional luminogens only exhibit efficient emission in a molecularly dispersive state and the aggregation of the molecules should be strictly avoided, which could lead to an obstruction of applications.<sup>3</sup> The discovery of the fantastic AIE phenomenon furnishes a high possibility of conquering the ACQ problem.<sup>4</sup> Therefore, various AIE luminescent materials have been designed and synthesized, among which hexaphenylsilole (HPS), tetraphenylethene (TPE) and distyreneanthracene (DSA) moieties are typical representatives.<sup>4a,5</sup>

Nevertheless, a majority of the AIE active molecules are organic and this phenomenon have been rarely explored for metal complexes.<sup>6</sup> Not to mention AIE luminescent materials with precise structures have been synthesized.<sup>7</sup> In addition, compared with the conventional chromophores, luminescent metal complexes have better photostability, which may result in much better performance in catalytic processes and optoelectronics (*e.g.*, LEDs).<sup>8</sup> Coinage metal complexes have attracted extensive interest in recent years owing to their novel properties and potential applications in various fields.<sup>9,10</sup> Among these physical/chemical properties, luminescence represents one of the most intriguing features of these materials.<sup>11</sup> However, a technical challenge is that the photoluminescence of coinage metal complexes are often weak, and their quantum yields (QYs) rarely exceed 0.1%. Recently, the discovery of the AIE phenomenon has led to its use in designing high-luminescence metal complexes to address these obstacles.<sup>12</sup> Xie and co-workers have synthesized Au<sub>22</sub>(SG)<sub>18</sub> clusters, which showed red emission with a QY of ~8%. By comparison with those of Au(I)-thiolate complexes, their stronger luminescence intensity was ascribed to AIE. However, the origin of emission in these clusters still remains unaddressed, which is a major hurdle for the design of excellent AIE materials.<sup>13</sup> Therefore, it remains a challenge to develop a method for the rational design of coinage metal complexes with excellent AIE properties.<sup>4b,14</sup>

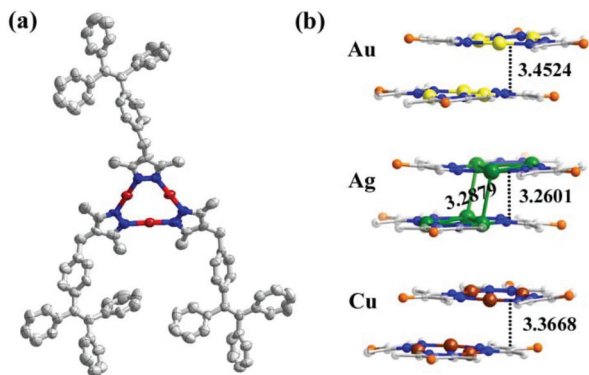
Herein, we successfully synthesized a series of prominent AIE active coinage metal complexes, namely [Au<sub>3</sub>(DTBP)<sub>3</sub>·CH<sub>2</sub>Cl<sub>2</sub>·CHCl<sub>3</sub>], [Ag<sub>3</sub>(DTBP)<sub>3</sub>·2CHCl<sub>3</sub>] and [Cu<sub>3</sub>(DTBP)<sub>3</sub>·2CHCl<sub>3</sub>] (denoted as **M**<sub>3</sub>, M = Au, Ag or Cu), by reacting 3,5-dimethyl-4-(4-(1,2,2-triphenylvinyl)benzyl)-1*H*-pyrazole (DTBP) with coinage metal ions in the presence of triethylamine (Fig. 1a). All of these complexes were successfully elucidated by X-ray crystallography. The triangular assemblies of coinage metal atoms and pyrazole groups promoted the formation of an ordered layered structure which increased the restriction of the intramolecular rotation (RIR) effect of the AIE-active units, thus effectively restricting non-radiative decay. Therefore the

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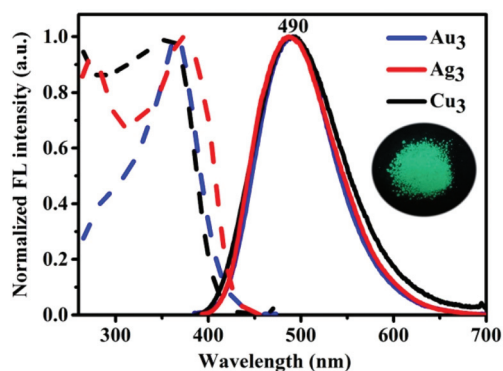
**Fig. 1** (a) Schematic representing the crystal structure of  $M_3$ . Thermal ellipsoids are set at 50% thermal probability. (b) Interlayer stacking structures of  $Au_3$ ,  $Ag_3$  and  $Cu_3$ . Color codes: yellow, Au; green, Ag; brown, Cu; blue, N; gray, C (red, M; orange, R =  $-CH_2TPE$ ). All hydrogen atoms are omitted for clarity.

process promoted a highly emissive and strong AIE response, which led to the QYs reaching up to 12.01% ( $Ag_3$ , about 7.5 times that of the ligand) in the solid state. Our approach using a rigid pyrazolyl ligand paves a new way to construct precise structure coinage metal complexes with intense luminescence and prominent AIE performance.

## Results and discussion

A number of TPE-based discrete organic molecular structures have been determined by experimental characterization.<sup>15</sup> However, coinage metal complexes with precise structures are still rare.  $Au_3$ ,  $Ag_3$  and  $Cu_3$  were facilely synthesized *via* a one-pot method by reacting stoichiometric coinage metal salts or precursors with pyrazole ligands in the presence of trimethylamine.<sup>16</sup> Needle-shaped single crystals suitable for X-ray structural analysis were obtained by recrystallizing the samples *via* vapor diffusion of *n*-hexane into their chloroform solutions (ESI† for details). DTBP was first synthesized and its purity was confirmed by  $^1H$  NMR and  $^{13}C$  NMR (Fig. S1–4†). The phase purity and chemical formulas of  $Au_3$ ,  $Ag_3$  and  $Cu_3$  were further verified using *in situ* power X-ray diffraction (PXRD) patterns, thermogravimetric analysis (TGA) and elemental analysis (Fig. S5–9†).

Single crystal X-ray structure analyses revealed that  $Au_3$  and  $Ag_3$  crystallize in triclinic space group  $P\bar{1}$ , while  $Cu_3$  crystallizes in monoclinic space group  $P2_1/c$  (Table S1†). Their asymmetric units exhibit cyclic trinuclear isostructures (Fig. 1a). Three pyrazole groups uniformly adopt the  $\mu_2-\eta_1, \eta_1$  ligation mode to link two metal ions to build a stable triangular plane. Notably, the  $Ag(i)\cdots Ag(i)$  distances between adjacent molecules are about 3.2879 Å in  $Ag_3$ , indicating the existence of argentophilic interactions. However, due to the steric hindrance of TPE molecules, intermolecular metallophilic interactions do not occur in the stacked structures of  $Au_3$  and  $Cu_3$  (Fig. 1b).<sup>17</sup> Interestingly, in the dispersed state, the stable  $M_3$  structures



**Fig. 2** Fluorescence spectra of  $Au_3$  (blue curve),  $Ag_3$  (red curve), and  $Cu_3$  (black curve) excited (dotted line) at 366 nm in the solid state at room temperature. Inset shows the fluorescence image of  $Ag_3$  under 365 nm UV light.

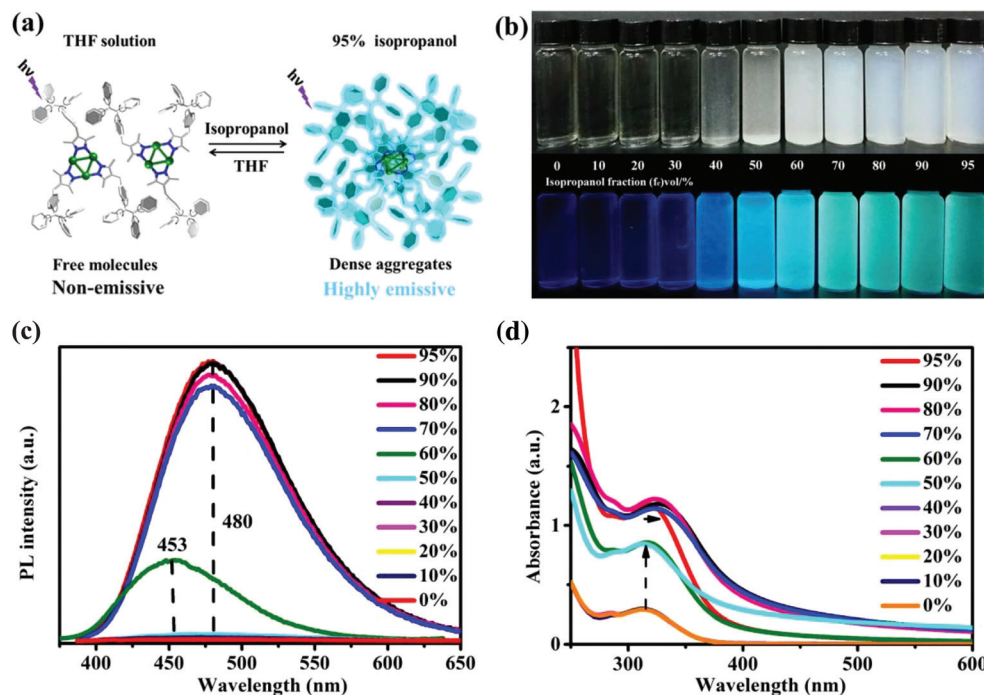
do not restrict the incorporated TPE molecules (free to rotate) due to the presence of methylene.

The luminescence properties of  $M_3$  were further investigated.  $M_3$  crystals emitted intense blue-green luminescence ( $\lambda_{max} = 490$  nm) under UV light (Fig. 2), while the DTBP ligand showed blue-green emission ( $\lambda_{max} = 494$  nm) (Fig. S10†). The similar profiles indicated that the emissions of complexes are ligand-centered. Furthermore, the nanosecond-scale emissive lifetime of  $M_3$  emission bands (*e.g.*,  $\tau_{Ag_3} = 6.42$  ns) (Fig. S11–13†) and DTBP emission bands ( $\tau_{DTBP} = 2.60$  ns) (Fig. S14†) indicated the characteristics of fluorescence, originating from ligand emission.<sup>7,18</sup>

As a typical AIE luminogen, TPE has a unique structure with phenyl rotors, which could display AIE effect under RIR process.<sup>1</sup> Irradiation of a dilute tetrahydrofuran solution (a good solvent of DTBP ( $<10^{-3}$  M)) at 365 nm showed almost no fluorescence at 474 nm. However, increasing the amount of water (a poor solvent for DTBP) in the mixed solution greatly enhanced the blue-green emission intensity ( $3.2 \times 10^4$  times), and the QY was measured using an integrating sphere to be 1.59% in the solid state, indicating its strong AIE behavior (Fig. S15–17†).

Interestingly,  $M_3$  complexes suggested a similar emission pattern with higher fluorescence intensity and QYs compared to those of the free ligand. The QYs of  $Au_3$ ,  $Ag_3$  and  $Cu_3$  were found to be 6.92%, 12.01% and 9.82%, respectively, at room temperature in the solid state ( $Ag_3$ , about 7.5 times that of the ligand), which should be ascribed to the structural rigidity resulting from the stack between planes, which could effectively restrict non-radiative decay. Furthermore, the ordered stack of the interlayer can form a more stable internal complex architecture. In particular, argentophilic interactions within  $Ag_3$  shortened the overall intermolecular  $Ag(i)\cdots Ag(i)$  distances, resulting in a QY higher than those of  $Au_3$  and  $Cu_3$  (Fig. 1b).<sup>19</sup>

The intense blue-green emission of  $Ag_3$  as an example encouraged us to study its AIE properties. The dependence between the aggregates and the luminescence intensity of  $Ag_3$  was detected for the aggregates from solvent-induced aggrega-



**Fig. 3** (a) Schematic illustration of solvent-induced AIE properties of the  $\text{Ag}_3$  complex. (b) Digital photograph of the  $\text{Ag}_3$  complex (concentration:  $2 \times 10^{-4}$  M) in mixed solvents with different  $f_e$  under visible (top row) and UV (bottom row) light. (c) Photoemission spectra and (d) UV-vis absorption spectra of  $\text{Ag}_3$  in mixed solvents with different  $f_e$ .

tion. As illustrated in Fig. 3a, the aggregation degree was controlled by the polarity of the mixed solvent, which could be varied by the volume fraction of isopropanol in the solvent  $f_e$  ( $f_e = \text{vol}_{\text{isopropanol}}/\text{vol}_{\text{tetrahydrofuran+isopropanol}}$ ; isopropanol is a poor solvent). The  $\text{Ag}_3$  solution turned cloudy with weak blue emission when the  $f_e$  reached 40% due to the initial aggregation. When the  $f_e$  increased to 70%, the solution showed stronger blue-green luminescence emission, suggesting the presence of well-dispersed colloids of denser aggregates (Fig. 3b). The solvent-induced AIE of  $\text{Ag}_3$  was reversible since decreasing the  $f_e$  could re-dissolve the aggregates, thereby annulling the AIE mechanism and subsequently quenching the luminescence.

Photoemission spectra were recorded to interpret the emission changes due to variations in the aggregation degree (Fig. 3c). Before the  $f_e$  reached 60%, the luminescence intensity increased monotonically with the increase in the aggregation degree. When the  $f_e$  reached 70%, the main peak in the emission spectrum shifted from 454 (blue emitting) to 473 nm (blue-green emitting), which was similar to the photoemission spectra of DTBP shown in Fig. S16,† and it could be attributed to the solvent effect.<sup>20</sup> Moreover, the QYs of  $\text{Au}_3$ ,  $\text{Ag}_3$  and  $\text{Cu}_3$  were found to be 5.48%, 7.94% and 3.98%, respectively, with  $f_e = 80\%$  and concentration =  $1.57 \times 10^{-4}$  M, which are lower than the corresponding QYs in the solid state. These results further support the AIE behaviours of these complexes. To keep the concentration of DTBP the same, the QY of the DTBP ligand was found to be 0.84% with a concentration of  $4.71 \times 10^{-4}$  M.

UV-vis spectroscopy was also used to follow the aggregation degree of  $\text{Ag}_3$  with an increasing  $f_e$ , while it showed a main peak at 314 nm (Fig. 3d). When the  $f_e$  increased further from 50% to 95%, the formation of smaller and denser aggregates was indicated by bathochromic shifts of the spectra (Fig. 3d, solid black arrow). In the process, the high polarity of the solvent gave impetus to a red shift. In addition, a hyperchromic shift of the UV-vis absorption spectra appeared due to the large increase in background scattering (dotted black arrow).<sup>6a</sup>

$\text{Au}_3$  and  $\text{Cu}_3$  also showed similar AIE properties. Unlike  $\text{Au}_3$  and  $\text{Ag}_3$ , dichloromethane was used as a good solvent for  $\text{Cu}_3$ , as  $\text{Cu}_3$  could be oxidized more easily in THF. Fig. S18–20† show that the  $\text{Au}_3$  solution was non-luminescent until the  $f_e$  reached 70%; afterwards the solution exhibited intense blue-green emission. The spectra showed that their fluorescence intensity gradually increased ( $\lambda_{\text{max}} = 469$  nm). Correspondingly, the AIE images of  $\text{Cu}_3$  showed the same pattern as that of  $\text{Ag}_3$ , with the  $\lambda_{\text{max}} = 470$  nm until the  $f_e$  reached 70% or higher (Fig. S21–23†). With such excellent AIE performance in a solution-induced system, these materials could find their applications in biosensing, bioimaging, and dual-functional uses.

## Conclusions

In summary, we have developed a series of TPE-incorporated coinage metal complexes  $\text{Au}_3$ ,  $\text{Ag}_3$  and  $\text{Cu}_3$ , which exhibit excellent AIE performance. The crystal structures of these

cyclic trinuclear complexes were successfully elucidated by X-ray crystallography. These complexes showed intense ligand-centered blue-green emission at 490 nm. The coordination induced intermolecular stacking interactions in these complexes highly promoted structural rigidity and their QYs were significantly enhanced (12.01% for Ag<sub>3</sub>, about 7.5 times that of the ligand in the solid state). Meanwhile, the aggregation degree positively correlated with the fluorescence intensity, indicating that the solution-induced AIE activity was significantly affected by the RIR and the emissive efficiency improved in the aggregation process. As a new strategy to expand the field of AIE molecules, the coinage metal complexes produced precise structure nanomaterials with strong fluorescence which may be applied to diverse fields. Moreover, the self-assembly provided a promising method for enhancing the luminescence of AIE molecules in nanoscale complexes.

## Conflicts of interest

There are no conflicts to declare.

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