Circularrayly polarized luminescence of coordination aggregates

Zhong-Liang Gong1  |  Zhong-Qiu Li1,2  |  Yu-Wu Zhong1,2

1 Beijing National Laboratory for Molecular Sciences, CAS Key Laboratory of Photocatalysis, CAS Research/Education Center for Excellence in Molecular Sciences, Institute of Chemistry, Chinese Academy of Sciences, Beijing, China
2 School of Chemical Sciences, University of Chinese Academy of Sciences, Beijing, China

Correspondence
Yu-Wu Zhong, Beijing National Laboratory for Molecular Sciences, CAS Key Laboratory of Photochemistry, CAS Research/Education Center for Excellence in Molecular Sciences, Institute of Chemistry, Chinese Academy of Sciences, Beijing 100190, China. Email: zhongyuwu@iccas.ac.cn

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Abstract
The development and applications of materials with efficient circularly polarized luminescence (CPL) have become an interdisciplinary frontier research topic. We summarize herein the recent advance in the development and applications of CPL-active aggregates based on metal-ligand coordination materials (termed as “coordination aggregates”). The materials surveyed are classified as aggregates of small-molecular metal complexes, which include monocomponent assemblies of Pt(II) complexes and other complexes and binary aggregates of metal complexes, and CPL-active metal-ligand coordination helicates, polymers, and frameworks. The efforts in improving the dissymmetry luminescence factors and quantum yields of these materials and the use of the aggregation strategy in enhancing the performance of isolated molecules are discussed. The recent applications of chiral metal complexes in circularly polarized organic light-emitting diodes (OLEDs) based on solution- or evaporation-processed procedures are surveyed. In addition, the uses of lanthanide complexes in CPL-contrast imaging and as CPL probes are highlighted. The common discussion on the mechanism of aggregation-enhanced CPLs and a perspective on future works of CPL-active coordination aggregates are finally given.

Keywords
chiral metal complexes, circularly polarized luminescence, coordination helicates and polymers, liquid crystals, metal-organic frameworks, supramolecular assemblies

1 | INTRODUCTION

Circularrayly polarized luminescence (CPL) is generated by chiral materials that can emit differential left- and right-handed circularly polarized (CP) light, and it reflects the structural information of chiral emitters in excited states.[1–4] Since the first CPL measurement was recorded for the solution of trans-β-hydroxindanone by Emeis and Oosterhoff,[5] the exploitation of CPL-active molecular materials has attracted tremendous attention, in view of their applications in three-dimensional (3D) display, chiral optoelectronic devices, chiral recognition and catalysis, contrast imaging, security-enhanced information storage and transportation, etc.[1–4] Especially in the past decade, a large amount of CPL-active molecules and materials have been developed and investigated, including chiral organic small molecules and conjugated polymers, chiral lanthanide (Ln) and transition-metal complexes, supramolecular assemblies and liquid crystalline (LC) materials, as well as chiral metal clusters and inorganic nanomaterials.[6–27] CPL performance is generally evaluated by the luminescence dissymmetry factor (Δ(lum)) and quantum yield (Φ). The former refers to the degree of “enantiorichness” of the emitted CP light, and its value is in the range of −2 to +2.[3,28–31] Due to the trade-off between these two parameters, materials with both large |Δ(lum)| and Φ values are still limited so far.[1–36]

In the early stage of this area, much effort was devoted to chiral Ln(III) complexes useful as CPL probes and CPL-contrast imaging reagents.[37] Among them, the cesium tetrakis(3-heptafluorobutyryl)-(+)camphorato) Eu(III) complex exhibited a particularly high |Δ(lum)| value of 1.38, making it and related chiral Ln(III) complexes useful as CPL probes and CPL-contrast imaging reagents.[37] However, these Ln(III) complexes generally display low Φ and labile coordination structures, which are later improved by the use of light-harvesting organic ligands with high coordination numbers.[14–16]

CPL-active molecular materials, including chiral organic small molecules and metal complexes, are characterized by their relatively high Φ, precise molecular composition, and tunable structures and emission wavelengths. Although a great amount of CPL-active molecules have been developed, the low Δ(lum) values are generally small (10−5 to 10−2),
partially because of the size mismatch between the excitation wavelength (hundreds of nanometers) and that of common isolated molecular materials (typically a few of nanometers or less).\textsuperscript{[3,4,11]} In this context, aggregation has become an efficient strategy for the chirality amplification and $g_{\text{lum}}$ enhancement of molecular materials, including supramolecular assembly and polymerization,\textsuperscript{[2,18,19]} the construction of energy/electron transfer system,\textsuperscript{[38–41]} and the formation of LC phase.\textsuperscript{[23,24]} Remarkably, $g_{\text{lum}}$ values as high as 1.79 have been recorded for CPL-active polymers combined with chiral nematic LC (N*-LC) materials, based on the excellent capability of N*-LC to selectively reflect/transmit handedness-specific CP light.\textsuperscript{[42]} It is of note that aggregated or solid samples often suffer from the issue of aggregation-caused emission quenching, which however can be addressed by the combination of chromophores with aggregation-induced emission (AIEgens)\textsuperscript{[43–47]} and chiral inducers.\textsuperscript{[20,21]}

As the homemade or commercial CPL measurement systems are gradually available in the past 10 years, the development of CPL-active materials has been largely boosted. This is evidenced by appearance of numerous reviews on related topics, including CPL-active organic small molecules,\textsuperscript{[4,12,13]} metal complexes,\textsuperscript{[14–17]} conjugated polymers,\textsuperscript{[18,19]} supramolecular assemblies,\textsuperscript{[2,22,23]} chiral metal clusters and inorganic nanomaterials,\textsuperscript{[25–27,48]} and AIEgens and mesogens,\textsuperscript{[20,21,32–34]} as well as optoelectronic and bioimaging applications of these materials.\textsuperscript{[6–10]} Considering the importance of metal complexes in the development of CPL-active materials and the aggregation strategy in enhancing the performance of isolated molecules, we summarize herein the recent advance in the development of CPL-active materials and the aggregation strategy in the perspective on the future development of this area.

## 2 GENERAL PRINCIPLE AND CHARACTERIZATION METHOD OF CPL

Considering that the physical basis and related theory of CPL have been fully discussed in a number of reviews,\textsuperscript{[2,11,15,29,49]} only a brief overview of basic principle and information is highlighted herein. CPL spectroscopy measures the intensity difference ($\Delta I$) between left- and right-handed CPL (L-CPL and R-CPL) of a chiral emitter, as defined by Equation (1).

$$\Delta I = I_L - I_R$$  \hspace{1cm} (1)

wherein $I_L$ and $I_R$ describe the intensity of L-CPL and R-CPL, respectively. The polarized degree of CPL is assessed by $g_{\text{lum}}$ ($g_{\text{EL}}$ is used for electroluminescence), as defined by Equation (2).

$$g_{\text{lum}} = \frac{(I_L - I_R)}{(1/2)(I_L + I_R)}$$ \hspace{1cm} (2)

The extra factor of $(1/2)$ in Equation (2) is purposely introduced to keep consistent with the absorption dissymmetry factor $R_{\text{CD}}$ (CD: circular dichroism; or $g_{\text{abs}}$) at definition, which is given by Equation (3).

$$g_{\text{CD}} = \frac{(\varepsilon_L - \varepsilon_R)}{(1/2)(\varepsilon_L + \varepsilon_R)}$$ \hspace{1cm} (3)

where $\varepsilon_L$ and $\varepsilon_R$ refer to the molar absorption coefficients for the left- and right-handed CP light, respectively. Experimentally, the $g_{\text{CD}}$ value is determined according to Equation (4).

$$g_{\text{CD}} = \frac{\varepsilon}{32,980 \times \text{absorbance}}$$ \hspace{1cm} (4)

in which the values of “ellipticity” (unit: mdeg) and “absorbance” can be both directly obtained from the CD spectral measurement. The $g_{\text{lum}}$ factor could be theoretically expressed by Equation (5).

$$g_{\text{lum}} = 4 \frac{|\mu|m|\cos \vartheta|}{|\mu|^2 + |m|^2}$$ \hspace{1cm} (5)

wherein $\mu$ and $m$ represent the electric and magnetic transition dipole moment, respectively, and $\vartheta$ is the angle between those two vectors. The $|\mu|$ value of organic molecular materials is usually much larger than $|m|$ and Equation (5) can be simplified to Equation (6).

$$g_{\text{lum}} = 4 \frac{|m|\cos \vartheta}{|\mu|}$$ \hspace{1cm} (6)

However, Equation (6) is not applicable for materials, for example, chiral Ln(III) complexes, with a large $|m|$. Instead, another parameter $r$, which equals to $|\mu|/|m|$, is introduced. In this case, Equation (5) is transformed to Equation (7).

$$g_{\text{lum}} = 4 \frac{r \cos \vartheta}{r^2 + 1}$$ \hspace{1cm} (7)

It is clear from the above equation that when $|\mu|$ equals to $|m|$ ($r = 1$) and $\vartheta$ is 0° or 180°, the theoretical maximal $g_{\text{lum}}$ of ±2 will be obtained.

In order to further assess the overall merit of CPL, the concept of CPL brightness ($B_{\text{CPL}}$), similar to the luminescence brightness ($B$) analog, is recently proposed by Di Bari and coworkers.\textsuperscript{[28]} The $B_{\text{CPL}}$ value is defined as Equation (8).

$$B_{\text{CPL}} = \varepsilon_{\lambda} \times \Phi \times \frac{|g_{\text{lum}}|}{2} = B \times \frac{|g_{\text{lum}}|}{2}$$ \hspace{1cm} (8)

wherein $\varepsilon_{\lambda}$ is molar extinction coefficient measured at the excitation wavelength ($\lambda$) and the fluorescence brightness $B$ equals to $\varepsilon_{\lambda} \times \Phi$. Equation (8) is only applicable to
solution-state samples. For solid-state samples, a related Equation (9) is introduced.

\[
B_{CPL} = \xi_{abs} \times \Phi \times \frac{|\frac{l_{lum}}{g_{abs}}|}{2} = B' \times \frac{|\frac{l_{lum}}{g_{abs}}|}{2} \quad (9)
\]

wherein the absorption efficiency \(\xi_{abs}\) is defined as the ratio between the absorbed and incident photon numbers, and measurable with an integrating sphere. Similar to \(B\) for solution-state samples, \(B'\) describes the luminescence brightness of solid-state samples.

3 | CPL-ACTIVE AGGREGATES OF SMALL-MOLECULAR METAL COMPLEXES

The exploration of CPL-active small-molecular metal complexes, mainly including chiral Ln(III) and transition-metal complexes, has attracted intense attention over the past few decades in view of their diverse coordination modes, rich excited-state properties, and high \(\Phi\) or \(\frac{l_{lum}}{g_{abs}}\) values.[14-17,21] Among them, a certain number of materials display distinct aggregation-induced or enhanced CPL via crystallization or supramolecular assembly. Chiral square-planar Pt(II) complexes are one representative material in these studies. Other metal complexes, for example, Ln(III) and linear Au(I) complexes, have also been investigated in this context. In addition, apart from monocomponent assembly, bicomponent assembly involving chiral metal complexes provides another means for the development CPL-active aggregates. The details are discussed as follows.

3.1 | Aggregates of chiral platinum(II) complexes

Chiral supramolecular assembly with desirable nanostructures, particularly chiral helical assembly, provides a powerful approach to modulate the arrangement of chiral building blocks and promote the amplification of \(\frac{l_{lum}}{g_{abs}}\).[22,50,51] In the area of CPL-active assemblies of metal complexes, most reports focus on chiral square-planar Pt(II) complexes as the assembly building block, benefiting from their appealing polymorphic aggregations and emissions associated with effective \(\pi-\pi\) stacking and/or Pt–Pt interactions.[21,52-59] For instance, You and coworkers[52] reported a pair of chiral tridentate mononuclear Pt(II) complexes (+/-)-1 that exhibited mirror-imaged red CPL (\(l_{lum} = 1.2 \times 10^{-3}\) to \(1.8 \times 10^{-3}\) at 640 nm; Table 1) upon aggregation in aqueous solutions (Figure 1A). The aggregation-induced CPL was ascribed to the formation of one-dimensional helical chain structures via multiple Pt–Pt, \(\pi-\pi\), and hydrophobic–hydrophobic interactions. Xiang and coworkers[53] prepared the tetradentate mononuclear Pt(II)–salen complexes (R/S)-2a and 2b with a chiral binaphthyl bridge (Figure 1B). The cast films of 2a and 2b showed aggregation-induced near-infrared (NIR) phosphorescence and amplified CPL with \(\frac{l_{lum}}{g_{abs}}\) of \(3 \times 10^{-3}\) to \(5 \times 10^{-3}\) at ca. 650 nm (\(\Phi = 1.8\%–5\%\); Table 1), as a result of the restriction of the intramolecular motions of the 1,1’-binaphthyl unit and the formation of helical molecular packing structures. The uses of different substituents (F or NEt2) on the salen ligand seem to have a minor effect on the CPL performance.

In order to improve the supramolecular assembly ability and further amplify \(\frac{l_{lum}}{g_{abs}}\), mononuclear Pt(II) complexes modified with long alkyl chains have been investigated. For example, Haino and coworkers[54] reported two enantiomeric mononuclear Pt(II) complexes (R/S)-3a possessing the bis(phenylisoxazolyl)phenylacetylene ligand with chiral alkoy chains (Figure 1C). Mirror-imaged enhanced CPLs with high \(\frac{l_{lum}}{g_{abs}}\) of \(1 \times 10^{-2}\) at 535 nm were detected for the helical supramolecular assemblies of (R/S)-3a formed in toluene, while no distinct CPL was observed for the non-helical assembly obtained from the CHCl3 solution. Subsequently, these authors further reported another chiral Pt(II) complex (S)-3b modified with multiple achiral alkoy chains, which showed similar aggregation-induced CPL with \(g_{abs}\) of \(1.1 \times 10^{-2}\) at 576 nm as gel in 1-decanol.[55] Although the aggregated samples of 3a showed high \(\frac{l_{lum}}{g_{abs}}\) in the \(10^{-2}\) order, these materials displayed low \(\Phi\) of less than 10%.[54] Recently, Gong and Zhong reported a pair of chiral Pt(II) complexes (R/S)-4 that were able to form aggregates with both high \(\frac{l_{lum}}{g_{abs}}\) and \(\Phi\) (Figure 1D).[56] By controlling the assembly pathway (cooperative vs. isodesmic), helical microribbon or nanofiber assemblies with an opposite supramolecular chirality and different emission colors could be obtained from 4 with the same molecular chirality. The helical ribbon samples Co-(R/S)-4 (Co stands for cooperative assembly) exhibited yellow phosphorescent emissions with \(g_{abs}\) of \(2.2 \times 10^{-2}/4.2 \times 10^{-2}\) at 555 nm and \(\Phi\) of \(36%/33\%\), respectively. In comparison, the helical nanofibers Iso-(R/S)-4 (Iso stands for isodesmic assembly) displayed red emissions with \(g_{abs}\) of \(2.7 \times 10^{-2}/2.8 \times 10^{-2}\) at 660 nm and \(\Phi\) of \(25%/27\%\), respectively. The structure of platinum complexes with a suitable chiral inducer has a significant impact on the emission and CPL properties of the obtained aggregate. Similar Pt(II) complex enantiomers (D/L)-5 modified with the chiral leucine methyl ester afforded nonhelical assemblies with red CPL with lower \(g_{abs}\) of \(8 \times 10^{-2}/7 \times 10^{-3}\) at 590 nm but higher \(\Phi\) of 63%/67% with respect to (R/S)-4 (Figure 1E).[57]

In addition to mononuclear Pt(II) complexes, chiral binuclear Pt(II) complexes have been used in CPL studies. Xiang and coworkers[58] reported the bidentate binuclear Pt(II) complexes (R,R,R,S,S,S)-6a and 6b bridged by the axially chiral binaphthyl quinoline (Figure 2A). Among them, chiral complexes 6a and 6b exhibited distinct aggregation-induced NIR phosphorescence and CPL in the crystalline solid state (\(\frac{l_{lum}}{g_{abs}} = 6 \times 10^{-3}\) to \(1 \times 10^{-2}\) at 660–672 nm, \(\Phi = 1.6\%–2.3\%\); Table 1), similar to the mononuclear Pt(II) complex 2 reported by the same group. Very recently, Xu and coworkers[59] reported the enantiomeric binuclear Pt(II) metallacycles (R/S)-7, which displayed aggregation-enhanced CPL with \(\frac{l_{lum}}{g_{abs}}\) of \(1.4 \times 10^{-2}\) at 600 nm in mixed CH3Cl/hexane (S/S) promoted by the Pt–Pt interaction-involved hierarchical assembly (Figure 2B).

In the above-discussed examples of Pt(II) complexes, some of them (3, 4, and 7) are modified with long alkyl chains to assist assembly. If Pt(II) complexes are functionalized with multiple alkyl chains with efficient lengths, they could be turned into liquid crystals (LCs), which are important soft materials and widely applied in optoelectronic fields.[60-63] In the past decade, a number of chiral emissive
FIGURE 1: Circularly polarized luminescence (CPL)-active aggregates of mononuclear Pt(II) complexes. (A) Aggregation-enhanced CPL of (+/-)-1. Reproduced with permission: Copyright 2014, American Chemical Society.[52] (B) Molecular structures of (R/S)-2a and 2b (left) and single-crystal X-ray structure and intermolecular helical packing of (R/S)-2a (right). Reproduced with permission: Copyright 2018, John Wiley and Sons.[53] (C) Molecular structures of (R/S)-3a and 3b (left) and assembly-dependent CPL of 3a. Reproduced with permission: Copyright 2015, The Royal Society of Chemistry.[54] (D) (i) Molecular structures of (R/S)-4, (ii and iii) transmission electron microscopy images of different helical assemblies of (R)-4, and (iv and v) CPL spectra of different helical assemblies of (R/S)-4 casted on quartz. Reproduced with permission: Copyright 2021, Springer Nature.[56] (E) Assembly-enhanced CPL of (R/S)-5. Reproduced with permission: Copyright 2021, John Wiley and Sons.[57]

FIGURE 2: Circularly polarized luminescence (CPL)-active aggregates of binuclear and liquid crystalline (LC) mononuclear Pt(II) complexes. (A) Molecular structures of 6a and 6b (left) and circular dichroism (CD) spectra in CH3CN and g_lum spectra of (R,R,R,S,S,S)-6a as cast films (right). Reproduced with permission: Copyright 2019, The Royal Society of Chemistry.[58] (B) Molecular structure of 7.[59] (C) (i) Molecular structure, (ii) heliconical architecture in SmC* phase, and (iii) photoluminescence (PL), CPL, and g_lum spectra of 8. Reproduced with permission: Copyright 2020, American Chemical Society.[68] (D) (i) Molecular structure, (ii) two stereoisomeric conformations, and (iii) chiral self-organization of 9 induced by biased symmetry breaking in mesophase. Reproduced with permission: Copyright 2021, John Wiley and Sons[69]
TABLE 1  Circularly polarized luminescence (CPL) parameters of chiral small metal complexes based aggregates

| Materials | \( \lambda_{\text{em}} \) (nm) | \( \Phi \) (%) | \( \theta_{\text{em}} \) | Ref. |
|-----------|-----------------|--------|-----------------|-----|
| (+/-)-1\(^a\) | 640 | NR | \( 1.2 \times 10^{-3}/-1.8 \times 10^{-3} \) | [52] |
| (R/S)-2\(^b\) | 650 | 1.8 | \( 5 \times 10^{-3}/-5 \times 10^{-3} \) | [53] |
| (R/S)-3\(^b\) | 655 | 5.0 | \( 3 \times 10^{-3}/-3 \times 10^{-3} \) | [53] |
| (R/S)-3\(^a\) | 535 | 2.3–9.3 | \( -1 \times 10^{-2}/1 \times 10^{-2} \) | [54] |
| (S)-3b\(^d\) | 576 | NR | \( 1.1 \times 10^{-2} \) | [55] |
| Co-(R/S)-4\(^b\) | 555 | 36/33 | \( -2.2 \times 10^{-2}/2.2 \times 10^{-2} \) | [56] |
| Iso-(R/S)-4\(^b\) | 660 | 25/27 | \( 2.7 \times 10^{-2}/-2.8 \times 10^{-2} \) | [56] |
| (D/L)-5\(^b\) | 590 | 63/67 | \( 8 \times 10^{-3}/-7 \times 10^{-3} \) | [57] |
| (R,R/R/S/S,S)-6\(^a\) | 660 | 2.3/2.2 | \( -6 \times 10^{-3}/+6 \times 10^{-3} \) | [58] |
| (R,R/R/S,S,S)-6\(^b\) | 672 | 1.6 | \( -1 \times 10^{-2}/+1 \times 10^{-2} \) | [58] |
| (R/S)-7\(^b\) | 600 | NR | \( 1.4 \times 10^{-2}/1 \times 10^{-2} \) | [59] |
| (R/S)-8\(^b\) | 608 | 46 | \( 4.0 \times 10^{-2}/-4.3 \times 10^{-2} \) | [68] |
| (A/A)-9\(^b\) | 630 | NR | \( +3.1 \times 10^{-3}/-3.4 \times 10^{-3} \) | [69] |
| (R/R,S)-10\(^b\) | 500 | 15 | \( 4 \times 10^{-3}/-4 \times 10^{-3} \) | [70] |
| (R/S)-11\(^b\) | 594 | NR | \( -0.190.19 \) | [71] |
| (R/S)-11\(^b\) | 594 | NR | \( -0.110.13 \) | [71] |
| (+/-)-12\(^a\) | 595 | 0.2 | NR | [72] |
| (+/-)-12\(^b\) | 595 | 0.31 | 0.43 | [72] |
| (+/-)-12\(^c\) | 595 | 0.68 | 1.21 | [72] |
| (+/-)-12\(^d\) | 595 | 1.04 | 1.45/-1.44 | [72] |
| 13+-14\(^b\) | 615 | 1.0 | \( -6.4 \times 10^{-2}/5 \times 10^{-2} \) | [74] |
| (R/S)-15+-BPEA\(^b\) | 560 | NR | \( -2.5 \times 10^{-3}/0.1 \times 10^{-3} \) | [75] |
| (R/S)-15+-TPE\(^b\) | 470 | NR | \( -3.0 \times 10^{-3}/5 \times 10^{-3} \) | [75] |
| (R/S)-15+-TPP-4M\(^b\) | 410 | NR | \( -3.0 \times 10^{-3}/5 \times 10^{-3} \) | [75] |
| (R/S)-16+-17\(^b\) | 450–540 | NR | \( 1.6 \times 10^{-3}/-1.45 \times 10^{-3} \) | [76] |

Abbreviations: BPEA, 9,10-bis(phenylethynyl)anthracene; NR, not reported; TPE, tetraphenyl ethylene; TPP-4M, tetra(4-methoxyphenyl)pyrazine.

LCs have been developed as promising CPL-active molecular materials. The highly arranged helixes in LC phase is proved beneficial for the amplification of \( \theta_{\text{em}} \). In this context, Luo and coworkers reported the CPL-active LCs of the chiral bidentate Pt(II) complexes (R,S)-8, showing largely enhanced CPL with \( \theta_{\text{em}} \) of around \( 4 \times 10^{-2} \) at 608 nm and \( \Phi \) of 46% in the high-temperature chiral smectic (SmC\(^*\)) phase (Figure 2C). Another interesting example reported by these authors is the tetradentate Pt(II) complex 9 (Figure 2D). The biased symmetry breaking of the racemic LCs of 9 self-organized into an enantiomERICally enriched single domain without selection of handedness in twist grain boundary (TGB\(^{11}\)) mesophase. By using this homochiral LC films with determined handedness as a template, CPLs with \( \theta_{\text{em}} \) up to \( 3.4 \times 10^{-3} \) at 630 nm were obtained, demonstrating a promising means to achieve chiral assemblies from achiral molecules. It should be noted that the CPL origins of LCs 8 and 9 are largely caused by the chirality amplification of the intrinsic point or helical chirality of these molecules, which is different from the mechanism based on the CP light reflection of N*-LC.

3.2  Aggregates of other metal complexes

Apart from chiral Pt(II) complexes, CPL-active aggregates of Au(I) complexes, featuring linear molecular structures and potential Au–Au interactions, have received attention. For example, Tang and coworkers presented two enantiomeric binuclear Au(I) complexes (R,R/S,S)-10 linked by the 1,2-diisocyano-1,2-diphenylethane ligand (Figure 3A). Under the stimuli of mechanical force, the powders of 10 can be transformed from non-emissive isolated crystallites to highly emissive well-defined microcrystals, mainly as a result of subtle molecular motions driven by multiple aurophilic, C–H···F and π–π inter/intramolecular interactions. The
emission of 10 could be quenched by heating and further recovered by cooling and scratching. More interestingly, the emissive microcrystals displayed enhanced CPLs with $\delta_{\text{lum}}$ of $4 \times 10^{-3}$ at 500 nm and $\Phi$ of 15%.

Chiral lanthanide complexes represent one important type of metal complexes for CPL studies. Considering their excellent performance in solutions, researchers are interested in knowing whether the CPL properties could be further improved by assembly or agglomeration. Okayasu and Yuasa reported the chiral neutral Eu(III) complexes (R/S)-11a and 11b, in which 11a could form ensembles via coordination between the pendant NO$_2$ group and the Eu(III) center under UV irradiation. Reproduced with permission: Copyright 2016, Royal Society of Chemistry.

(B) (i) Molecular structures of (S)-11a (R = NO$_2$), 11b (R = CN) and (ii) plots of emission intensity ($I_{\text{obs}}$) at $\lambda = 594$ nm versus the angle ($\theta$) between the rotateable 2/4 filter and the fixed linearly polarized (LP) plate for (S)-11a and (S)-11b ensemble. Reproduced with permission: Copyright 2018, Royal Society of Chemistry.

(C) (i) Molecular structures of (+)-12a, (+)-12c, and (ii) CPL and photoluminescence (PL) of (+)-12a–12d in chloroform (dashed lines) and CH$_2$Cl$_2$/hexane (1/24) mixture. Reproduced with permission: Copyright 2016, Royal Society of Chemistry.

Figure 3 Circularly polarized luminescence (CPL-active) aggregates of other metal complexes. (A) (i) Molecular structures of (R,R,S,S)-10, (ii) photos under ultraviolet (UV) irradiation of (R,R)-10 powder in different states upon scratching or heating/cooling, and (iii) CPL spectra and photos (inset) of ground 10 under UV irradiation. Reproduced with permission: Copyright 2020, American Chemical Society.

3.3 Binary aggregates of metal complexes

In comparison with the aforementioned self-assemblies, chiral co-assemblies possess several advantages, for example, the avoidance of tedious synthesis and the self-emission quenching of monocomponent assemblies, and the potential to tune CPLs by energy transfer. These advantages make co-assembly an appealing strategy to obtain CPL-active aggregate materials. Several CPL-active binary aggregates of metal complexes have been prepared in past few years either by direct supramolecular co-assembly with the assistance of various noncovalent interactions or by a doping method. For instance, You and coworkers reported the CPL-active helical co-assemblies of the achiral bidentate Pt(II) complex 13 with a small fraction of the chiral Pt(II) analog (R/S)-14 (Figure 4A). The co-assemblies displayed much improved $\delta_{\text{lum}}$ values (0.05–0.064 at 615 nm; $\Phi = 0.01$) relative to those (<$10^{-3}$) of individual molecules of (R/S)-14. Additionally, Tang and coworkers constructed helical structures via the hierarchical self-assembly of the chiral binuclear Au(I) complexes (R/S)-15, which could serve as the chiral template to co-assemble with other achiral dyes, for example, 9,10-bis(phenethyl)anthracene (BPEA), tetraphenyl ethylene (TPE), and tetra(4-methoxyphenyl)pyrazine (TPP-4M), to realize tunable CPLs with $\delta_{\text{lum}}$ of 1.0 $\times$ 10$^{-3}$ to 5.0 $\times$ 10$^{-3}$ at 410–560 nm (Figure 4B and Table 1). Chen and coworkers recently prepared the organometallic double salts of the chiral cationic Au(I) complex (R,R,S,S)-16 with the achiral anionic Au(I) analog 17 in doped polymer films and in ground powder (Figure 4C). These binary materials showed the excitation wavelength-dependent CPL with $\delta_{\text{lum}}$ of 1.45 $\times$ 10$^{-3}$ to 1.6 $\times$ 10$^{-3}$ at 450–540 nm originated from the multiple excited states due to the varied Au(I)–Au(I) distances in ground state of aggregates. An anticounterfeiting application of the double salts of 16 and 17 has been demonstrated on the basis of the excitation wavelength-dependent emissions.
4 | CPL-ACTIVE AGGREGATES OF COORDINATION HELICATES, POLYMERS, AND FRAMEWORKS

Aggregates of small-molecular metal complexes are assembled together by various noncovalent interactions. In comparison, those of metal-organic helicates, polymers (MOPs) and frameworks (MOFs) are linked together by coordination bonds with much improved stability. The constructions of CPL-active coordination helicates, MOPs, and MOFs have attracted much attention in the past decade and some of them are of practical use in enantioselective recognition and separation, asymmetric catalysis, nonlinear optics, etc.\[77–84\]

4.1 | CPL-active coordination helicates

Chiral metal-ligand coordination materials with determined spatial geometrical structures, for example, molecular helicates and cages, are very promising in terms of creating novel CPL-active materials. Related works have been reported by several research groups separately or cooperatively and some materials show intense CPLs in solid states. Through the traditional chiral resolution by high performance liquid chromatography (HPLC), Ono et al.\[78\] prepared a series of enantiomeric triple-stranded coordination helicates (P/M)-18a–18c comprised of Al(III) ions and achiral tetradenate ligands with methyl substituents at different positions (Figure 5A). These enantiomeric Al(III) helicates displayed mirror-imaged tunable CPLs with $|g_{\text{lum}}|$ in the range of $5 \times 10^{-4}$ to $4 \times 10^{-3}$ at 450–586 nm and $\Phi$ of 10%–50% both in solution and solid states (Table 2). Kawai and coworkers\[79\] fabricated the $D_2$-symmetrical homochiral circular Eu(III) helicates (R/S)-19 via ligand-to-ligand interactions between the chiral bis(4-phenyl-2-oxazolinyl)pyridine and achiral bis-$\beta$-diketonate ligands (Figure 5B). The enantiomers (R/S)-19 showed distinct CPL with $|g_{\text{lum}}|$ of 0.22–0.24 at 591 nm and $\Phi$ of 5% in solid state with the identical spectral signatures as that in solution, indicating their stable helicate conformation in both states. In addition, Kawai and coworkers\[80\] constructed the $D_4$-symmetrical octanuclear circular Ln(III) helicates (R/S)-20a and 20b composed of the achiral trianionic tris-$\beta$-diketonate (THP) and chiral bis(4-isopropyl-2-oxazolinyl)pyridine (‘Pr-pybox) ligands.
FIGURE 5 Circularly polarized luminescence (CPL-active) coordination helicates. (A) (i) Molecular structures of 18a–18c, (ii) single crystal X-ray structures of (P)-18a–18c, (iii) CPL and photoluminescence (PL) spectra of (P/M)-18a–18c in (iii) CH2Cl2 or (iv) KBr pellets. Reproduced with permission: Copyright 2020, John Wiley and Sons.[78] (B) Molecular structure and CPL and PL spectra in CHCl3 (solid line) and KBr pellet (dash line) of (R/S)-19. Reproduced with permission: Copyright 2018, American Chemical Society.[79] (C) (i) Synthetic pathway of (R/S)-20a and 20b, (ii) CPL spectra in KBr pellets and PMMA films, and (iii) PL images in CHCl3 (upper) and PMMA films (lower) with a bandpass filter (592 nm) and (a) left-handed and (b) right-handed circularly polarized filter of helicates (R/S)-20a. Reproduced with permission: Copyright 2020, American Chemical Society[80] with Eu(III) or Tb(III) ion, respectively (Figure 5C). Similar to the homochiral Eu(III) helicates 19 mentioned above, the homocentric 20a and 20b are prone to be formed by the ligand-to-ligand interactions between THP and Pr-pybox, and exhibit remarkable CPL with $g_{\text{lum}}$ values of 0.17 at 590 nm and total $\Phi$ of 30% with respect to the chiral monomer (+)-22 ($g_{\text{lum}} = 0.09$ at 586 nm). In addition, the chiral mixed Eu(III)–Sm(III) polymer (+)-21b showed similar $g_{\text{lum}}$ yet enhanced total $\Phi$ of 52%, which was believed to be caused by the increase in the ligand-to-metal charge transfer (LMCT) level with the presence of equivalent Sm(III) ions and partial inhibition of the self-emission quenching of the Eu(III) ions.[84]

4.2 CPL-active coordination polymers and frameworks

Chiral MOPs are attractive candidates for CPL-active aggregate materials. Relative to corresponding monomeric complexes, chiral coordination polymers generally display much higher luminescence efficiencies and larger $g_{\text{lum}}$ as revealed by recent reports.[81–84] Among them, Hasegawa and coworkers[83,84] reported the spiral Eu(III) MOPs (+/-)-21a consisting of the chiral [Eu(tfc)3] segment (tfc: 3-trifluoroacetyl camphorate) and the achiral 4,4-bis(diphenylphosphoryl)bisphenyl (dpdp) ligand (Figure 6A). The solid-state chiral polymer (+)-21a exhibited enhanced CPL with $g_{\text{lum}}$ of 0.17 at 590 nm and total $\Phi$ of 30% with respect to the chiral monomer (+)-22 ($g_{\text{lum}} = 0.09$ at 586 nm). In addition, the chiral mixed Eu(III)–Sm(III) polymer (+)-21b showed similar $g_{\text{lum}}$ yet enhanced total $\Phi$ of 52%, which was believed to be caused by the increase in the ligand-to-metal charge transfer (LMCT) level with the presence of equivalent Sm(III) ions and partial inhibition of the self-emission quenching of the Eu(III) ions.[84]

CPL-active MOFs are appealing functional materials in light of their unique nanoporous crystalline structures and potential applications in enantiomeric separation, chiral sensors, asymmetric catalysis, etc. Chiral emissive MOFs could be formed by the direct coordination assembly from chiral ligands or achiral ligands accompanied by symmetry breaking.[85–87] For instance, Li and coworkers[85] recently fabricated a series of homochiral emissive Zn-imidazolate MOFs (P/M)-23a and 23b and coordination cages ($\Delta$/$\Lambda$)-24 based on the in situ formed chiral tetradeate ligands with different substituents (H or Me; Figure 6B). The coordination cages ($\Delta$/$\Lambda$)-24 are CPL-silent, while the Zn-MOFs...
23a and 23b showed CPLs with $|g_{\text{lum}}|$ of $1.0 \times 10^{-3}$ to $2.3 \times 10^{-3}$ at 485–520 nm and $\Phi$ of 4.9%–6.4%, benefiting from the formation of trifold coordination helical chains. In addition, homochiral MOFs were constructed by Liu and coworkers[86] via the spontaneous assembly of AIEgens of tetrakis(4-pyridylphenyl)ethylene and chiral camphoric acid with Cd(II) ions, which showed interesting green and blue dual-mode CPLs under external mechanical forces.

Symmetry breaking provides an interesting means to obtain chiral materials. On the basis of symmetry breaking, homochiral MOF (−)-25 was prepared from the achiral tris(4′-carboxy-1,1′-biphenyl)amine (TCPA) ligand and Cd(II) ions (Figure 6C).[87] Remarkably, it exhibited interesting CPL ($g_{\text{lum}} = -2.7 \times 10^{-3}$ at 480 nm; $\Phi = 43.2\%$) and room temperature phosphorescence (RTP; $\Phi = 6.1\%$) simultaneously, attributed to the coordination-driven chiral assembly and well-defined arrangement of TCPA in crystal lattice.

4.3 Dye-encapsulated coordination frameworks

The high porosity and stability of MOFs is beneficial to act as a host system for the encapsulation of functional dyes to build host–guest materials. The host MOFs could be either self-emissive or non-emissive. After the incorporation of guest dyes, potential energy and chirality transfer processes will allow to greatly tune the photophysical properties of the obtained host–guest MOFs.[88–92] The above discussed MOF (−)-25 has been used to load different dyes to modulate the emission colors; however, no CPL properties were reported for the obtained host–guest MOFs.[87] Liu and coworkers[88] manufactured the chiral host–guest systems 26a–26g by loading various dyes into the emergent cubic void of the in situ formed MOFs from γCD and K⁺ ions (Figure 7A). These dye-encapsulated γCD-MOFs display tunable emissions and boosted CPLs. Notably, the sizes of the guest dyes significantly affect their located positions in the frameworks as well as the CPL signs and magnitudes (Table 2). With the inclusion of most of the dyes studied, the γCD-MOFs show negative CPLs. However, the CPL sign of the ruthenium trisbipyridine (Ru-BPY)-loaded MOF 26d is uncontrollable, possible due to the weak host–guest interaction in this case.[88] Another series of CPL-active MOFs 27a–27e were disclosed by Zang and coworkers[89] (Figure 7B). Two enantiomeric Zn-MOFs (D/L)-27a with inner helical channels were obtained from the reaction Zn²⁺ ion with a chiral amino acid-derived ligand, which were used as chiral templates to incorporate a diversity of achiral dyes to realize red (27e), green (27d), blue (27c), and white (27b) CPLs with $|g_{\text{lum}}|$ of $1.0 \times 10^{-3}$ to $11.5 \times 10^{-3}$ at 435–618 nm and $\Phi$ of 19%–66% (Table 2). The confinement effect of chiral nanopores in these frameworks plays a vital role in enhancing the tunable CPLs of encapsulated dyes.

On the basis of layer-by-layer dye encapsulation, Gu and coworkers[90] recently fabricated the CPL-active Zn-MOF thin films (D/L)-29a–29d by incorporating various achiral [Ln(acac)₃] complexes (acac: acetylacetone; Ln: Eu, Tb, Gd, and a mixture of different ions, respectively) into the pores of the surface-confined chiral MOF thin films...
FIGURE 7 Circularly polarized luminescence (CPL)-active dye-loaded metal-organic frameworks (MOFs). (A) Construction of γCD-MOFs 26a–26g with different achiral guest dyes. Reproduced with permission: Copyright 2020, John Wiley and Sons.[88] (B) (i) Formation of chiral Zn-MOFs (D/L)-27a–27e, (ii) chemical structures of doped dyes, and (iii) CPL spectra of dye-contained Zn-MOFs 27c–27e. Reproduced with permission: Copyright 2020, John Wiley and Sons.[89] (C) Fabrication of CPL-active MOF thin films 29a–29d enhanced by energy transfer process. Reproduced with permission: Copyright 2021, Springer Nature.[90] (D) MOFs (D/L)-30a with photo-switchable downshifting CPL and the upconversion nanoparticles (UCNP)-containing MOFs (D/L)-30b with photo-switchable upconversion CPL. Reproduced with permission: Copyright 2021, John Wiley and Sons.[91]

Aggregates of MOFs (D/L)-30a, [Zn$_2$((D/L)-cam)$_2$]$_n$[dabco]$_n$ (cam: camphorate; dabco: 1,4-diazobicyclo[2.2.2]octane) (Figure 7C). MOFs (D/L)-29a–29d showed mirror-imaged amplified red, green, blue, and white CPL (|$\ell_{\text{gum}}$| = 7.4 × 10$^{-3}$ to 9.2 × 10$^{-3}$ at 450–614 nm; Table 2) benefited from the effective energy transfer from [Ln(acac)$_3$] to the framework skeleton. In comparison, the simple physical mixtures of [Ln(acac)$_3$] and 28 are CPL-silent due to the inefficient energy and chirality transfer. Energy-transfer processes were also employed by Duan and coworkers[91] for CPL amplification. The light-responsive CPL-active MOF 30a was prepared by loading the diarylethene derivative (DAEC) as a photoswitch into the chiral emissive Ln(III) MOFs with the 1,3,5-tris(4-carboxyphenyl)benzene ligand (Figure 7D). Under alternating UV and visible light irradiation, the downshifting (DS) CPL of 30a was reversibly switched on and off as DAEC took a closed (DAECc) or open (DAECo) conformation. By further introducing energy-level-matched upconversion (UC) nanoparticles (NPs) into MOFs 30a, another MOF 30b with switchable UC-CPL was realized upon NIR and visible light irradiation. Relative to the DS-CPL of MOFs 30a ($|\ell_{\text{gum}}|$ = 0.014–0.020 at 546 nm), the UC-CPL of 30b showed improved $|\ell_{\text{gum}}|$ of 0.078 at 546 nm as a result of the effective energy transfer from the chiral MOF to the ring-closed DAEC components.[91]

5 APPLICATIONS OF CPL-ACTIVE COORDINATION AGGREGATES

With a great number of CPL-active molecules and materials being invented, their applications have received increasing attention.[2,6–10] For CPL-active metal-ligand coordination aggregates, the most interesting applications to date include CP-OLEDs, CPL-contrast biological imaging, and CPL probes.[1,8–10]

5.1 CP-OLEDs

Compared to the traditional method of obtaining CP light by using a polarizer and quarter-wave plate to filter out equal amount light with an opposite CP direction, devices with self-emissive CPL, for example, CP-OLEDs, can avoid such complicated filtering process and corresponding energy loss. Consequently, great effort has been devoted to the fabrication process...
TABLE 2 Circularly polarized luminescence (CPL) parameters of metal-ligand coordination helicates, polymers, and frameworks

| Materials | \( \lambda_{\text{em}} \) (nm) | \( \Phi \) (%) | \( \delta_{\text{lum}} \) | Ref. |
|-----------|-----------------|----------------|-----------------|-----|
| (PIM)-18a | 450             | 14             | –5.0 \times 10^{-4}/5.0 \times 10^{-4} | [78] |
| (PIM)-18b | 568             | 10             | –4.0 \times 10^{-3}/4.0 \times 10^{-3} | [78] |
| (PIM)-18c | 586             | 50             | –3.0 \times 10^{-3}/3.0 \times 10^{-3} | [78] |
| (RIS)-19c | 591             | 5              | 0.22/–0.24       | [79] |
| (RIS)-20a | 592             | 14.5           | –1.25/1.24       | [80] |
| (RIS)-20b | 541             | 0.13           | –0.25/0.25       | [81] |
| (+-21)α   | 590             | 30             | 0.17             | [82] |
| (+-21)β   | 590             | 52             | 0.15             | [84] |
| (PIM)-23a | 520             | 6/6.4          | –2.3 \times 10^{-3}/2.3 \times 10^{-3} | [85] |
| (PIM)-23b | 485/488         | 5.6/4.9        | –1 \times 10^{-1}/1 \times 10^{-1} | [85] |
| (++)-25b  | 480             | 43.2           | –2.7 \times 10^{-3} | [87] |
| 26a       | 450             | 34.7           | –1.75 \times 10^{-2} | [88] |
| 26b       | 514             | 11.9           | NR              | [88] |
| 26c       | 614             | 1.0            | –6 \times 10^{-3} | [88] |
| 26d       | 611             | NR             | ±8 \times 10^{-3} | [88] |
| 26e       | 611             | 16.9           | –1.5 \times 10^{-2} | [88] |
| 26f       | 670             | 4.83           | –1.1 \times 10^{-2} | [88] |
| 26g       | 501             | 69.3           | –2 \times 10^{-3} | [88] |
| (DIL)-27a | NR              | NR             | 1.5 \times 10^{-3}/1.5 \times 10^{-3} | [89] |
| (DIL)-27b | White           | 30             | 1 \times 10^{-3}/1 \times 10^{-3} | [89] |
| (DIL)-27c | 435             | 66             | 11.5 \times 10^{-3}/11.5 \times 10^{-3} | [89] |
| (DIL)-27d | 535             | 28             | 1.6 \times 10^{-3}/1.6 \times 10^{-3} | [89] |
| (DIL)-27e | 618             | 19             | 5.2 \times 10^{-4}/5.2 \times 10^{-4} | [89] |
| (DIL)-29a | 614             | NR             | –9.2 \times 10^{-3}/9.2 \times 10^{-3} | [90] |
| (DIL)-29b | 550             | NR             | –8.5 \times 10^{-3}/8.5 \times 10^{-3} | [90] |
| (DIL)-29c | 450             | NR             | –7.4 \times 10^{-3}/7.4 \times 10^{-3} | [90] |
| (DIL)-29d | White           | NR             | –8.9 \times 10^{-3}/8.9 \times 10^{-3} | [90] |
| (DIL)-30a | 546             | 52/51          | –1.4 \times 10^{-2}/2.0 \times 10^{-2} | [91] |
| (DIL)-30b | 546             | NR             | –7.8 \times 10^{-2}/7.8 \times 10^{-2} | [91] |

Abbreviation: NR, not reported.

aKBr pellet.
bAggregate in CHCl3.
cSolid state.
dCrystal.
eAssembled thin film.

of efficient CP-OLEDs on the basis of CPL-active molecular materials. Among them, metal complexes are appealing due to their efficient harvesting of electrically produced excitons. Corresponding CP-OLEDs can be fabricated through either the solution- or evaporation-processed procedures depending on the solubility, stability, and sublimability of molecular materials.

The first solution-processed CP-OLEDs was reported by Meijer and coworkers[93] by using chiral poly(p-phenylenevinylene) (PPV) derivative as single-component emissive layer \( g_{\text{EL}} = –1.26 \times 10^{-3} \) at 600 nm. Subsequent works revealed that the chiral arrangement of aggregated emissive polymers played a significant role in enhancing CPL and CP electroluminescence[94,95] Fuchter and coworkers[96] later constructed another type of CP-OLEDs by doping the 1-aza[6]helicene enantiomer into the achiral poly[9,9-diocetylfluorene-co-benzothiadiazole] (F8BT) as the active emissive layer, displaying a high \( g_{\text{EL}} \) value of 0.2 at 575 nm even at a low doping ratio of 7%. Since then, the direct use of chiral emissive materials or doped films consisting of chiral small molecules and achiral polymers are widely adopted as the active emissive layer to fabricate CP-OLEDs. In the cases of solution-processed CP-OLEDs with chiral metal complexes, doped films are often used to improve the film quality and suppress self-emission quenching.

On the basis of the chiral doping strategy, Di Bari and coworkers[97,98] manufactured the solution-processed CP-OLEDs by blending the CPL-active ionic Eu(III) complex \( (--\text{31}) \) into polyvinylcarbazole (PVK) and 1,3-bis[2-(4-tert-butylphenyl)-1,3,4-oxidiazolo-5-yl]benzene (OXD-7) as the active emissive layer (Figure 8). The corresponding optimized devices with a semitransparent thin layer (around 6 nm) of aluminum back cathode exhibited a high \( g_{\text{EL}} \) value of \(-1.0 \) at 595 nm (comparable to the PL \( \delta_{\text{lum}} \), \( \delta_{\text{PL}} \) of \(-1.21 \) at 595 nm and \( \delta_{\text{PL}} \) of \(-1.50 \) at 595 nm), albeit with a low maximum external quantum efficiency (EQE\(_{\text{max}}\)) of 0.05% (Table 3). In contrast, when a thick layer (110 nm) of aluminum cathode was used, the \( g_{\text{EL}} \) value dropped to \(-0.15 \) at the same emission wavelength. This decrease of circular polarization is caused by the detrimental effect of the back electrode (cathode) reflection, which can reverse the spiral direction of CP light.[98] The use of a semitransparent back electrode would be helpful to reduce this effect. As shown in Table 3, the \( g_{\text{EL}} \) factors of most CP-OLEDs are smaller (comparable in a few of cases) with respect to their \( \delta_{\text{PL}} \) values.[1,6,11] Apart from the back electrode reflection, other factors such as CP light scattering, interlayer reflections, and inefficient chirality transfer of multicomponent systems are believed to be responsible for this difference.[98]

In addition to Eu(III) complexes, doped films with the enantiomeric platinahelicene (+/--)-32 were used in CP-OLEDs to show \( g_{\text{EL}} \) of 0.22 and \(-0.38 \), respectively, at 615 nm.[99] Using the dopied axial chiral platininanaphthalenes (R/S)-33 as the emitters, solution-processed CP-OLEDs with \( g_{\text{EL}} \) of ca. 1 \times 10^{-3} at about 640 nm and the maximum luminance (\( \lambda_{\text{lum}} \)) of 3500 cd m^{-2} and EQE\(_{\text{max}}\) of 2.15% were demonstrated.[100] Based on a chiral poly(3-vinylcarbazole) as the host and the axially chiral platininanaphthalenes (R/S)-34 as the dopants, You and coworkers[101] manufactured the multilayer CP-PLEDs to exhibit \( g_{\text{EL}} \) of ca. 1 \times 10^{-4} at 540 nm and EQE\(_{\text{max}}\) of 1.2%.

In spite of the above advance, the \( g_{\text{EL}} \) and EQE\(_{\text{max}}\) of CP-OLEDs are unsatisfactory. Wang and coworkers[102] recently employed the chiral LC Pt(II) complexes (R/S)-35 as the dopants in PVK/OXD-7 blend to fabricate CP-OLEDs. Upon annealing treatment at 100°C, the obtained devices presented large \( g_{\text{EL}} \) value of 0.06 and high EQE\(_{\text{max}}\) of 11.3%. In addition, the CP-OLEDs with the axially chiral dinuclear Pt(II) complexes (R/S)-36a and 36b were demonstrated by Xu and coworkers[103] to give \( g_{\text{EL}} \) of 3.0 \times 10^{-3} and EQE\(_{\text{max}}\) of 3.1%.

Metal complexes beyond Pt(II) complexes have received little interest for solution-processed CP-OLEDs. One of the few examples of the use of Ir(III) complexes is the axially chiral Ir(III)-isocyanide complexes (R/S)-37 (blended with PVK/OXD-7), which were employed by Huang and coworkers[104] to construct CP-OLEDs to show \( g_{\text{EL}} \) of 10^{-3} and the maximal current efficiency of 7.50 cd A^{-1}.


FIGURE 8 Metal complexes for circularly polarized organic light-emitting diodes (CP-OLEDs). (A) Complexes 31–37 for solution-processed devices. (B) Complexes 38–43 for evaporation-processed devices.

TABLE 3 Parameters of circularly polarized organic light-emitting diodes (CP-OLEDs)

| Material | $\lambda_{PL}$ (nm) | $g_{PL}^{c}$ | $\lambda_{EL}$ (nm) | $g_{EL}$ | EQEmax (%) | Ref. |
|----------|---------------------|--------------|---------------------|----------|------------|------|
| (−)-31° | 595/612             | −1.21/0.16   | 595/612             | −1.0/0.19| 0.05°      | [98] |
| (+/−)-32° | 625           | NR/-0.22     | 615                 | 0.22/-0.38| NR         | [99] |
| (R/S)-33a | 640          | 1.7 × 10^{-3}/1.4 × 10^{-3} | 645 | 1.2 × 10^{-3}/0.7 × 10^{-3} | 1.32/1.49 | [100] |
| (R/S)-33b | 620          | 2.6 × 10^{-3}/1.3 × 10^{-3} | 639 | 1.11 × 10^{-3}/1.02 × 10^{-3} | 2.15/2.01 | [100] |
| (R/S)-34 | 540/545       | 1.0 × 10^{-3}/0.94 × 10^{-3} | 540 | 1.09 × 10^{-4}/1.02 × 10^{-4} | 1.2/NR   | [101] |
| (R/S)-35 | 550          | 0.02/NR      | 550                 | 0.06/-0.05| 11.3       | [102] |
| (R/S)-36a | 620          | −4.2 × 10^{-3}/NR | 605 | −3 × 10^{-3}/3 × 10^{-3} | 1.4       | [103] |
| (R/S)-36b | 615          | −1.9 × 10^{-3}/NR | 597 | −2 × 10^{-3}/2 × 10^{-3} | 3.1       | [103] |
| (R/S)-37 | 527/558       | NR           | 525/555             | −1 × 10^{-3}/1 × 10^{-3} | NR        | [104] |
| (fac,Δ/Λ)-38 | 516/515 | 3.22 × 10^{-3}/3.3 × 10^{-3} | 515/522 | 6.8 × 10^{-3}/2.8 × 10^{-4} | 21       | [106] |
| (Δ,R/S)-39 | 601          | 1.1 × 10^{-3}/NR | 599 | 5 × 10^{-4}/NR | 23.7/NR  | [107] |
| (ΔL,R/S)-39 | 602       | NR/-1.1 × 10^{-3} | 599 | NR/-3 × 10^{-4} | NR/23.6    | [107] |
| (Δ,R)-40 | 520          | 5.92 × 10^{-3} | 520                 | 6.14 × 10^{-3}/7.7 × 10^{-3} | 30.6/18.8° | [108] |
| (Δ,R)-40 | 520          | −4.35 × 10^{-3} | 520 | −4.36 × 10^{-3}/6.5 × 10^{-3} | 30.5/15.2° | [108] |
| (Δ/R)-41 | 611          | −7.8 × 10^{-4}/8.4 × 10^{-4} | 647/649 | −1.2 × 10^{-4}/1.2 × 10^{-4} | 8.5/9.7   | [110] |
| (P/M)-42 | 600          | 1.88 × 10^{-3}/1.66 × 10^{-3} | 600 | 6.81 × 10^{-3}/6.49 × 10^{-4} | 12.6      | [111] |
| (R/S)-43 | 490          | −3 × 10^{-3}/3 × 10^{-3} | 490 | −4.4 × 10^{-2}/4.4 × 10^{-2} | 0.042/0.038| [112] |

Abbreviations: EQEmax, maximum external quantum efficiency; NR, not reported.
°Solution-processed.
Evaporation-processed.
The $g_{PL}$ value of photoluminescence (PL) of thin films.
With a semitransparent anode.

Though the solution processes are advantageous in simple device fabrication procedures, the EQEs of corresponding CP-OLEDs are not satisfactory. In comparison, the evaporation-processed CP-OLEDs usually show better device performances attributed to the effective balance of the hole and electron injection and transport. To meet the requirement of evaporation, chiral emitters should feature relatively small molecular weight and weak intermolecular π–π interaction. Among them, small molecular organic compounds, including those with thermally assisted delayed fluorescence (TADF) properties, and chiral neutral metal complexes have been commonly used.[1,6,105] Some representative and very
recent examples of evaporation-processed CP-OLEDs with chiral metal complexes are highlighted below. Other examples, including those with TADF molecules, have been discussed in a previous review.[111]

Chiral octahedral Ir(III) complexes have been frequently used in evaporation-processed CP-OLEDs. For instance, Zheng and coworkers[106] firstly introduced a series of stereoisomeric Ir(III) complexes, including the classical Ir(III) tris(phenylpyridine) complexes 38, for evaporation-processed multilayered CP-OLEDs. The obtained devices displayed |EL| of 2.8 × 10^{-3} to 6.8 × 10^{-4} at 512-522 nm with high EQEmax over 21%. The stereoisomeric Ir(III) complexes (Δ/R/S)-39 with the axially chiral disulfide ligand were found to give comparable performance (Table 3).[107]

By using a semitransparent aluminum or silver cathode, Yang and coworkers[108] constructed the CP-OLEDs based on the stereoisomeric iridium(III) complexes (Δ/A,R)-40 with the chiral β-diketone ligand to exhibit improved |EL| of 7.7 × 10^{-3} at 520 nm with EQEmax of 18.8%. The obtained |ET| values are about one order of magnitude larger than those measured from the conventional devices without the use of semitransparent cathodes (6.1 × 10^{-4}). The decrease of |ET| with nontransparent cathodes was caused by the spiral direction reversion of CPL by the flection of cathode, as has been discussed above on the devices with the Eu(III) complex (−)-31.[98]

Other than Ir(III) complexes, neutral Pt(II) and Zn(II) complexes have been tested in evaporation-processed CP-OLEDs. Enantiomeric platinahelicene derivatives with a similar structure as 32 have been examined as the chiral emitters in evaporation-processed CP-OLEDs to exhibit |EL| in the 10^{-3} order of magnitude and EQEmax of over 15%.[109] You and coworkers[109] employed the evaporated chiral tridentate Pt(II) complexes (R/S)-41 as the neat emissive layer to construct CP-OLEDs, yielding |ET| of 1.2 × 10^{-4} at ca. 650 nm and EQEmax of 12.6%.[111] In addition, the CP-OLEDs with the enantiomeric tetradentate Pt(II) complexes (P/M)-42 were shown recently to give |EL| of 6.8 × 10^{-3} at 600 nm and EQEmax of 12.6%.[111] One rare example using chiral Zn(II) complexes as the emitters in evaporation-processed CP-OLEDs were reported by Tang and coworkers,[112] who used the enantiomeric tetradentate Zn(II) salen complexes (R,R,S,S)-43 to achieve high |ET| of up to 4.4 × 10^{-2} at 490 nm albeit with low EQEmax of 0.042%. Interestingly, the |ET| factors of (R,R,S,S)-43 are around one order of magnitude higher with respect to their |EL| values (3 × 10^{-3}). However, the reason for this difference has not been discussed by the authors of this work, which remains to be an intriguing topic to be investigated in the future.

5.2 CPL-contrast imaging

CPL-contrast imaging is another appealing application for CPL-active metal complexes.[10] One prerequisite for CPL-contrast imaging is that the materials should possess emissions with large circular polarization. Thanks to the high gllum of Ln(III) complexes, several interesting proof-of-concept studies have been demonstrated recently. For instance, based on the enantiomeric Eu(III) complexes (Δ/Λ)-44 with both intense emission (Φ = 47%) and high gllum (0.15 at 599 nm, 0.19 at 655 nm, and 0.32 at 708 nm), Pal and coworkers[113] demonstrated the pioneering example in 2016 (Figure 9A). With the adaptation of an epifluorescence microscope for CPL analysis, time-resolved images and corresponding CPL spectra of (Δ/Λ)-44 showed a contrast ratio (CRΔ/Λ) of 3.23:1 via the right CP light channel, meaning that the L-CPL features were 3.23 times brighter than the R-CPL features. On the other hand, a CRΔ/Λ ratio of 1:3.64 was detected through the left CP light channel. Very recently, Hasegawa and coworkers[114] fabricated a chiral transparent lumino-glass (+/-)-45 composed of the chiral [Eu(tfc)3] complexes and the achiral glass promoter tris(2,6-dimethoxyphenyl)phosphine oxide ligand (Figure 9B). The high brightness (Φ = 13%) and CPL (g|llum| = 1.2 at 594 nm) of 45 favored its application as CPL-imaging security devices. Upon operation in a special CPL detection setup, in which biased L-CPL and R-CPL could selectively be obtained and imaged, a brightly visible encrypted sun or moon pattern was differentiated.

5.3 CPL probes and sensors

Polarized light is a subtle and delicate investigator of molecular condition. CPL spectrum contains rich information of excited states including the sign and intensity for each transition and fine spectral resolution of overlapping multiple emissions. It also provides information on the local chiral environment of molecular and aggregated materials. On account of these reasons, CPL spectrum has emerged as a promising analytical method to probe the substrate–analyte interactions, in particular those between Ln(III) complexes and bio-related molecules.[5,115-122]

Ln(III) complexes typically possess large gllum factors and dynamic coordination structures, making them advantageous as CPL probes. In this context, CPL could be induced either from achiral metal complexes after association with chiral analytes or from chiral metal complexes responsive to analytes. For instance, Parker and coworkers[117] prepared the racemic Eu(III) complex 46 with a danging dipyridylamino unit, which showed different affinities and CPL response upon the successive interaction with Zn(II) ions and nucleotides (Figure 10A). The adducts generated by complexation of 46 with Zn(II) displayed induced CPL with opposite chiral signs upon the addition of adenosine monophosphate (AMP) or adenosine diphosphate (ADP) versus adenosine triphosphate (ATP). This phenomenon was explained by the preferential formation of the chiral (Δ)-[46-Zn·AMP], (Δ)-[46-Zn·ADP], and (Δ)-[46-Zn·ATP] aggregate, respectively. Another interesting example is the racemic Eu(III) complex 47, which showed opposite CPL signs for upon aggregation with human or bovine serum protein z1-AGP (Figure 10B).[118]

In addition to achiral complexes, chiral Ln(III) complexes have been used as CPL probes. The Parker group have reported the use of chiral Eu(III) and Tb(III) complexes to sense HCO3⁻ and CO3²⁻ by CPL signal changes.[119-121] One interesting example is the chiral Eu(III) complex 48 with a pendant pyrazine derivative which could selectively and reversibly bind to the “drug site II” of serum albumin (Figure 10C).[122] The (S,S,S,Δ) form of 48 was shown to form aggregates with human or bovine serum albumin (BSA) to induce the inversion of CPL direction. In contrast, no CPL inversion occurred with the (R,R,R,Δ) form of 48, making it a unique chiroptical probe of albumin binding.
FIGURE 9 Eu(III) complexes for circularly polarized luminescence (CPL)-contrast imaging. (A) (i) Molecular structures of (Δ/Λ)-44, (ii) enantioselective CPL epifluorescence microscopy, and (iii) time-resolved CPL spectra (left) and contrast images (right) of (Δ/Λ)-44 using the left (upper) and right (lower) circularly polarized (CP) light channel. Reproduced with permission: Copyright 2016, The Royal Society of Chemistry 2016.[113] (B) (i) Molecular structures of (+/−)-45, (ii) CPL detection setup, and (iii) images of the films of (+/−)-45 under 365 nm irradiation and photographs of the emission at 594 nm related to (1) left and right, (2) left, or (3) right CP light from the samples. Reproduced with permission: Copyright 2020, Springer Nature[114]

FIGURE 10 Circularly polarized luminescence (CPL) probes of metal complexes. (A) (i) Structure of racemic 46, (ii and iii) optimized structure of (ii) (Λ)-[46 Zn-AMP] and (iii) (Δ)-[46 Zn-ATP] aggregate, and (iv) their CPL spectra. Reproduced with permission: Copyright 2018, John Wiley and Sons.[117] (B) CPL spectra of racemic 47 upon forming aggregates with bovine or human α1-AGP at pH of 9.3. Reproduced with permission: Copyright 2018, The Royal Society of Chemistry.[118] (C) CPL spectra of (S,S,S)-Δ-48 with gradual addition of bovine serum albumin (BSA). Reproduced with permission: Copyright 2008, The Royal Society of Chemistry[122]

6 | MECHANISM OF CPL ENHANCEMENT BY AGGREGATION

As was surveyed above, considerable progress has been made to date to enhance the |g| lum and Φ of coordination materials by aggregation. Depending on the specific material or system, the underlying mechanism of the aggregation-enhanced CPL could be very different. Generally speaking, the CPLs of aggregated materials could be either originating from the amplification of intrinsic chirality or structural chirality.[3,21,23,39] For instance, the CPLs of supramolecular helical assemblies and energy-transfer systems are typical
examples caused by the amplification of intrinsic chirality by exciton coupling. The materials discussed in Sections 3–6 are mostly associated with this type of mechanism. On the other hand, the CPL amplification by the circularly selective reflection with N*-LC is of origin of structural chirality.\(^{12,13}\)

Chiral supramolecular helical assembly has been proved to be a promising approach to amplify the chirality and [\(\varepsilon_{\text{lum}}\)] of chiral complexes.\(^{3}\) The helical assembly increases the size and long-range order of aggregated materials with respect to dispersed molecules, which are beneficial for the biased light-matter interactions to induce CPL. In addition, the exciton and magnetic coupling between neighboring chromophores are important for CPL performance. The CPL amplification is in particular distinct for the helical assemblies obtained from the metal complexes with aggregation-induced or enhanced emissions. For instance, the helical assemblies of the chiral Pt(II) complexes (\(R/S\)-3a and 3b) showed AIE CPLs with [\(\varepsilon_{\text{lum}}\)] of up to ca. 0.01 compared to their CPL-silent non-helical assemblies.\(^{54,55}\) The chiral Pt(II) complexes (\(R/S\)-4) are CPL-silent in solution, while corresponding helical assemblies showed highly amplified CPLs with [\(\varepsilon_{\text{lum}}\)] of over 0.02.\(^{56}\)

In cases when monocomponent assembly of chiral chromophores fails for the CPL amplification, binary assembly provides an alternative to obtain CPL-active aggregates. In this regard, numerous examples have been discussed in this review. For instance, the physical doping of the CPL-silent assemblies of the chiral di-Au(I) complexes (\(R/S\)-15) with various achiral emitters afforded tunable CPLs.\(^{75}\) By incorporating selected achiral emitters into the inner helical channels of the crystalline MOFs (\(D/L\)-27), amplified and tunable CPLs were achieved.\(^{89}\) By incorporating various achiral Ln complexes into the pores of the chiral Zn-MOF 28, CPL-active MOF films with [\(\varepsilon_{\text{lum}}\)] of around 10^{-2} were obtained, thanks to the effective host–guest energy transfer.\(^{190}\) Furthermore, by integrating UC process into energy-transfer systems, chiral coordination aggregates (29b) demonstrated enhanced CPLs.\(^{91}\) All of these processes are associated with the chirality transfer in excited states, possibly as a result of the coupling of \(\mu\) and \(m\) among individual components.\(^{39}\) However, more in-depth mechanism of chirality transfer remains to be investigated in the future.\(^{3}\)

The structural chirality of CPL-active materials generally refers to the situation in N*-LCs, in which chiraloptical phenomena occurs when the helical pitch of chiral supramolecular assemblies matches the length scales of emissions.\(^{3}\) The CPLs originated from structural chirality are mainly caused by circularly selective reflection/transmission of light (Bragg reflection) in N*-LCs. The [\(\varepsilon_{\text{lum}}\)] values in such circumstances are relatively high and strongly affected by the thickness of the chiral LC medium.\(^{13,23,125}\) An extremely high [\(\varepsilon_{\text{lum}}\)] value of 1.79 has been reported by Akagi and coworkers\(^{62}\) by the selective transmission of CPL emitted from chiral disubstituted polyacetylenes across the thermotropic N*-LCs. However, this method has not yet been used for the CPL amplification of chiral metal complexes. The known CPL-active LC materials of metal complexes, for example, platinum complexes 8 and 9 (Figure 2), are mainly a result of the amplification of intrinsic chirality in the highly ordered LC phases.\(^{68,69}\)

7 | CONCLUSION AND PERSPECTIVE

The research on the development and applications of CPL-active molecular materials has been largely boosted in recent years. Metal-ligand coordination aggregates are characterized by high [\(\varepsilon_{\text{lum}}\)] and \(\Phi\) which are important from the viewpoint of practical optoelectronic and biological applications. This review highlights the recent progresses in CPL-active coordination aggregates, including those assembled from discrete small-molecular metal complexes and coordination helicates, polymers, and frameworks. Square-planar Pt(II) and linear Au(I) complexes are the mostly employed molecular complexes to make CPL-active aggregates, benefiting from their excellent assembly capability and tunable polymorphic emissions. Relative to solids with disordered molecular arrangement, the formation of supramolecular helical assemblies/co-assemblies and LC phases can amplify the [\(\varepsilon_{\text{lum}}\)] values of chiral emitters by orders of magnitude. CPL-active coordination helicates, polymers, and frameworks have received increasing attention due to their extended structural features and wide potential applications. Among them, Ln(III), Zn(II), Cd(II), and Al(III) metal ions are often used as the coordination nodes, taking into account of their tunable coordination-enhanced emissions. In particular, luminescent MOFs have recently emerged as promising materials for the exploration of CPL activity by host–guest chemistry. The fast development of these materials are believed to further boost the applications of CPL-active aggregates in CP-OLEDs, CPL-contrast imaging, CPL probes, etc.

The research on CPL is believed to attract continuing and growing interest in the coming decade. The focus will be placed on the development of high-performance materials and their explorations in new applications. In terms of coordination aggregates, a number of CPL-active assemblies of Pt(II), Au(I), and Eu(III) complexes have been disclosed in recent years; however, the relationship between the assembly structure and CPL property and the underlying mechanism of chirality transfer and amplification remain to be explored. In order to further improve the CPL parameters, new assembly building blocks, for example, those made of Cu(I), Ag(I), Au(III), and Pd(II) complexes with excellent luminescent properties,\(^{124–126}\) are in demand to conquer the trade-off between [\(\varepsilon_{\text{lum}}\)] and \(\Phi\). In addition, the assembly method or strategy will play a crucial role in determining the performance. CPL-active LCs and conjugated polymers are promising materials for practical optoelectronic applications with the view of their high [\(\varepsilon_{\text{lum}}\)] and \(\Phi\).\(^{18,23,24}\) Recent works on metal complex-involved LCs, polymers, and MOFs have clearly demonstrated their advantages in the amplification of [\(\varepsilon_{\text{lum}}\)], which will further stimulate the design and synthesis of these aggregated materials.\(^{71,81,79,102}\) In addition to the direct self-assembly into mesophase and coordination assembly, chiral doping in LCs or helical polymers is another effective method to explore efficient CPL materials, and more attention needs to be paid in this aspect.

One appealing application of CPL-active coordination materials is CP-OLEDs; however, it still remains a challenge to realize good device performance with high \(\varepsilon_{\text{EL}}\) factors. This needs to be tackled from both the material and device structure optimization. As more and more chiral crystalline MOFs with excellent and tunable CPLs have been prepared,
their potential applications in chiral recognition and separation, asymmetric catalysis, and nonlinear optics are expected to be studied. In addition, nano- and microcrystals are another type of important functional materials in photonic and electronic fields.\textsuperscript{127,128} The constructions of CPL-active nano/microcrystals from chiral metal complexes will be attractive for applications such as circular polarization organic light-emitting transistors, CP light detectors, and so on.\textsuperscript{129–131} We believe that coordination aggregates will be promising for the development of high-performance CPL-active materials and related applications.

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**CONFLICT OF INTEREST**

The authors declare that there is no conflict of interest.

**ORCID**

Zhong-Liang Gong https://orcid.org/0000-0002-4713-5071

Yu-Wu Zhong https://orcid.org/0000-0003-0712-0374

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Zhong-Qiu Li received her BS degree from Nankai University in 2017. She is currently a PhD candidate at ICCAS, under the supervision of Prof. Yu-Wu Zhong. Her research interests focus on emissive molecular materials.

Yu-Wu Zhong obtained his PhD degree in 2004 from Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences. After working as a postdoctoral associate at the University of Tokyo (2004–2006) and Cornell University (2006–2009), respectively, he started his independent career from 2009 and worked to present as a full professor at ICCAS. He received the National Science Fund for Distinguished Young Scholars in 2019 and his research interests focus on the synthesis, assembly, and applications of photo- and electro-functional molecular materials.

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