Mechanism and application of aggregation induced luminescence compounds

Zhongan Xu¹*

¹High School Affiliated to Shanghai Jiaotong University, Shanghai, China
*Corresponding author’s e-mail: jane_xu@smicschool.com

Abstract. Organic Light-Emitting Diode (OLED) technology has gained wide acceptance in recent years. The popularity of OLED is attributed to the superior properties of organic luminescent materials such as low biotoxicity, outstanding optical properties and easy preparation and modification. Traditional organic luminescent materials usually suffer from aggregation-caused quenching (ACQ) effect and can be hardly utilized in aqueous or solid conditions, while the compounds exhibiting aggregation-induced emission (AIE) property can overcome this limitation. Thus, AIE luminogens have attracted wide attention in the past ten years. Based on the research on the mechanism, classification and application of AIE compounds, a novel aggregation induced luminescence compound based on triphenylamine structure was synthesized and characterized. Our work suggested and confirmed that the synthesized compound showed apparent aggregation-induced emission (AIE) property.

1. Introduction
Light is an extremely important physical phenomena that has found wide applications in our daily life. In the past few decades, research and application of organic luminescent materials has been one of the primary areas of focus for the scientists. Compared to inorganic luminescent materials, organic luminescent materials have found wide application in various fields such as luminescent coatings, analytical detection, display devices and medical labeling imaging. This is primarily due to the advantages associated with their high color purity, wide variety, low price, lower biological toxicity and simplified and easier approach involved in preparation, control, and functional modification.

The performance of organic luminescent materials is completely different in the state of dispersion and aggregation. In the past, the luminescence mechanism of traditional organic luminescent materials was usually carried out in very dilute solution, and their optical properties in solution state were also better. However, once these organic luminescent materials form highly concentrated solution or in solid state, their fluorescence is reported to be partially or completely quenched, referred to as the aggregation induced quenching (ACQ) phenomenon (Fig. 1). This behavior is attributed to their structure; the traditional organic light-emitting materials generally have planar structures. However, strong π-π stacking is formed between adjacent organic conjugated molecules in agglomerated state. This results in the emission of non radiating energy in the form of thermal motion and collision of organic molecules in excited state, leading to the quenching phenomenon [2]. This characteristic of luminescence in dispersed state and quenching in aggregation state greatly limits the application of organic luminescent materials in aqueous and solid conditions. For example, if the traditional ACQ organic fluorescent probe is added for labeling a specific human cell or organelle, it will inevitably spontaneously gather and quench, thus affecting the labeling. This is because most of the organic matter is hydrophobic and the internal and external environment of human cells is mostly aqueous.
Many scientists have tried to avoid the ACQ phenomenon by introducing hydrophilic groups or large steric groups, but the results are mostly unsatisfactory [3].

However, the aggregation-induced emission (AIE) phenomenon discovered by Tang Benzhong's research group in 2001 was found to successfully break through the above bottleneck [4]. The team unexpectedly found that although hexaphenylsilole (HPS) hardly emits light in dilute solution, a significantly enhanced fluorescence can be observed when hexaphenylsilole is precipitated in an aggregation state either by the addition of water or volatilization of the solvent (Figure 2). The AIE phenomenon, which does not emit light in the state of dispersion but emits light in the state of aggregation, is in complete contrast to the ACQ phenomenon mentioned above, thus overcoming its limitation. This expands the application range of organic luminescent materials and attracts extensive attention from the scientific community.

After more than ten years of development, a large number of AIE compounds have been synthesized, characterized, studied and explored in various applications. This data has helped to overcome the limitations and helped to further enrich and improve the related theories. This paper presents a review of the mechanism, classification and application of AIE compounds. The authors have discussed some recently published representative achievements with an endeavor to have a deeper understanding of this phenomena so as to encourage further development and application in various fields.
2. AIE correlation mechanism
In order to better understand and utilize AIE effect, researchers have proposed many mechanisms that induce or assist in regulation of AIE. Although some of the theories are still controversial or have insufficient data, some proposed theories are worth studying and exploring.

2.1. Restriction of Intramolecular Motion (RIM)
Since the discovery of the AIE phenomenon of HPS in 2001, Tang Benzhong's research group has reported that in contrast to the planar structure of traditional ACQ compounds, the six phenyl compounds of HPS are propeller shaped (Fig. 3), and it is believed that this may be an important factor in causing AIE [4]. Furthermore, in 2003, Tang Benzhong and Zhu Daoben proposed the mechanism of Restriction of Intramolecular Motion (RIM) based on the crystal structure and AIE performance of different substituted 2,3,4,5-tetraphenylthiazole derivatives [5]. This mechanism has been subsequently confirmed by a large number of experiments and theoretical studies, and is also reported the most widely recognized mechanism.

RIM mechanism suggests that after gaining energy, organic luminous molecules have two main channels to release energy in excited state and return to the ground state: either through the thermal motion channel of molecules (including the rotation and vibration of molecules), releasing energy in long waves or through an optical channel, wherein wavelengths of light fall in ultraviolet, visible and near-infrared wavelengths. HPS, in dispersed state (Fig. 4) exhibits no fluorescence. This is due to the steric hindrance effect of adjacent phenyl groups; each phenyl cannot form an effective conjugation system, the time channel is closed, and the activation energy of molecules can only be consumed in the form of thermal motion thus influencing fluorescence. In the aggregation state, however, the adjacent HPS molecules are squeezed and stacked against each other, such that the six phenyl groups form conjugates in a planar structure, thus opening up the light channel and allowing the activation energy of the molecules to be released in the form of fluorescence.
hydroquinone (Fig. 5), resulting in an AIE effect with a higher fluorescence quantum yield than that of testyrene-, which directly and powerfully demonstrated the RIM mechanism.

![Figure 5. intramolecular cyclized derivatives of tetrastyrene](image)

2.2. **Excimer**

The plane structure of ACQ molecule (FIG. 6a) makes it easy to form strong π - π stacking in aggregation state resulting in excitation association complex [7], which further leads to the redshift and quenching of the emitted light [8]. On the contrary, due to steric hindrance and other reasons, some AIE molecules have a non-planar structure in the dispersed state (FIG. 6b), and cannot be conjugated effectively, leading to no fluorescence. However, in an aggregated state, although the steric hindrance prevents it from forming excitation association complexes, the molecules are squeezed and stacked together, making the molecules form conjugated with planar structures, thus generating fluorescence [9].

![Figure 6. Mechanism of excitation association](image)

2.3. **J-aggregate**

In 2002, Park et al reported AIE effect by J-aggregation of cyano substituted distyrene derivatives (FIG. 7) [10]. In the dispersed state, the interaction between cyano group and phenyl group results in a distorted structured compound without fluorescence. In the state of aggregation, the compound tends to be planar due to intermolecular extrusion and stacking, and due to the steric hindrance effect of cyano, the H-aggregation of face-to-face stacking is reported to be effectively suppressed, thus forming the first connected J-aggregation, and the conjugated chain grows, thus producing fluorescence.

![Figure 7. J-aggregated α - cyano substituted stilbene derivatives](image)
2.4. Twisted Intramolecular Charge Transfer (TICT)
There are usually two ways to redshift the fluorescence. One is to increase the length of the conjugate chain on the original conjugated fluorescence system. The molecules prepared by this method generally have a large planar structure and can cause ACQ effect easily. The other method is called intramolecular charge transfer (ICT) effect or D (Donor) -a (Acceptor) effect or push-pull effect and involves introducing electron-pushing and electron-absorbing groups at both ends of the original system. However, if the electron-pushing group in an ICT system is connected to the original fluorescence system through a single bond, the strong intramolecular photoinduced charge transfer makes the electron-pushing group rotate and break the conjugate system on being excited. This inhibits the molecule from producing fluorescence, and is called TICT effect. Tang Benzhong et al reported AIE effect in 2009 based on TICT mechanism of Dipyrrole methylene boron derivative (BODIPY) [11]. This is shown in figure 8.

2.5. Excited State Intramolecular Proton Transfer (ESIPT)
The ESIPT effect often occurs in compounds containing intramolecular hydrogen bonds [12]. ESIPT compounds in an excited state transfer protons by means of intramolecular hydrogen bonds or hydrogen bridges to achieve tautomerism [13]. For example, BTHPB does not emit light in the dispersed state (FIG. 9). The results of spectral analysis proved that due to the free rotation of the single bond between dihydrobenzothiazole and phenol, the energy is consumed in a non-radiative manner. However, in the aggregation state, intramolecular hydrogen bonds are strengthened, and intramolecular hydrogen bonds are formed between dihydrobenzothiazole and phenol. The phenol changes from enol to ketone, the optical channel is opened, and the energy is consumed in the form of radiation, resulting in fluorescence [14].

2.6. Forster Resonance Energy Transfer (FRET)
FRET is an energy transfer between two fluorescent molecules close to each other. If the emission spectrum of the donor fluorescent molecule overlaps the absorption spectrum of another receptor fluorescent molecule, the receptor absorbs the donor’s emission light, excites to the excited state and emits light (sensitized fluorescence). The overall result is that a set of absorption and emission spectra with large Stokes shift are obtained. The fluorescence efficiency of FRET is mainly regulated by three methods: regulating the distance between the donor and the recipient; overlapping degree of donor emission spectrum and receptor absorption spectrum; relative orientation of donor emission dipole moment and receptor absorption dipole moment.
2.7. Through-bond Energy Transfer (TBET)
FRET requires sufficient overlap between the emission spectrum of donor and the absorption spectrum of acceptor, while obtaining a larger Stokes shift requires that the emission spectrum of donor is far away from that of receptor, which is in contradiction to the other process.

Burgess et al. solved this problem with the help of TBET system. In TBET system, the twisted rigid conjugated structure (such as double bond, benzene ring, etc.) connects the donor and the receptor, rather than the flexible aliphatic structure in FRET system. Therefore, the energy between the donor and the receptor can be non radiative and can be transferred through chemical bonds, thus being less dependent on the spectral overlap between the two [15].

2.8. Dark Resonance Energy Transfer (DRET) and Dark Through-bond Energy Transfer (DTBET)
In FRET system, the fluorescence leakage of donor emission light often leads to the decrease of fluorescence efficiency and energy utilization efficiency, and the leakage of fluorescence also limits its biological application. Professor Young-Tae Chang of National University of Singapore and his research team replaced the donors in FRET system with ones with lower quantum yield rate (< 1%), thus achieving a resonant energy transfer system with high fluorescence efficiency and energy utilization, thus forming the DRET system [16]. This concept introduced into the TBET system, is named as the DTBET system [17].

3. Classification of AIE compounds
Since the development of AIE system, a variety of AIE compounds have been synthesized and studied. Some of them have further developed a series of derivatives due to their typicality and superiority. This paper reviews and summarizes several common AIE compounds with a view to have a clear understanding of AIE system.

3.1. Hydrocarbons
When it comes to hydrocarbons, we have to talk about tetraphenylethylene (TPE). TPE and its derivatives have become the most common class of compounds in AIE system because of their stable properties and easy preparation and modification. Literature survey has indicated use of TPE as a parent or modifier in production of new compounds and have reported studies on their properties and applications. In 2016, Misra and others worked on combining TPE with benzothiadiazole to synthesize a new compound with D-A effect [18]. The compound has AIE effect, can change from blue light to green light under mechanical pressure, and has good response to temperature and organic vapor and is expected to be further developed into a multifunctional smart sensing material (Fig. 10).

Figure 10. TPE derivatives with multiple response capabilities
Generally speaking, the hydrocarbon AIE represented by TPE exhibits advantages such as simple structure, stable property and easy preparation and modification. However, AIE effect of single hydrocarbon usually emits blue light, while medical imaging and other applications mostly need AIE with red light property. Extension of the conjugated chain can easily lead to ACQ effect, so hydrocarbons are often decorated with other types of compounds in order to achieve the purpose of modulating its property and achieve multi-functional application.

3.2. Tertiary amine compounds

It is also one of the simple methods to synthesize AIE compounds by linking one or more conjugated systems to nitrogen atoms. A typical example is triphenylamine (TPA) derivative. In the dispersed state, due to the tetrahedral configuration of nitrogen, the three phenyl groups cannot be effectively conjugated and the activation energy is consumed through the intramolecular motion. In the aggregation state, however, the triphenylamine is forced to become a planar structure, and the three benzene groups are effectively conjugated by the lone pair electrons on the nitrogen, leading to the AIE effect. Compared with hydrocarbon AIE compounds, this method of introducing nitrogen atom makes the compounds possess more potential conditions for modification and regulation, such as hydrogen bonding, D-A effect, TICT effect, ESIPT effect, etc., thus greatly enriching the types and application scope of AIE compounds. Zhou Hongping et al reported a detailed study on the structure, multiple interactions and fluorescence properties of TPA type AIE compounds (Fig. 11) [19]. Other AIE compounds containing heteroatoms have similar properties, but have not been discussed in this article.

3.3. Compounds containing hydrogen bonds

The hydrogen bonds of AIE compounds are beneficial to the formation of supramolecules between molecules, and to the initiation of ESIPT effect. At the same time, it is also conducive to the immobilization of molecules and the reduction of intramolecular motion, thus improving the fluorescence quantum yield. Bo Zhishan and others used the dendrite structure formed by hydrogen bond (Fig. 12) to induce AIE effect. This special dendrite structure and hydrogen bond made the compound not only successfully achieve the purpose of improving fluorescence quantum yield, but also made it possible to form sol in some organic solvents[20].

![Figure 11. AIE effect of triphenylamine compounds](image1)

![Figure 12. AIE compounds with dendrite structure formed by hydrogen bonding](image2)
3.4. Organometallic compounds
Several methods have been reported for the preparation of AIE compound form aggregation state, such as adding poor solvent, forming solid, applying pressure, etc. Using metal or metal ion complexation with organic matter is also one of the methods. This method is often used for qualitative and quantitative detection of metal or metal ions. Recently, Goel and others have successfully studied and proved the quantitative detection of Trace Ag+ ions in water by TPEN (Fig. 13), a naphthyridine derivative of TPE. The detection limit was 0.25μmol/L, which was lower than 0.93μmol/L of environmental standard [21]. In addition, TPEN exhibits mechanical discoloration effect and has been used as carbon paper material as well.

![Figure 13. detection of Ag+ ion by AIE organometallic compounds](image)

3.5. Polymer
Compared with the above-mentioned small molecule AIE compounds, polymers are also considered in the AIE system because of their stable properties, high mechanical strength, corrosion resistance and easy processing. However, due to the fact that the polymer itself is a kind of aggregation state, AIE molecules often exhibit aggregation-enhanced emission (AEE) effect after polymerization, that is, the polymer emits weak light in the dispersed state, while the luminous intensity increases significantly in the aggregation state. Recently, Tang Benzhong and others realized the rapid spontaneous polymerization of AIE molecules under mild conditions by using Click reaction of secondary amines and alkynes. The reaction achieved 100% atom economy and could have regional and chiral selectivity. Moreover, it should be noted that the AIE effect rather than the AEE effect was shown in the polymer (Fig. 14)[22].

![Figure 14. AIE polymer prepared by click reaction](image)

3.6. Internal salt
Recently, Tang Benzhong and others reported a new type of AIE compound, TPO-P, an internal salt using anion - π + interaction to achieve AIE effect (Fig. 15) [23]. TPO-P exists in the form of free ions in the solution, and the intramolecular motion consumes the activation energy, so it does not emit light. When it forms an aggregation state, the cation and anion are arranged in order through the anion -π+ interaction to form sandwich structure. The PF6- ion of TPO-P blocks the ACQ effect caused by π-π stacking in aromatic system, but the steric hindrance effect between molecules initiates RIM.
mechanism, which leads to AIE effect. The results show that some ACQ compounds can be transformed into AIE compounds by forming internal salts, and internal salts can be more easily formed into crystals with higher thermal stability and conductivity. It can be predicted that its application potential in the field of electronic devices. In addition, TPO-P has been applied to turn-on and wash free labeling of lysosomes due to its biocompatibility.

4. Application of AIE compounds
AIE compounds, due to their characteristic of emitting light in the state of aggregation but not in the state of dispersion, make up for the shortcomings of ACQ compounds. They are easy to form turn-on mechanism in aqueous phase and solid aggregation conditions, and have higher signal-to-noise ratio. Therefore, AIE compounds are widely used in analysis and detection, medical labeling and display devices. In this paper, several representative applications of AIE compounds in recent two years are listed, especially in analytical detection and medical applications.

4.1. Analysis and detection

4.1.1. Determination of metal ions
The interaction between metal ions and organic compounds has the characteristics of strong force and high specificity, so many AIE compounds have been used to determine metal ions. Recently, Tang Benzhong and others combined this principle with DTBET mechanism to detect Hg$^{2+}$ ions with high toxicity in water [17]. Before adding Hg$^{2+}$, plm-TPE-RNS showed AIE effect and blue light in water. However, after Hg$^{2+}$ was added, due to the DTBET interaction between TPE and Rhodamine, the spiro was opened, and the rhodamine group emitted strong red light (Fig. 16). The detection limit of this method is 0.3 ppb and has a Stokes shift of 280 nm.

4.1.2. Determination of anions
Anions play an important role in life system and ecological environment. For example, anionic surfactants have important applications in life and industry. However, excessive anionic surfactants will pose a great threat to life and environment. Tang
Benzhong and others designed an AIE compound HBT-C18, which can specifically combine with anionic surfactant SDBS to form cationic aggregates and emit light (Fig. 17) [24]. The emission intensity of the aggregates was positively correlated with the ratio of keto/enol tautomerism induced by ESIPT mechanism. Therefore, SDBS can be quantitatively detected with the detection limit of 0.051 μmol/L, which is lower than that of the traditional ethylene blue colorimetry (0.144 μmol/L).

![Figure 17. detection of anionic surfactant SDBS by AIE compound HBT-C18](image)

### 4.1.3. Detection of small molecules

In recent two years, environmental problems have become the focus of the state and society. The effective technologies for the determination of organic or inorganic small molecules such as carbon dioxide, nitrogen oxides and formaldehyde in the environment need to be developed. Tang Benzhong and others designed a nanosheet for the detection of volatile organic compounds (VOC) in the environment [25]. The nanosheets are self-aggregated by cyclodextrin modified TPE and other compounds. Due to RIM mechanism, the nanosheets can emit strong fluorescence. When the nanosheets encounter VOCs, the TPE derivatives are dissolved and dispersed, resulting in fluorescence quenching (Fig. 18). The detection limit of this method is up to 5 μg/L, but unfortunately, the method experiences turn-off mechanism.

![Figure 18. detection of volatile organic compounds (VOC) by TPE derivatives](image)

### 4.1.4. Biological monitoring

The operation process of life is so complex that human beings know little about the truth of life since the development of science and technology. Biomolecules or units, such as sugar, protein, RNA, DNA, organelles and cells, are the cornerstones of building life. Monitoring their content, distribution and movement is an important way for scientists to understand life. Because of its non-invasive, visible, real-time, good biocompatibility and other characteristics, fluorescent probes have become an ideal tool. Traditional fluorescent probes can not be well applied to the water environment of life because of ACQ effect, and AIE compounds just make up for this. Telomerase is one of the indicators for monitoring cancer cells. In 2015, Tang Benzhong and others developed a real-time, quantitative, in-situ and rapid detection method of telomerase in urine of patients with bladder cancer [26]. The amphoteric TPE derivative TPE-Z with positive charge does not emit light when dissolved in urine, and it can combine with the negative cytoskeleton of DNA. After adding short DNA fragments to urine, TPE-Z emits weak light. If telomerase is present in urine, these short DNA fragments are catalysed to connect and aggregate long DNA fragments. At this time, RIM mechanism of TPE-Z starts and emits intense light (Fig. 19). The detection time of this method is only 1 minute, and the sensitivity is up to 10 cells. It does not need complicated pretreatment and expensive instruments. It has a very good application prospect in clinical diagnosis.
Similarly, prostate specific antigen (PSA) is one of the diagnostic indicators of prostate cancer. Qu Fengli and others developed a non-standard quantitative method for the detection of PSA [27]. The functional amination of SiO2 makes it have a positive charge, and a layer of PSA aptamer is coated by electrostatic interaction. If there is PSA, it can specifically combine with PSA aptamer, so that the amino SiO2 with positive point is exposed and bound with BSPOTPE, which is negatively charged AIE compound, thus triggering AIE effect and emitting light (Fig. 20). The detection limit was 0.5 ng/mL.

4.1.5. Micro environment monitoring

The detection of physical and chemical parameters in microenvironment is of great significance. In particular, factors such as temperature, viscosity, pressure and pH in the biological microenvironment of the cytoplasm also play an important role in life. But the conventional detection methods are difficult to achieve effective real-time monitoring in such a small environment. Lin Quan and others synthesized d-TPE, a derivative of TPE, which was immobilized on the surface of nanoparticles and emitted fluorescence response to changes in pH and temperature respectively (Fig. 21). The detection range of pH is 6.5-7.6, and the detection range of temperature is 10-80 °C. It basically meets the detection needs of living organisms, and finds good prospects in biological and medical applications[28].
Figure 21. bifunctional AIE fluorescent probe capable of simultaneously monitoring pH and temperature

4.2. Medical marker imaging

Medical marker imaging is an important method in clinical diagnosis besides index test. In the past, imaging technology was often associated with limitations of real-time imaging, long operation time, high price and radiation damage to human body. However, fluorescent labeling imaging has attracted great interest in the field of imaging technology due to its non-invasive, controllable and multi-functional real-time characteristics. AIE compounds also stand out from many fluorescent probes due to their good performance in aqueous phase.

Kim and others prepared AIE conjugated polymer in the form of sol in water by in-situ one pot method [29]. The size of the sol formed by the polymer is reported to be very small whereas its near infrared fluorescence signal-to-noise ratio is high, thus making it suitable for medical imaging. The polymer was injected into mice to bind to specific subcutaneous lymph nodes. When irradiated with a strong 650 nm laser, fluorescent spots labeled with subcutaneous lymph nodes could be directly observed from outside (Fig. 22). This method realizes real-time controllable visual imaging in vivo, which is a typical example of AIE fluorescent probe in medical imaging application.

Figure 22. accurate labeling of fluorescent spots in subcutaneous lymph nodes of experimental mice

4.3. Organic luminescent materials

Organic light-emitting materials are easy to prepare, cheap, easy to control, exhibit high energy utilization and so on, and are thus considered as ideal luminescent materials. However, the solid conditions of AIE compounds provide opportunities for their application. Based on the blue AIE compound, Tang Benzhong and others developed three kinds of high-performance undoped mixed white organic light-emitting diodes (OLEDs), one with a two-color white light, and the other two with three-color white light [30]. The three kinds of organic light-emitting diodes have the common characteristics of good flexibility, high brightness and high energy utilization (Fig. 23). By adjusting the composition and structure of the three kinds of organic light-emitting diodes, they can offer different characteristics, such as color gamut similar to sunlight, different thickness etc.
5. Summary and Prospect

Organic light emitting materials (OLEDs) have been widely studied and find wide applications due to their easy preparation and processing, easy functional modification, low biological toxicity and low energy consumption. However, most of the traditional organic light-emitting materials are often posed with limitations of ACQ effect and cannot be well used in aqueous phase and solid state. AIE compounds are a class of compounds that do not emit light in dispersed state but in aggregation state, and can help to overcome the limitations. In addition, they have high signal-to-noise ratio and are widely used in the field of analytical detection and medical labeling imaging, and many exciting results have been obtained.

Looking at the development of AIE system in the past few years, we can find the following development trends: to make the emission light develop towards the direction of long wave, high brightness and high efficiency; from single electron excitation to multi electron excitation; from absorption emission spectrum to high Stokes shift direction; from fluorescence to phosphorescence; to make AIE compound develop towards multi-function direction; combination of AIE system with other systems, such as CT, MRI, etc.

In the foreseeable future, the monitoring or imaging methods based on AIE system can be explored further to solve the following problems: high sensitivity and high selectivity; anti-interference in complex biological environment; reproducibility and commercialization; biological compatibility.

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