Aggregation induced emission of chalcones

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Abstract
There is always a need for efficient luminescent materials with simple synthesis and possible ease of hydrogen atom or functional group manipulation for use in different optoelectronic and biological applications. However, for certain real-world uses aggregation caused quenching effect of luminophores in their solid/aggregate state is undesirable, and is a cause of concern in areas, wherein the solid-state optical performance is more crucial. In this regard, chalcones have been explored for their ability to display aggregation-induced emission (AIE) or aggregation-induced enhanced emission (AIEE), which can be of practical use. This article is thus focused on an integrated evidence-based report on the AIE/AIEE-active chalcone systems for potential technological and biological applications.

Graphic abstract

Keywords Aggregation-induced emission · Biological · Chalcones · Fluorescence · Opto-electronic

Introduction
Design of luminescent materials that exhibit solid-state emission is of prime research interest due to their potential photonics, solid-state lasing, optoelectronics, telecommunications, bioimaging, chemical sensing, electronics and information display technology applications (Albrecht et al. 2017; Anthony 2006; Geng et al. 2012; Kim, Inada et al. 2017b; Kim, Sandanayaka et al. 2017a; Li et al. 2017; Sandanayaka et al. 2017). Organic luminophores can display either (i) aggregation caused quenching (ACQ), wherein typical fluorophores emit intensely in dilute solutions, but do not show fluorescence or are weakly fluorescent in their solid state, or (ii) aggregation-induced emission (AIE), where the fluorophores emit effectively in the condensed state, but their solutions have poor or quenched emissions (Luo et al. 2001). However, the intermolecular interactions in organic planar small molecules and the \( \pi-\pi \) stacking effect of aromatic rings, which “turns off” the emission of ACQ...
molecules in solid state is a limiting factor for many practical applications (Prajapati and Surati 2019). Nevertheless, the presence of aromatic rings, bulkier substituent groups or quaternary centers and hydrogen bonding can hamper π-π cofacial stacking to avoid ACQ, specifically in H-aggregates that are highly emissive (Espitia Cogollo et al. 2020). Conformational planarization mechanisms and restriction in intramolecular rotation (RIR) and vibration (RIV) have significantly contributed to the development of highly efficient luminophores with AIE and AIEE features, which can be exploited for various applications (Hong 2009a, b; Islam et al. 2019). The design and synthesis of π-conjugated organic luminescent small molecules with inherent AIE or AIEE property have progressed as a dynamic realm in materials science research (Mei et al. 2015). Several mechanisms were proposed to understand the AIE phenomenon such as conformational planarization, H/J-aggregation, RIR, RIV, excimer formation (Cui et al. 2020), non-covalent intermolecular interactions (Leduskrasts et al. 2019), hindrance to intramolecular charge transfer (ICT), twisted intramolecular charge transfer (TICT), and inhibition of photo-cyclization or photo-isomerisation (Gao et al. 2010; Aldred et al. 2012). RIR is established to be the vital parameter among all for the fascinating and highly useful AIE/AIE behavior (Li et al. 2007; Zhao et al. 2010). In this mini-review, general synthetic method and basic structural framework of chalcones are explained. Further, the well-utilization of AIE/AIEE property of various chalcone derivatives for different applications is illustrated. The current limitations and future scope of AIE-active chalcones are also discussed.

**Structure and synthesis of chalcones**

Chalcones are a group of organic small molecules, abundant in plants that possess various medicinal features (Mah 2020; Schnekenburger and Diederich 2015). Structurally, they are α, β-unsaturated ketones (aromatic ketone and enone) with substituted aromatic rings. The presence of conjugated double bond and complete electron delocalization on phenyl rings attached to ketoethylenic group (–CO–CH=CH–) make their molecular framework attractive and permit desired structural modifications (Awasthi et al. 2009; Yeragunta et al. 2013). Though they can exist as geometric isomers of trans (E) and cis (Z) forms, from the thermodynamic perspective the trans conformation is more stable and predominant, coupled with the strong steric hindrances between the aromatic ring in the Z-isomer and the carbonyl group (Evranos and Ertan 2011; Peng et al. 2017). The replaceable hydrogen in chalcones permits the synthesis of numerous derivatives and endows them with diverse and favorable biological activities (Aoki et al. 2008; Birari et al. 2011; Chen et al. 1993; Cho et al. 2011; Israf et al. 2007; Kim et al. 2014; Yamamoto et al. 2004). Moreover, quite a few material applications of these versatile molecules such as optical sensors, solar cells, nonlinear optics, liquid crystals have also been reported (Ekbote et al. 2017; He et al. 2019; Korkmaz et al. 2018; Schnekenburger and Diederich 2015).

Chalcones have a basic chemistry that provides easier synthesis with a variety of substitutions. Though there are many methods to prepare chalcones, the most commonly adopted process is the Claisen Schmidt condensation in which benzaldehyde and acetophenone derivatives in a liquid solvent reacts with basic or acid catalysts at room temperature or around 50–100 °C (Sachdeva and Milton 2020). A one pot synthetic process with acetonaphone and phenylmethanol mixture in the presence of CrO3 as the oxidizing agent, solid acid catalyst mediated reaction, Sonogashira isomerization coupling reaction, reaction between phenylacetylene and benzaldehyde in the presence of HBr and ionic liquids such as 1-buty1-3-methyl-1H-imidazolium 4-methylbenzenesulfonate (BmimOTs), Suzuki–Miyaura coupling, continuous-flow deuteration and carbynylative Heck coupling reaction (Wu et al. 2010a, b; Rueping et al. 2011) are the various other methods to synthesize chalcones.

**AIE/AIEE properties of chalcones**

Tang and coworkers have shown that fluorophores capable of dual-state emission can not only retain substantial rigidity with minimal intramolecular movements to emit in solution phase, but also own significant twisted conformations to give AIE/AIEE in the aggregated/condensed state (Chen et al. 2015). Some of the chalcones exhibit AIE property and have attracted material researchers due to their potential to be used in versatile applications. They show unusual redox, optoelectronic and luminescent properties attributed to a push–pull conjugation effect, while the α, β-unsaturated ketone spacer together with the angle strain in the keto group augments the non-linearity (Aléo et al. 2015; Karuppusamy et al. 2017). Moreover, their molecular structure contains an electron donor and an electron-deficient keto group coupled with vinyl as an electron acceptor. The Donor-π-Acceptor (D-π-A) conformation accounts for the origin of fascinating AIE/AIE feature to enhance their solid-state fluorescence. Besides, the enone group serves as an electron acceptor and offers the chalcone system an extended π-conjugation that enables long-wavelength emission with broad stokes shifts (Shkir et al. 2015). This red emission that arises due to the lower HOMO–LUMO band gap is highly significant for the fabrication of photovoltaic devices. Moreover, these large emissions and stokes shift values are of prime significance in bioimaging applications and fluorescence microscopy, as they reduce the interference produced by self-absorption...
or auto-fluorescence and aid in improved image sensitivity (Gao et al. 2017).

**Optoelectronic applications of AIE-active chalcones**

The captivating AIE phenomenon has fascinated many material chemists and significant investigations have been performed to explore various avenues in using different chalcone derivatives as functional luminogens for high-tech applications. Some fluorescent chalcones have revealed their potential use in several thrust areas such as optoelectronic devices, chemosensing, and various biological fields. The following section will highlight chalcone-based AIE/AIEE active fluorophores in optoelectronic applications.

Chalcones 1a–e and 2a–e adorned with electron-donating (OMe and NMe₂) and halogen (Cl and Br) atoms were synthesized (Vaz et al. 2016); substituted benzaldehydes and p-methoxyacetophenone were first halogenated with N-bromosuccinimide (NBS) or N-chlorosuccinimide (NCS) in acetonitrile, and subsequently, aldol condensation was carried out to obtain halochalcones in good yields (63–85%) (Fig. 1a). The halogen functionalization coupled with the carbonyl group and the D-π-A-π-D push–pull-push system promoted the fluorescence in the prepared chalcones. The presence of a stronger electron-donating dimethylamino (NMe₂) functionality compared to OMe increased the ICT and maximized the push–pull feature of the chalcones and made them more emissive with a better quantum yield. The chalcone dimers were held through C–H–O, C–H–π, C–H–Cl and C–H–Br hydrogen bonds and some close contact between the aromatic rings. Thus, the presence of halogen atoms that efficiently blocked the torsional vibrations of the enone moiety and the benzene units, together with the extended π–π interactions enabled the synthesized chalcones AIEE active with higher solid-state emission intensity.

D-π-A architecture based chalcones 3a–d, wherein π-conjugated chalcone unit served as an electron acceptor and phenothiazine moiety acted as an electron donor were synthesized using Claisen-Schmidt condensation reaction (Sachdeva and Milton 2020). The molecular arrangements and photophysical properties of these chalcones were tuned by the introduction of various aryl units having different steric and electronic features substituted onto phenothiazine unit (Fig. 1b). Among the four synthesized chalcones, sterically hindered 3c displayed the highest observed AIEE characteristics with large stokes shift because the bulkier naphthyl ring resulted in a twisted structure and restricted the intramolecular rotation, thereby reducing the excited state energy dissipation through non-radiative relaxation modes. Moreover, the bulky naphthyl unit favored loose molecular packing, which consequently increased the generation of light-emitting species in the aggregated state. However, the free intramolecular rotation allowed in the solution phase led to excited state relaxation through non-radiative channel, which resulted in weak fluorescence intensity. The restricted rotation in the aggregate/solid state was thus responsible for the AIEE behavior, which could be tapped for optoelectronic device fabrication and bioimaging applications.

**Fig. 1** a AIEE in halochalcones with electron-donating substituents, (Vaz et al. 2016) b AIEE active D-π-A phenothiazine chalcones as red-emitters (Sachdeva and Milton 2020) and c AIE of light-sensitive 4-dimethylamino-2′-hydroxychalcones (Mortika et al. 2020)
Chloro and bromo derivatives of 4-dimethylamino-2′-hydroxychalcones 4a–c and 5a–c were synthesized using base-catalyzed Claisen-Schmidt condensation (Morika et al. 2020), which displayed AIE in the red region of the visible spectrum. Photo-induced intramolecular cyclisation of these molecules in aprotic solvents led to the generation of respective 4′-dimethylaminoflavonone derivatives with more than 90% conversion yield (Fig. 1c). Fascinatingly, this photo-induced conversion was effectively inhibited by aggregate formation or addition of protic solvents. AIE feature increases photostability of chalcones by blocking the ESIPT process. Hence, the use of these chalcones in optoelectronic devices via solution processing techniques is of special significance.

Efficient red to Near Infrared (NIR) emission was perceived from push–pull structure-based chalcones with an electron donor attached to a strong electron accepting borondifluoride complexes 6a–d with acetylnaphtholate or acetylphenolate units (D’Aléo et al. 2017). Claisen-Schmidt coupling between 2-hydroxy acetophenone and methoxy benzaldehyde generated the respective chalcones, which were further reacted with borontrifluoride etherate in dichloro methane to obtain the complexes with D-A molecular architecture (Fig. 2). The complexes with methoxy groups placed at different positions on the phenyl ring displayed tight supramolecular packing arrangements, and experienced robust excitonic coupling. Complex 6c exhibited a considerably red-shifted solid-phase emission, with a maximum at 784 nm, a Stock shift of >10,000 cm⁻¹ and a quantum yield value of 7%. The NIR emission and AIEE stemmed from the combined effects of tight head-to-tail molecular packing with extended intermolecular π–π overlap and chromophore planarization on the radiative deactivation mode of the excited state in the condensed phase.

Later halogenated borondifluoride complexes 7a–j of 2′-hydroxychalcones (Fig. 2) that incorporated a dihalogeno-substituted acetylphenolate moiety and an anisole unit as electron donor were synthesized (D’Aléo et al. 2019). These halogenated chalcones were prepared via a Claisen–Schmidt reaction between appropriate acetophenone derivatives and p-anisaldehyde and the complexes were obtained by reacting these chalcones with borontrifluoride etherate. The presence of halogens affected aggregation-induced NIR emission of both homo and hetero substituted chalcones, wherein the two halogen atoms are placed at ortho and para position of the 2′-hydroxyl group. The halogens played a crucial role in the crystalline arrangement and the intermolecular halogen–halogen bonds that could further reduce the rotational degrees of freedom in the condensed state did not occur in these compounds. The homo halogenated compounds demonstrated almost a planar π-conjugated system in their crystals, and exhibited intense far red and NIR emissions with higher quantum yield up to 24%. All complexes presented more than tenfold fluorescence emission intensity enhancement and the steric effect associated with bulkier halogens was responsible for J-aggregate formation. It is quite interesting to note that though heavy atoms trigger efficient spin orbit coupling and non-radiatively depopulate excited singlet species (Chen et al. 2020), heavier halogens like Br and I did not quench the emission of these molecules. Instead, there was enhancement in the emission intensity in 7f and 7g with Cl and Br atoms. Moreover, the solid-state fluorescence was dependent not only on the type of the halogen atom, but also on its location in the molecular framework.

Chalcones 8a–c, which can be used as organic semiconducting materials were synthesized by Claisen-Schmidt condensation (Fig. 3a) between planar 1-pyrene aldehyde having an extensively π-electron delocalized skeleton and respective ketones (Karuppusamy et al. 2017). Their
molecular framework incorporated electron-deficient keto vinyl acceptor spaced between the two aromatic donor units on either end, which introduced RIR and AIEE feature that resulted in solid-state luminescence. Though all three compounds possessed D-π-A molecular structure, 8a showed maximum fluorescence intensity due to π-π stacking arrangement and J-aggregation in condensed phase. Low fluorescence quantum yield for 8b (0.68) and 8c (0.53) was attributed to push–pull effect in these chalcones, which changed ICT and hydrogen bonding effectively.

Organic nonlinear optic (NLO) materials with fast quadratic and cubic NLO responses, high non-linearity and a good optical threshold particularly with delocalized π electrons were extensively studied because of their wide application (Boyd 2007; D’Silva et al. 2011; Wu et al. 2010a, b). Chalcones of coumarin derivatives (9a–f and 10a–c) were prepared by refluxing 3-acetyl-2H-1-naphtho[2,1-b]pyran-2-one with corresponding aldehydes in presence of piperidine (Sun et al. 2012). These chalcones exhibited AIE and second-order nonlinear optical features and displayed green to red-tuned solid-state emission (Fig. 3b), on introducing various functional groups into the benzo-coumarin unit. The nature of the substituents significantly influenced the luminescence properties. Chalcone 9b exhibited red-shifted enhancement in fluorescence intensity due to aggregate formation. These compounds can find use in nonlinear optics, solid-state lighting technologies and optoelectronic devices.

Biological applications of AIE-active chalcones

Taking benefit of the unique property that AIE-active molecules are non/less emissive in their solution, but display intense fluorescence in their solid or aggregate forms, a wide variety of AIE biomarkers for sensitive detection of different biomolecules such as biothiols, proteins, alkaline phosphatase (ALP), and minerals in biological samples have been reported (Chen et al. 2013; Ding et al. 2013; Hong et al. 2011; Hong et al. 2009a, b; Jiang et al. 2017). 2’-Hydroxychalcone and its derivatives display excited state intermolecular proton transfer (ESIPT) (Teshima et al. 2009) via enol to keto forms to produce a well-conjugated molecular skeleton with alternate single and double bonds. Chalcone-based AIE fluorophores feature dual fluorescence in solution and aggregated/solid states with sufficiently differing wavelengths and the conventional “off–on” fluorescence can be transformed with ease into a ratiometric response. Visible emission color, lower background noise, higher brightness, enhanced photostability, analyte-triggered ratiometric fluorescence, high sensitivity, selectivity, less complicated sensing mechanism and less cytotoxicity are advantageous for fluorescent imaging in live cells. In addition, paper-based microfluidic analytical devices that incorporate the AIEgens and associated detection units into a low-cost paper platform can enable an easily accessible testing (Qian et al. 2008).
An efficient chalcone-based fluorescent probe 12 was fabricated for ratiometric detection of biothiols (Dai et al. 2020). The 2′-hydroxychalcone 11 fluorophore prepared using Claisen-Schmidt condensation between 4-dimethylamino benzaldehyde and 2′-hydroxyacetophenone was appended with a 2,4-dinitrobenzenesulfonyl (DNBS) unit that acted as a biothiol-responsive moiety (Fig. 4). The fluorescence of chalcone probe 12 was quenched through photo-induced electron transfer (PET) effect as well as due to hindrance of ESIPT property, when hydroxyl group was caged by DNBS. But the fluorescence in the red region was turned on when ESIPT and AIE phenomenon was recovered, as chalcone 11 was formed when the biothiols reacted with probe 12. The N, N-dimethylamino unit served as electron donor and 2′-hydroxyacetophenone unit as electron acceptor in fluorophore 11, which were engaged in ICT process. The limit of detection (LOD) was found to be 0.227, 0.283 and 1.826 μM for biothiols such as Glutathione, Cysteine and Homocysteine respectively. The biocompatible probe 12 was selective toward biothiols even in the presence of other biological species such as amino acids, metal ions and Bovine Serum Albumin. The ESIPT coupled AIE features with large Stokes’s shift greater than 140 nm enabled the remarkable fluorescence intensity enhancement (51–94 fold) with a fast response time of 3 to 15 min, and hence the feasible detection of biothiols in the condensed-state on indicator papers.

A ratiometric AIE-active fluorescent phosphorylated chalcone-based probe 14 was prepared (Song et al. 2014) to detect ALP, as its activity is recognized as a biomarker in medicine, and its abundance in blood can reflect diseases like prostatic cancer, bone tumor, osteoporosis and hepatitis. The chalcone 13 was synthesized through phosphorylation of 11 with diethyl chlorophosphate in presence of sodium hydride, and eventually deprotected using iodotrimethylsilane (TMS-I) to obtain probe 14. The phosphate group of 14 served as the recognition site, which was cleaved in presence of ALP to form chalcone 11 on further protonation. In solution form, intramolecular hydrogen bonding of 14 suppressed ESIPT to provide greenish-yellow fluorescence. However, in the presence of ALP, the poorly water-soluble enzymatic chalcone product 11 crystallized in aqueous media forming intramolecular hydrogen bond, and thereby activated the AIE and ESIPT processes turning on the red fluorescence. The head-to-head and edge-to-face crystal packing restricted the π-π interactions of 11, whereas strong hydrogen bonding with 1.75 Å distance facilitated ESIPT in aggregate keto form to emit red fluorescence intensively. This ratiometric bio-probe thus enables ALP assay to be performed in the concentration range of 0 - 150 mU/mL with an LOD of 0.15 mU/mL. The red emission prevented the auto-fluorescence interference from serum samples, and can also be used to detect ALP in intracellular fluids and visualization of the respective live cells as presented in Fig. 4.

2′-Hydroxychalcone derivatives that own D-A skeletons display ESIPT-activated intense solid-phase emission in the red or NIR region upon excitation, while in protic solvents, they emit at a much shorter wavelength (Zhang et al. 2015). A dual-state emissive 2′-hydroxychalcone 15 with AIE property was prepared by Claisen–Schmidt condensation to monitor human serum albumin (HSA) (Luo et al. 2020).

Fig. 4 ESIPT coupled AIE fluorescent paper-based probe for detection and imaging of biothiols (Dai et al. 2020) and ESIPT and AIE based ratiometric fluorescence probe for ALP activity assay and visualization in live cells (Song et al. 2014)
methoxyl group at the para position of the keto group led to edge-to-face packing to lessen the \(\pi-\pi\) stacking and produced red fluorescence in solid/aggregated form. The chalcone presented a gradual change in the fluorescence emission from red to yellow and finally green after addition of HSA from 0 to 9 \(\mu\)M with a LOD of 16.4 nM. The fluorophore 15 was highly selective to HSA in presence of other proteins, polypeptides and amino acids, which might co-exist with HSA in the serum. HSA provided a hydrophobic environment for 15, induced disassembly into its monomeric form and engaged in non-covalent hydrogen bonded interactions to limit the free molecular rotation of the chalconic probe (Fig. 5a). The paper-based analytical device centered on nanoaggregates of 15 not only allowed accurate determination of HSA and diagnosis of hypoalbuminemia in blood, but also offered a simple and compatible instrument-free testing of HSA.

The presence of tetraphenylethene moiety in structural framework of organic molecules typically leads to AIE due to hindrance to \(\pi-\pi\) stacking and RIR and RIV. Hence four tetraphenylene-based chalcone hybrid derivatives (16a–d) as presented in Fig. 5b were synthesized (Chen et al. 2020). Lower quantum yields (0.006–0.12) in these compounds in different organic solvents were due to the rotation and vibration of tetraphenylethene and benzothiazole derivatives. The molecules showed AIE properties with solid state quantum yields ranging from 3.71 to 10.7% due to their push–pull design incorporating D-\(\pi\)-A structural framework. The electron-donating ability of the triphenylamine unit and N,N-diethyl quinoline unit significantly induced enhancement of ICT process. The molecules adopted twisted conformation and a head-to-tail slipped stacking arrangement. Chalcone 16c exhibited maximum solid state fluorescence due to the non-coplanar triphenylamine unit that resulted in increased \(\pi-\pi\) distances and weakened intermolecular interaction. The incubation of B-16 cells with these chalcone derivatives suggested their potential utility in live cell imaging.

**Conclusion and future prospects**

Chalcones with D-\(\pi\)-A architecture, which can be prepared with easy synthetic protocols display AIE and AIEE features which could be suitably used in various solid-state devices. The chalcone derivatives undergo excited state fluorescence quenching through internal conversion in solution. But their tight molecular packing and the various interactions and restrictions to molecular movements in terms of rotation and vibration can result in red-shifted or enhanced emission intensity in solid state, when compared to their solution form. The modifications of the donor and acceptor units with not only different substituents, but also altering the position of the substituents can facilitate diverse packing arrangements in their condensed state, which can trigger AIE/AIEE. This review is useful in designing and synthesizing novel AIE/AIEE-active chalcones for applications in various unexplored technological sectors.

Though solvent-induced aggregation has been demonstrated with suitable evidences, new methods to trigger the aggregation of AIE-active fluorophores are, however, rarely understood and further research in this path is still required. Even though there are huge developments observed in designing different types of complexes, AIE-active chalcone complexes can be further explored for various applications. Besides, the molecular design strategy to construct highly sensitive ratiometric fluorophores with red fluorescence emission in aggregated state utilizing natural products is

![Fig. 5](image_url)
yet another area that needs more investigations. Moreover, biocompatible materials with aggregation-induced emission that display ratiometric fluorescence signals with high selectivity and sensitivity are keenly sought after as potential bioimaging and bio probes. It is highly demanding to explore AIE/AIEE active chalcone derivatives that facilitate significant applications in the area of opto-electronics and life science.

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Declarations

Conflict of interest The authors declare that they have no known competing financial interests.

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