



INTRAMOLECULAR EXCIMERS OF ORGANIC COMPOUNDS

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Cite this: *INEOS OPEN*, 2024, 7 (4–5), 117–131
DOI: 10.32931/io2448a

Received 8 June 2024,
Accepted 6 August 2024

<http://ineosopen.org>

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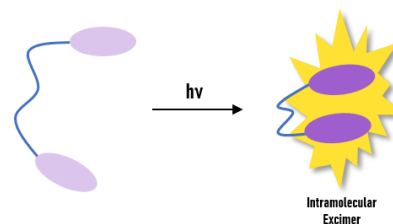
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Abstract

Nowadays, the compounds with unique fluorescence and sensor properties attract much attention of researchers. One of the promising classes of compounds are organic fluorophores capable of forming excimers in an excited state. This review highlights intramolecular excimers based on organic compounds, their properties and applications, as well as matrices that are used to graft such fluorophores.

Key words: excimer, fluorophore, fluorescence.



Introduction

Excimers are dimers formed by the interaction of molecules (monomers) one of which is in an excited state. A distinctive feature of excimers is their dynamic nature and the absence of interaction between molecules in the ground state. The most well-studied excimers are those of aromatic compounds, such as benzene, naphthalene, anthracene, pyrene, perylene, and their derivatives. A characteristic feature of excimers is the presence of a broad structureless fluorescence band shifted to the long-wavelength region of the spectrum, compared to the fluorescence band of the monomer. The systems with monomer–excimer fluorescence are of great interest in materials science, since they can be used as sensors reacting to changes in various external factors (temperature, medium polarity, the presence of various cations and anions or organic molecules) and as light-emitting layers in organic diodes for modern OLED technologies. Since the photophysical properties of complex aromatic molecules strongly depend on π – π interactions between fragments, the design of molecular structures with a given geometry allows for suppressing or enhancing this type of π – π interactions and thus ensures fine-tuning the properties of the resulting systems. A literature survey revealed quite a large number of studies on intramolecular excimers of different derivatives of aromatic and polyaromatic compounds grafted onto matrices of various structures, some of which will be further discussed in this review.

Excimers

Excimers are excited-state complexes. Excimers are dimers that form when one excited-state molecule interacts with another such molecule in the ground state. If the interaction occurs

between an excited molecule of one type and a ground-state molecule of another type, the resulting excited-state complex is called an exciplex.

These complexes have attracted great research interest from the viewpoint of theoretical and applied chemistry since the 1970s. Molecular excimers are usually formed when there is a small intermolecular distance between two interacting molecules, which facilitates the formation of excimers due to π – π interactions. As a rule, this is achieved in crystals, amorphous films and concentrated solutions of aromatic hydrocarbons, such as benzene, naphthalene, anthracene, pyrene, perylene, and others, as well as their derivatives [1–4].

The term excimer was introduced by B. Stevens and E. Hutton in 1960 [5]. They suggested using this term because an excimer is not a primary absorption product and does not exist in the ground state, and also to distinguish it from short-lived dimers excited directly in solutions and having a concentration-dependent absorption spectrum. Therefore, an excimer was defined as a dimer that is bound in an excited electronic state and dissociates (decomposes) upon transition to the ground electronic state. This definition is suitable not only for intermolecular excimers in solutions, but also for excimers in crystals, polymers, and intramolecular excimers where environmental constraints and/or steric motions of the molecules exist. To include these cases, in 1975 J. B. Birks redefined an excimer as "a dimer that is bound in an excited electronic state and is dissociative (*i.e.*, dissociates in the absence of external constraints) in its ground electronic state." The revised definition includes the objects such as crystalline dimers, sandwich dimers, and intramolecular excimers, which were previously excluded from the original definition by B. Stevens and E. Hutton.

A scheme of the excimer formation in solution can be represented as follows: an excimer is formed from a solvate-separated pair of monomers when an excited molecule interacts

with another such molecule in the ground state, or from a dimeric complex, that is, a dimer in the ground state (Fig. 1).

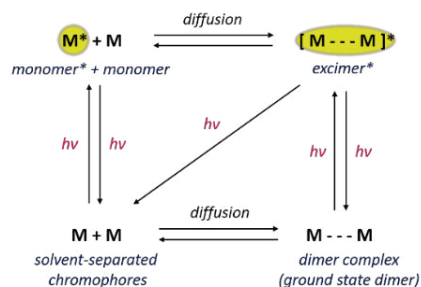


Figure 1. Formation and emission routes of excimers in solution.

The excimer emission has characteristic spectral features and usually provides a broad fluorescence band without vibrational structure, with a maximum shifted in the case of most aromatic molecules towards lower energies (with a longer wavelength) compared to the emission of the fluorophore monomer. The intensity of the excimer emission band relative to the emission intensity of the monomer in solution depends on the fluorophore concentration: the higher the concentration, the greater the proportion of the excimer emission.

There are two types of excimers: static and dynamic. Under normal conditions, the difference between static and dynamic excimers is that in a time-resolved experiment one can observe an increase in the intensity of excimer emission for dynamic excimers, but not for static ones. Another difference between dynamic and static excimers formed from pre-associated fragments of molecules is that in the latter case, the fluorophore molecules are close enough to each other and exhibit perturbed absorption and excitation spectra [6].

It is also noteworthy that, since an excimer can only be formed from an excited monomer, the monomer–excited monomer interaction must occur during the lifetime of the latter. Therefore, excimers are more likely to be formed in the systems with a relatively long-lived excited state of the monomer. The factors that affect excimer formation may include the rate of fluorophore diffusion (especially in viscous solvents) [7–10] as well as the relative orientation of several fluorophore units attached to ligands.

The main compounds that form excimers in the excited state are organic molecules.

For organic compounds, the excimer formation is observed in solutions of monochromophoric molecules, in crystals, in polymer matrices, and in other media. Excimer fluorescence in an aromatic hydrocarbon was first detected by T. Forster and K. Kasper [1], who observed the concentration dependence of the fluorescence spectrum of pyrene in solution. For several years, excimer fluorescence of pyrene solutions seemed exceptional. However, the situation changed dramatically in 1962, when it was established that other aromatic hydrocarbons, including benzene, naphthalene [11], 1,2-benzanthracene [12], and many of their derivatives also exhibit excimer fluorescence in solution. It soon became clear that excimer formation by aromatic hydrocarbons is the rule rather than the exception, and covers virtually all planar aromatic molecules and some of their heteroanalogs [3, 13].

In solutions, excimers are formed by diffusion and at high

fluorophore concentrations. In this case, intermolecular excimers are predominantly formed. A solution to this issue may be the combination of several fluorophores in one molecule, which allows for the formation of intramolecular excimers at a low fluorophore concentration in the solution.

Among polymers, the excimer-forming ones include almost all vinylaromatic polymers, some heterocyclic polymers and various copolymers based on them. Luminescence is observed both for solid samples and for liquid solutions. The same properties are characteristic of solid solutions of excimer-forming molecules in polymer matrices. In synthetic aromatic polymers, the effective local concentration of aromatic molecules can be high, while the concentration of polymer chains in solution can be very low. This implies that excimer formation in such polymers can be significant even in dilute solution. Excimer formation in a polymer can be caused by various interactions, including the following: 1) interaction between chromophores belonging to different chains (formation of intermolecular excimers, which strongly depends on concentration); 2) interaction between two chromophores remote from each other, but located in the same chain; this requires conformational changes in the main chain and movements of the side groups during the lifetime of the excited state in order to combine both molecules; 3) interaction of preformed chain segments suitable for excimer formation, present in the polymer before the occurrence of the initial absorption of light. These may be formed by interactions between nearest adjacent chromophores or between two chromophores in loops of the same chain or in different chains in cases where chain movement is restricted [14].

Intramolecular excimers based on organic compounds

As it was mentioned above, the probability of formation of intramolecular excimers in the excited state can be increased by fixing two or more fluorophores in one molecule. Hence, a high local concentration of the fluorophore is achieved even at a low concentration of the excimer-forming compound in solution, and the close distance between the fluorophores promotes their effective π – π overlapping, with subsequent emission of a significant fraction of the excimer. In 1965, Hirayama [15] reported that 1,3-diphenylpropane exhibits both monomer and excimer emission and that the ratio of excimer to monomer emission is independent of dilution [15]. This was the first report on intramolecular excimer fluorescence. As in the intermolecular process, the rate of formation of intramolecular excimers competes with the fluorescence of the monomer. The difference between intermolecular and intramolecular excimers is shown in Fig. 2. It should be noted that excimer formation in case A can only occur at a high concentration of fluorophore in the solution (more than 10^{-3} M, depending on the fluorophore); for case B, highly diluted solutions should be used.

The most studied excimer-forming compounds are planar aromatic molecules, such as benzene, naphthalene, anthracene, pyrene, perylene, and their derivatives. Some of them will be considered further in the article along with the matrices that are used to obtain compounds with intramolecular excimer fluorescence.

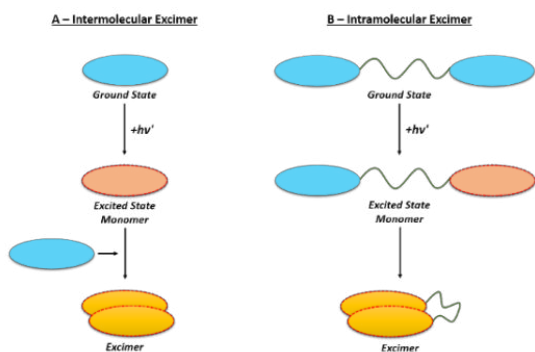


Figure 2. Scheme of the formation of intermolecular and intramolecular excimers.

Naphthalene

Currently, naphthalene and its derivatives are among the most explored organic compounds capable of forming fluorescent excimers in the excited state. Excimers of these compounds have been observed in the crystalline [16–20] and liquid [21, 22] states. Naphthalene derivatives are particularly attractive fluorescent compounds because their excimers are sensitive to local environmental properties and temperature, which leads to a redistribution of the monomer/excimer emission intensity [23, 24]. As a consequence, these phenomena may find many applications, for example, in the development of fluorescent molecular thermometers [25] or sensors for detecting metal ions [25, 26, 27–35].

In concentrated solutions, naphthalene derivatives exhibit intense excimer fluorescence as a result of interactions between adjacent molecules. An alternative effective approach to increasing the probability of excimer formation is to increase the local concentration of chromophores by fixing two or more molecules at close distances, usually using alkyl spacers. A number of studies have shown that the nature and conformation of the alkyl spacer play a key role in the formation of intramolecular excimers, and a stable excimer configuration occurs only in the case of a symmetrical sandwich arrangement [36–38]. There are many examples of grafting several naphthalene molecules onto matrices with different architectures, such as propane [39–41], silanes [42], linear, cyclic, and polymeric siloxanes [43–46], cyclodextrins [35, 47–57], dendrimers [58], cyclotriphosphazenes [59, 60], polymers [61–64], and others [65, 66]. There are also several reports on the combination of naphthalene derivatives and siloxane matrices: interesting examples include cyclic dimers with a siloxane bridge and naphthalene fragments [43] and naphthalene-containing polyhedral oligomeric silsesquioxanes (POSSs) [44], which exhibit excimer fluorescence.

The number of investigations on intramolecular naphthalene excimers is quite extensive and includes dozens of articles [27–29, 34, 36, 40, 42, 44, 48, 61, 64, 67–97]. Particular attention should be drawn to bischromophore compounds, the study of intramolecular excimerization of which has been the subject of many reports (Fig. 3) [36, 46, 71, 72, 76–78, 92]. High intensity of intramolecular excimer luminescence was detected only in symmetrical 1,3-dinaphthylpropanes, which means that the stable excimer configuration is a symmetrical sandwich structure (Fig. 3, left) [36].

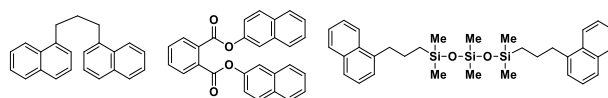


Figure 3. Bichromophore compounds based on naphthalene.

Of note is also the investigations devoted to the synthesis of the compounds for detecting metals, such as calcium [27], nickel [29], zinc [34], cadmium [34], barium [27], as well as temperature and pH sensors [64]. Banerjee *et al.* [29] synthesized and characterized a naphthalene-containing compound with two sulfur atoms. The free compound shows only monomer emission, but in the presence of Ni^{2+} ions, it forms an excimer due to rotation of the C–C single bond. The presence of two sulfur atoms in the molecule promotes the binding of Ni^{2+} ions (Fig. 4).

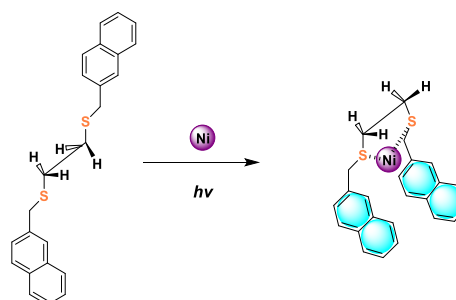


Figure 4. Schematic representation of the probable mechanism of formation of a Ni^{2+} -induced intramolecular excimer [29].

Kawakami *et al.* [27] used the same principle of coordinating the donor atom near the metal ion to detect barium and calcium atoms. Various types of macrocyclic compounds, *e.g.*, crown ethers, are often used for analytical applications such as chemical sensors and in spectrophotometric analysis. Fluorescent reagents that have two aromatic hydrocarbons at both ends of a linear polyether as an open-chain analog of the crown ether are also used in fluorophotometric applications. Figure 5 shows a polyether capable of coordinating barium and calcium ions, and the naphthalene units exhibit intramolecular excimer fluorescence due to their spatial proximity.

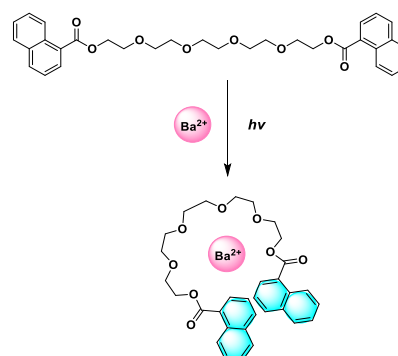


Figure 5. Fluorescent complex formed by the polyether with naphthalene terminal units and a barium ion [27].

It was shown that a chemosensor consisting of a tripodal polyamine receptor bearing naphthalene fluorophores at its ends exhibits intense excimer emission due to the increased probability of excimer formation (Fig. 6). One of the most

interesting characteristics of this system is the dependence of the kinetic and thermodynamic parameters on the dielectric constant of the medium and the protonation degree of the chains. This result opens up the possibility of using such compounds as temperature and pH sensors [64].

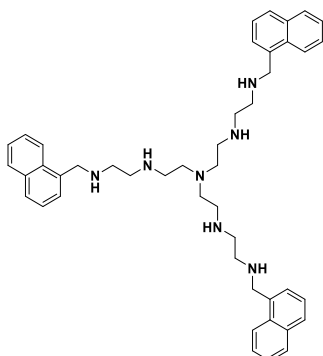


Figure 6. Polyamine receptor containing naphthalene fluorophores at its ends.

Another interesting object for study appeared to be polymers of various structures with naphthalene units distributed along the chain or at the end (Fig. 7). For such systems, the variation of the properties under various external conditions as well as the influence of these factors on the formation of excimers have been studied [45, 61, 68, 74, 75, 84, 86, 87, 96, 98]. Among the polymers explored, polyglutamates [61], polyamides [80], copolymers with methacrylate, methyl methacrylate and butyl methacrylate [74, 84, 86, 87, 98] can be outlined.

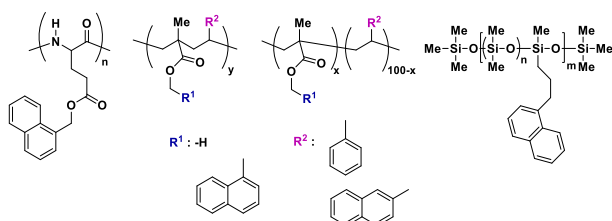


Figure 7. Structures of the polymers bearing naphthalene units in the chain.

The formation of fluorescent intramolecular excimers has been demonstrated experimentally for a series of *N*-methylated polyamides containing naphthyl groups in the main chain. Intramolecular excimers are formed between adjacent and non-adjacent naphthalene groups in ratios depending on the length of the methylene chain separating the aromatic groups. The intensity of excimer fluorescence depends on the solvent and temperature, which affect the conformation of the polymer, and it can be concluded that for naphthalene intramolecular excimer formation can occur at large distances between the chromophores if the intermediate chain is flexible, and polyamides in which the chromophores are linked by a double bond in the polymer chain are flexible heterochain systems in which intramolecular excimer formation is observed [80].

One of the naphthalene derivatives exhibiting excimer fluorescence is naphthalene imide [99, 100]. The photophysical properties of bis-1,8-naphthalimide dyads (NI-L-NI, where L = 3, 4, 6, 8, 9) with different linker lengths and a model compound (NI-C7) were studied (Fig. 8).

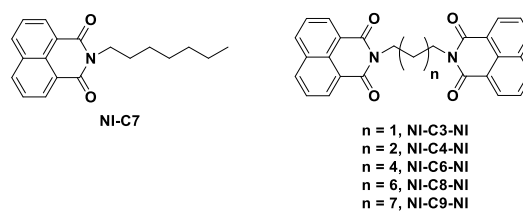


Figure 8. Structures of bis-1,8-naphthalimide dyads with different linker lengths and the model compound.

The monomer fluorescence peak was observed at 379 nm along with a broad emission band at longer wavelengths. When studying the effect of the linker length, it was shown that the excimer emission maximum shifts to the shorter wavelength region with the increasing linker length. This suggests that in the excited state, the interaction between fragments decreases with the increasing linker length [99].

Anthracene

The number of reports on intramolecular excimers of anthracene is quite small compared to the naphthalene and pyrene counterparts [44, 67, 101–109]. This is due to the fact that anthracene molecules tend to dimerize under ultraviolet irradiation, but despite this, excimer emission of anthracene and its derivatives is of interest to researchers. For example, their excimer fluorescence can be used to detect various anions and cations, as well as for the adsorption of metal ions. The formation of an excimer for anthracene occurs from its excited dimer, which has four possible structures: a normal dimer, a twisted dimer, a shifted dimer, and a T-shaped dimer [110, 111].

Most of the works on intramolecular anthracene excimers are devoted to the determination of various anions and the detection and adsorption of metal ions. For example, the polyhedral oligomeric silsesquioxane conjugated with anthracene (AnSQ) was synthesized by the Heck cross-coupling between octavinylsilsesquioxane (OVS) and 9-bromoanthracene (Fig. 9). These host compounds are sensitive to each anionic guest, which results in a change in the formation of the anthracene excimer, exhibiting solvent-dependent fluorescence and allowing the discrimination of ions such as F^- , OH^- , CN^- , and PO_4^{3-} by fluorescence spectroscopy. Depending on the solvent polarity, for example, F^- and CN^- anions quench the fluorescence intensity in THF, but only F^- anion can enhance fluorescence in all other solvents [101].

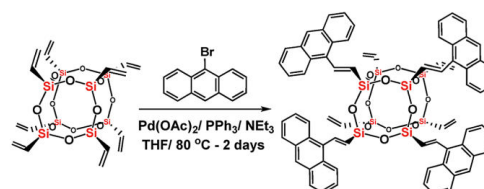


Figure 9. Synthesis of the anthracene-conjugated octameric silsesquioxane [101].

Zhang *et al.* [110] obtained a structural T-shaped excimer between two anthracene fragments from a highly luminescent zinc(II) bis(9-anthryldiamine) complex, which showed intense excimer fluorescence both in solution and in the solid state

[110]. The scheme for obtaining the zinc complex is presented in Fig. 10.

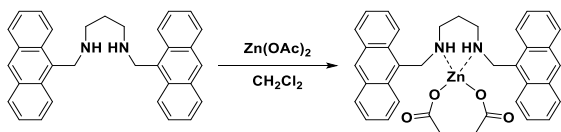


Figure 10. Synthesis of zinc(II) bis(9-anthryldiamine) complex.

Schwarze *et al.* [103] prepared an organic–inorganic hybrid material, which represents a rare example of an anthracene-based heterogeneous excimer sensor probe for Hg^{2+} and Cu^{2+} cations, which can be used as a probe in living organisms without any toxicity. This material can also adsorb the above-mentioned toxic metal ions in real food samples. Therefore, the dual functional activity of selective detection and removal by the same material with the possibility of material recycling is an ideal system with an "on–off" excimer mechanism [103].

Pyrene

Pyrene is the most studied molecule with monomer–excimer fluorescence among all polyaromatic molecules owing to the following properties: a relatively long lifetime, high fluorescence quantum yield, propensity for π -stacking, and sensitivity to the environment (solvatochromism). Pyrene molecules in the excited and ground states intermolecularly form the so-called sandwich excimers, which leads to structureless broad emission at 465–500 nm (monomer emission of pyrene occurs in the range of 370–390 nm). Intermolecular excimers of pyrene and its derivatives are usually formed at high concentrations, but covalent binding of two or more fluorophores is an effective way to increase the probability of excimer formation in the excited state.

The number of works on intramolecular pyrene excimers amounts to more than a hundred. Below we will consider the most interesting examples. Many reports are devoted to the compounds capable of detecting various metal ions, such as lithium [112], sodium [113], magnesium [112], aluminum [114], calcium [112, 115], iron [116, 117], cobalt [118], copper [119, 120], zinc [120], gallium [117], strontium [112, 115], barium [112], neodymium [121], ytterbium [121], and lead [116, 118].

A fluorescent sensor for the detection and removal of Fe^{3+} and Pb^{2+} ions from aqueous solutions was obtained based on pyrene [122]. This molecule contains a large hydrophobic group and a hydrophilic amide group. Due to the flexible aliphatic chain, the conformation of the molecule can be distorted, which promotes the formation of aggregates between the fluorophores. Figure 11 shows the structure of this sensor.

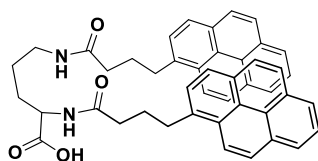


Figure 11. Structure of the pyrene-based sensor.

In order to gain insight into the emission properties depending on the geometric orientation of the pyrene units in a

cyclotriphosphazene framework, Alidađi *et al.* [123] synthesized a series of fluorescent compounds and studied their photophysical properties. The sensor properties of the compounds were investigated for the detection of nitroaromatic compounds (NACs). Figure 12 shows the structure of the compound containing 4 anthracene moieties. The addition of trinitrotoluene, nitrobenzene, and nitrophenol significantly reduced the fluorescence intensity, indicating the possibility of using these compounds as fluorescent sensors with high selectivity and sensitivity to NACs.

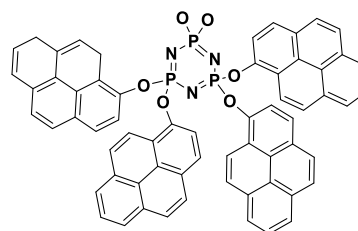


Figure 12. Structure of the fluorophore based on pyrene and cyclotriphosphazene.

A fluorescent sensor for lead, cobalt, and fluorine ions was obtained based on calix[4]arene and pyrene featuring an amide group [118]. When excited with 343 nm light, the excimer emission appears at 450 nm. When heavy metal ions such as Pb^{2+} and Co^{2+} bind to the sensor, the fluorescence intensity of both the monomer and excimer decreases, while the formation of a hydrogen bond with the F^- anion causes quenching of the monomer emission with a slight change in the excimer emission.

One of the most studied methods that provides high sensitivity and selectivity of the analysis of different compounds is high-performance liquid chromatography (HPLC) with intramolecular excimer-forming fluorescent derivatization. In simple terms, this method works as follows: a functional derivative of pyrene is added to a compound with two functional groups (amine, hydroxy, carboxy) to form the product with two pyrenyl units, which is then detected using the HPLC method. Figure 13 shows the scheme of derivatization of 2,4-toluediamine using 4-(1-pyrenyl)butanoyl chloride in the presence of 4-dimethylaminopyridine (DMAP) [124].

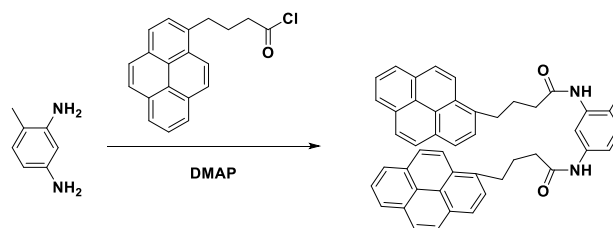


Figure 13. Scheme of the fluorescent derivatization of an aromatic diamine using the pyrene derivative.

This method can also be used to detect other aliphatic diamines [125–127], aromatic diamines [124], catecholamines, indolamines [128], dicarboxylic acids [129–132], tyrosine and tyramine [133], polythiols [134,135], bisphenols [136], and many other compounds (Fig. 14).

Diseases such as diabetes mellitus, arterial hypertension, and Alzheimer's disease are closely related to the changes in membrane fluidity. There are a number of studies dealing with

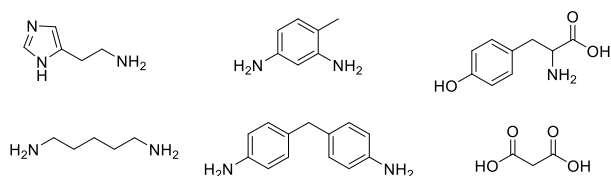


Figure 14. Structures of the compounds that can be detected using the fluorescence derivatization method.

the fluidity of erythrocyte membranes [137–140] and submitochondrial particles of platelets [139] using an intramolecular sensor based on pyrene (Fig. 15). Pyrene was also used to study the fluidity of rat hepatocyte plasma membranes [141], sarcoplasmic reticulum membranes [142, 143], and bacterial membranes [144].

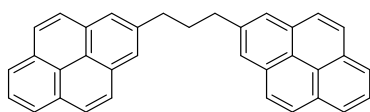


Figure 15. Structure of 1,3-dipyrenylpropane serving as a sensor for determining membrane fluidity.

A series of dendrimeric compounds containing pyrene fragments were synthesized using the 1,3-dipolar cycloaddition between azides and terminal alkynes (Fig. 16) [145]. These compounds can be used as light-harvesting antennas based on the formation of intramolecular excimers. The resulting molecular antenna exhibited efficient energy transfer both in solution and in the solid state.

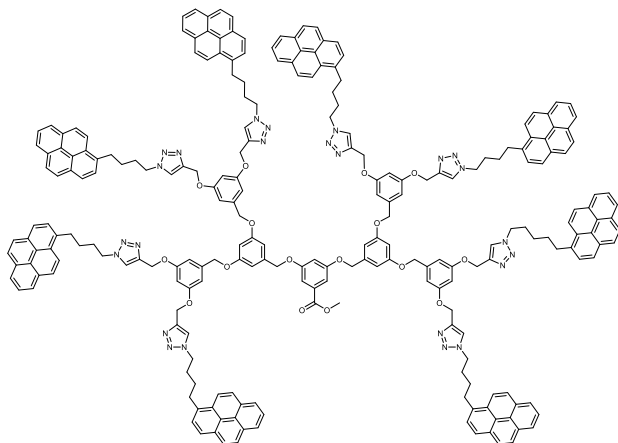


Figure 16. Structure of the dendrimer bearing pyrene units.

In recent years, progress in the analysis of fluorescence decay of pyrene-labeled macromolecules have afforded the quantitative studies on the formation kinetics of pyrene excimers with a diverse range of macromolecular architectures and properties. As reviewed [146], pyrene labeling of macromolecules can provide quantitative information on the properties of a macromolecule. Using pyrene excimer formation, self-associating water-soluble polymers, end-labeled dendrimers, surfactants, and lipids labeled with pyrenyl groups can be studied using the quantitative fluorescence decay analysis as the method for exploring their internal dynamics.

Grafting of pyrene onto polydimethylsiloxane (PDMS) (Py-PDMS-Py, $M_n = 3100$, $M_w/M_n = 1.07$) allowed us to study the

tail-to-tail dynamics and energetics of linear PDMS polymers in ethyl acetate in the temperature range from 255 to 323 K (Fig. 17). It was shown that the activation energy of cyclization is approximately four times higher than the barrier for internal rotations of PDMS. The intramolecular excimer binding energy for Py-PDMS-Py in ethyl acetate is similar to that of the intermolecular pyrene excimer [147].

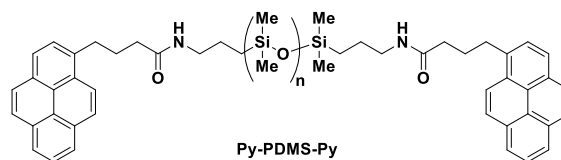


Figure 17. Linear PDMS with terminal pyrene units.

Perylene

Perylene tetracarboxylic acid diimide (PTCDI) derivatives are widely used as dyes owing to their high thermal stability and chemical inertness. They are convenient fluorophores for single-molecule spectroscopy due to their high fluorescence quantum yield and photostability. They also find application as pigments (especially in automotive trimming) owing to their favorable combination of insolubility and stability, light and weather resistance, thermal stability and chemical inertness, and high tinting strength, with the ability to vary shades from red to violet and even black. Modern applications of perylene diimides relate to electronic materials, where they are the best n-type semiconductors available today. In addition, based on the unique combination of optical and redox properties along with stability, perylene diimide dyes have been investigated for many years in electrophotography (xerographic photoreceptors) and photovoltaics. However, most PTCDI derivatives are insoluble in water and poorly soluble in organic solvents, and the soluble ones exhibit a strong tendency to aggregate. This behavior leads to fluorescence quenching, resulting in PTCDI derivatives being rarely used as fluorescent labels for biosensor applications. There are few studies on the intramolecular excimers of perylene [102, 148] and perylene diimide [149, 150] compared to other polycyclic compounds.

For perylene dimers, using a xanthene framework as a matrix, the effect of interchromophore coupling on the mechanisms of dissociation of monomeric units from the dimeric excited state was investigated (Fig. 18). It was found that the coupling between the chromophores was reduced by the inclusion of a xylyl spacer between perylene and xanthine, as well as by using non-polar hexane as a solvent compared to polar acetonitrile [148].

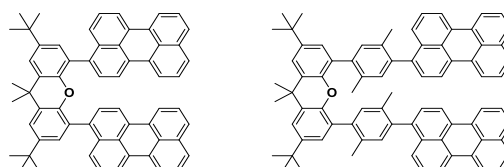


Figure 18. Structures of the xanthene- and perylene-based chromophores.

The creation of artificial light-harvesting complexes requires the ordered arrangement of chromophores in space. To ensure

efficient energy transfer processes, organic dyes must be in close proximity to each other, which often leads to the aggregation and formation of excimer states. In recent years, the attachment of chromophores to nanoparticles has attracted great interest due to improved solar energy harvesting. Such a system was obtained based on two new derivatives of PTCDI and silicon dioxide nanoparticles. PTCDI units were covalently attached to the SiO₂ surface [149]. In the case of a sterically hindered chromophore with phenolic groups, PTCDI fragments undergo only minor aggregation, while in the case of an unsubstituted PTCDI derivative, the excimer with a long lifetime and a significant shift to the red region of the visible spectrum is formed. This work represents the initial investigation of the photophysical and aggregation effects of chromophoric ligands when they are in close proximity upon grafting onto the nanoparticle surface.

Quinoline

Almost all reports on intramolecular quinoline excimers are related to the detection of metal cations, such as magnesium [151], iron [152], copper [152], zinc [151, 153–156], cadmium [153–155], mercury [152], lead [152], as well as fluorine [152] and hydrogen sulfate [152] anions.

Mikata *et al.* [153] demonstrated that the heptadentate nature of the ligands generates specificity to Cd²⁺ ions with enhanced fluorescence due to the formation of an intramolecular excimer between adjacent quinoline rings. In the case of Zn²⁺ ions, in contrast, a non-fluorescent binuclear complex is formed, which emphasizes the strict difference between Cd²⁺ and Zn²⁺ with TQOPEN derivatives (*N,N,N',N'*-tetrakis(2-quinolylmethyl)-3-oxa-1,5-pentanediamine) and the possibility of their selective detection.

A series of fluorescent chemosensors based on calixarenes containing an amide group as a binding unit and a quinoline ring as a fluorescent moiety have been prepared and characterized [152]. These compounds were prepared with different conformations and steric twisting in the calixarene moiety to study its effect on ion selectivity and, consequently, on excimer emission (Fig. 19).

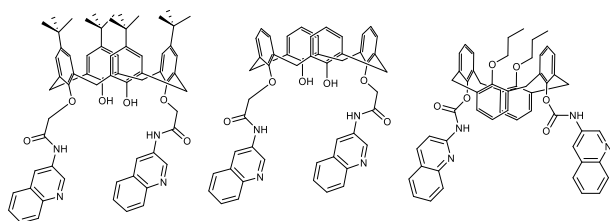


Figure 19. Structures of the fluorescent chemosensors based on calixarenes and quinoline.

The ion binding of the resulting compounds was investigated with a large number of cations and anions. Among the large number of cations, Hg²⁺, Pb²⁺, Fe³⁺, and Cu²⁺ showed strong complexation with all ionophores, which was confirmed by fluorescence and UV-vis spectroscopy. In the case of F⁻ and HSO₄⁻ anions, strong complexation with two ionophores was observed. Interestingly, when interacting with metal ions, strong quenching of the monomer emission was observed with the

formation of excimer emission in the lower energy region, whereas when interacting with anions, excimer emission was absent.

The binuclear zinc complexes themselves can be used to detect various phosphate ions, including pyrophosphate (PPi) [155, 157, 158]. Complexation of the ligand with two equivalents of Zn²⁺ promotes an increase in fluorescence at about 380 nm, and the addition of PPi causes a significant increase in fluorescence at 450 nm. This enhancement in the long-wavelength region is associated with a conformational change in the quinoline moiety, which promotes the formation of an intramolecular excimer between adjacent quinoline units upon binding to pyrophosphate [157].

Carbazole

Among the reports on intramolecular excimers based on carbazole, the most interesting works deal with the investigations on polymers. The properties of carbazole (Cz) and poly(*N*-vinylcarbazole) (PNVC) in the excited state were studied using femtosecond time-resolved fluorescence and time-dependent density functional theory (TDDFT) (Fig. 20). When excited with 280 nm light, the polymer exhibits monomer fluorescence peaks at 351 and 362 nm and an excimer peak at a wavelength greater than 400 nm. The TDDFT calculations show that the excitation energy is almost independent of conformational changes and the degree of polymerization [159].

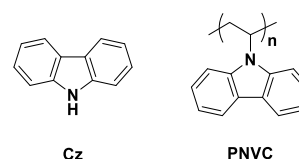


Figure 20. Structures of carbazole (Cz) and poly(*N*-vinylcarbazole) (PNVC).

The photophysical properties of a series of amphiphilic alternating copolymers based on *N*-vinylcarbazole (VCz) with diethyl fumarate (DEF), maleic anhydride (MAN), and citraconic anhydride (CAN) (a-CzEF, a-CzMAN, and a-CzCAN, respectively) were studied (Fig. 21). These polymers may be promising candidates for light-harvesting materials. The fluorescence spectra of all alternating copolymers of VCz and the three acceptor monomers showed excimer emission which depended on the monomer ratio and the steric hindrance of the comonomers. The effect of solvent on the fluorescence spectra suggests that the alternating copolymers have few fluorescence quenching sites other than the excimer formation sites and that efficient energy migration occurs [160].

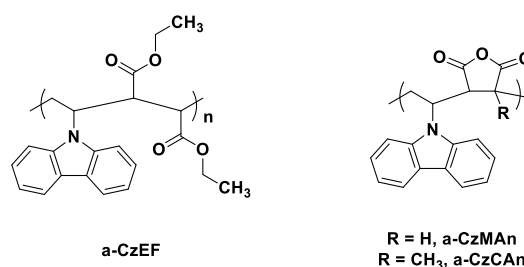


Figure 21. Structures of the amphiphilic alternating copolymers.

Boron difluoride β -diketonates

Fluorophores based on boron difluoride complexes, in particular, boron difluoride β -diketonates are promising compounds for obtaining various sensors and materials based on them owing to their unique photophysical properties and the ability to form excimers and exciplexes in an excited state.

A bischromophore compound and a polymer based on a difunctional derivative of DBMBF₂ and tetramethyldisiloxane were obtained based on a monofunctional derivative of DBMBF₂ and tetramethyldisiloxane (Fig. 22) [161]. Both reactions were carried out in isopropanol using Spyer's catalyst.

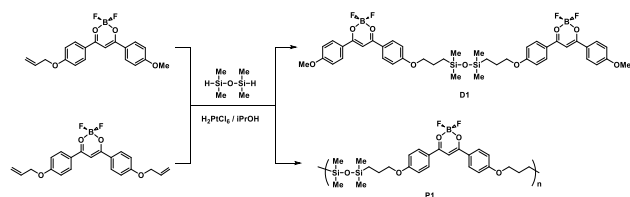


Figure 22. Synthesis of the bichromophore and polymer compounds containing a boron difluoride complex in their structures.

The starting compounds have almost indistinguishable spectra in the blue region (with a maximum at 437 nm). However, for the dimer and polymer, despite their unchanged emission maxima at 437 nm, an additional low-energy shoulder with a maximum at about 550 nm was observed. As a result, in contrast to the dark blue emission of the monomer, a dilute solution of polymer P1 in dichloromethane exhibits white fluorescence under UV irradiation. When studying the physical properties of these complexes in the solid state, it was demonstrated that they exhibit mechanochromic luminescence.

Interesting objects for investigations are curcuminoid boron difluoride derivatives. Three compounds and their covalent homodimers chemically linked by a polymethylene chain were synthesized, characterized, and studied (Fig. 23) [162].

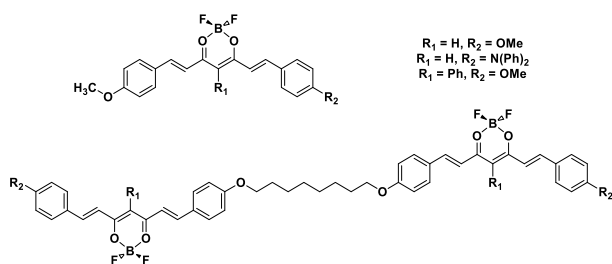


Figure 23. Structures of curcuminoid boron difluoride derivatives: the monomer and two homodimers.

Curcuminoid boron difluorides with different linkers between fluorophores were also prepared and their photophysical properties in solution were studied (Fig. 24) [163].

Such curcuminoids may be attributed to a versatile class of organic dyes, based on which it is possible to create supramolecular systems with fluorescence emission in the range from visible to near infrared and photochemical properties for use in sensor and photochromic materials.

Fedorenko *et al.* [164] obtained the polymers containing benzoylacetate groups of boron difluoride by double acylation

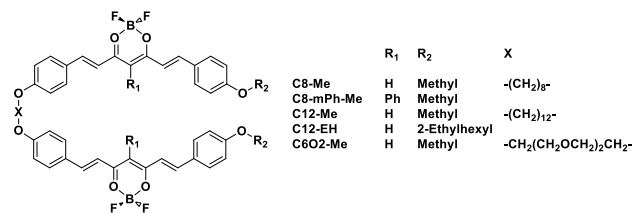


Figure 24. Structure of the curcuminoid boron difluoride dimers featuring different linkers.

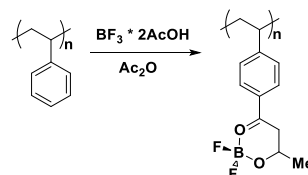


Figure 25. Synthesis of the polymers with BF₂-benzoylacetate groups.

with acetic anhydride and boron trifluoride (Fig. 25). Polystyrene and poly(styrene-co-methyl methacrylate) were chosen as polymers.

Luminescence of the resulting polymers in solutions and films was studied. The quantum yields of luminescence of the polymer solutions appeared to be significantly higher than those of the low-molecular analog, boron difluoride benzoylacetate. For the polymer in which the styrene units were separated by methyl methacrylate groups, monomer luminescence of BF₂-benzoylacetate groups was observed at the low polymer concentrations in solution, and excimer luminescence—at high concentrations. In the case of the polystyrene-based polymers in which the BF₂-benzoylacetate groups and phenyl rings were not separated, fluorescence of intramolecular exciplexes was observed in dilute solutions and, with increasing concentration, luminescence of intermolecular exciplexes. The ability to form excimers determined the increased photostability of the resulting polymers.

Bifunctional linear siloxanes containing methyl and/or phenyl substituents at the silicon atom can be used as a flexible and convenient matrix. Our research group obtained a series of dimers and showed that an increase in the length of the siloxane spacer in dyads significantly affects the formation of H-dimers in the ground state: in the absorption spectra, there is an increase in the intensity of the 0–0 transition from di-, trisiloxane to pentasiloxane and, in the emission spectra, the contribution of excimer emission decreases upon transition from di-, trisiloxane linkers to pentasiloxane (Fig. 26) [165, 166].

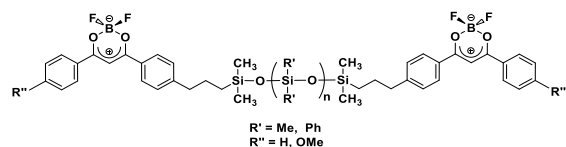


Figure 26. Dimers based on the linear siloxanes and derivatives of boron dibenzoylmethane difluoride.

Moreover, based on stereoregular cyclic siloxanes, tetrachromophore derivatives containing four DBMBF₂ moieties in one plane were obtained (Fig. 27). These compounds exhibit bright intramolecular excimer fluorescence and in solutions have

CIE color coordinates very close to the white point, which allows these compounds to be considered as promising objects for the development of new-generation monofluorophore emission materials for white illumination [167].

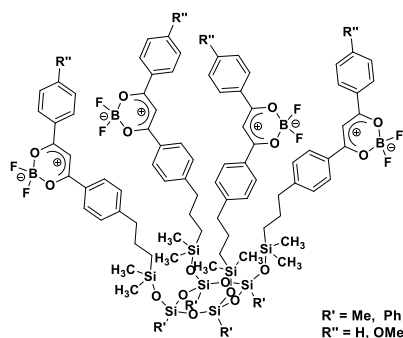


Figure 27. Tetrachromophore derivatives containing four DBMBF₂ units in one plane.

Polysiloxanes with various distributions of fluorophores along the chain were prepared (Fig. 28). It was shown that the intramolecular H-dimers exist in the ground state. The polymers explored had bright fluorescence in solution and in the solid state, consisting of monomer and excimer emission bands. Thermally activated delayed fluorescence was observed in solution and in the solid state. The resulting properties of the polymers make them useful as optical materials, sensors, or visualization tools [168].

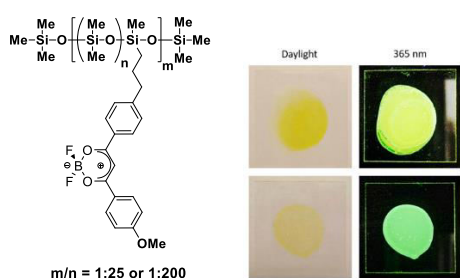


Figure 28. Structure of the polysiloxanes with different fluorophore distributions (left) and photographs of the polymers in daylight and under excitation with 365 nm light (right) [168].

Matrices used to obtain compounds with intramolecular excimer fluorescence

As mentioned earlier, the most effective way to increase the probability of intramolecular excimer formation is to graft two or more fluorophores onto a single matrix. Different compounds can be used as matrices, from methane to polymers of various structures.

It is believed that the formation of intramolecular excimers is determined by the microstructure of the molecules to which the chromophores are attached. Apparently, there are two factors that control the formation of an intramolecular excimer: conformational changes in the molecule and the geometric arrangement of the two chromophores in the excimer state, which is determined by the structure of the molecule. In this regard, Hirayama proposed the $n = 3$ rule for the formation of an intramolecular excimer [15]. In this work, the fluorescence

spectra of various diphenyl- and triphenylalkanes were measured in cyclohexane and 1,4-dioxane. It was found that the compounds having such a structure where the phenyl groups are located along the main alkane chain and are separated by three carbon atoms, such as 1,3-diphenylpropane or 1,3,5-triphenylpentane, exhibit unique fluorescence characteristics, including a decrease in the fluorescence quantum yield and the appearance of a long-wavelength band at 330 nm, responsible for the emission of an excimer. Therefore, the results show that the most favorable arrangement of chromophores is a symmetrical parallel sandwich arrangement, which can be formed precisely in a compound having a molecular structure in which two aromatic groups along the alkane chain are separated by three carbon atoms.

Therefore, the most frequently used matrices for studying the influence of conformations on intramolecular excimerization are alkanes, such as methane [169, 170], ethane [108, 109], propane [7, 8, 40, 81, 100, 142, 171–179], butane [100], pentane [105, 180, 181] and others [7, 36, 76, 78, 99, 107, 182–186]. Due to the different lengths of the alkane chain, it is possible to regulate the monomer–excimer ratio and to determine the optimal conditions for the given goal.

Another class of compounds that is most frequently mentioned in the literature are amines [187], aliphatic diamines [103, 125–127, 153, 154, 157, 188, 189], aromatic diamines [124], and polyamines [91].

Calixarenes represent a family of macrocyclic compounds with a variable number of phenolic units linked by methylene bridges at the *ortho*-position. The number of aromatic units of these *meta*-cyclophanes can range from 4 to 20, although the most common calixarenes involve 4, 5, 6, 7, and 8 units. Such cyclooligomeric phenols are of great interest as molecular scaffolds for the creation of selective ionophores, many of which can be used as fluorescent ion sensors [190]. The advantage of this matrix is the possibility of grafting a large number of fluorophores into a single molecule. The examples of calixarenes structures with different numbers of units and stereoregularities are shown in Fig. 29. Calixarenes as host molecules for anions or cations are of particular interest owing to the presence of the following two different reactive centers: phenolic OH and other groups at the *para*-position, which can be easily functionalized with various cation-doping groups, such as carboxy, amides, crown ether, and azacrown ether. In particular, amide groups are known to capture not only cations

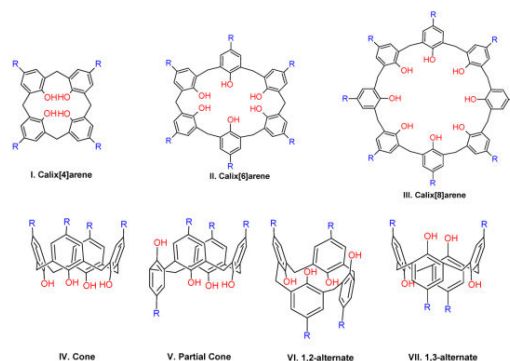


Figure 29. Examples of the calixarene structures with different numbers of units (top) and stereoregularities (bottom).

via the carbonyl oxygen atoms but also anions via hydrogen bonds between the anions and the acidic hydrogen atoms on the nitrogen atoms. Therefore, calixarenes can be a convenient matrix for the fixation of several fluorophores with various applications. In most studies, calixarenes are used for the grafting of pyrene molecules and the detection of metal ions [113, 115, 152, 191–193].

Hexachlorocyclotriphosphazene ($N_3P_3Cl_6$) can be chosen as a cyclic matrix due to its unique properties, such as high thermal stability and high reactivity in nucleophilic reactions under basic conditions. In addition, $N_3P_3Cl_6$ is a commercially available reagent and can serve as an ideal platform for fluorophore systems since it is optically inert due to the lack of absorption in the visible light range and allows for the arrangement of one to six fluorophore moieties in one molecule. A number of fluorescent dyes were synthesized based on cyclotriphosphazenes and organic fluorophores and their photophysical properties were studied [123, 194, 195]. Electroluminescence [195] and sensor properties for the detection of NACs [123] were studied for some of the resulting compounds.

Dendrimers are macromolecules with a symmetrical tree-like structure and regular branching. These unique compounds allow a large number of fluorophores to be grafted onto a single macromolecule depending on the degree of generation: the higher the generation, the greater the number of fluorophores that can be grafted. A number of dendrimeric compounds containing pyrene (Fig. 16) [145, 196] and anthracene units [197] (Fig. 30) were obtained. These structures can be used to produce light-harvesting antennas owing to the formation of intramolecular excimers. Such molecular antennas exhibit efficient energy transfer both in solution and in the solid state owing to the high degree of generation and a large number of fluorophores [145]. It is also expected that the dendrimers with aggregation-induced excimer emission can be used in the field of photoactive systems in the future [196].

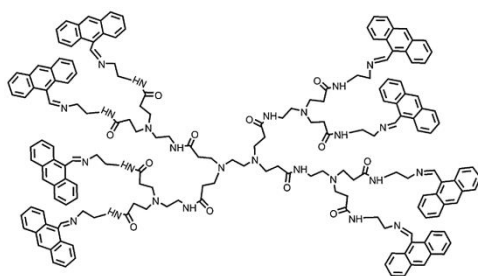


Figure 30. Dendrimeric compound containing anthracene units.

Furthermore, the compounds such as ethers and esters [71, 72, 85, 187, 198], glycols [73, 75], xanthenes [148, 150], oligopeptides [199, 200], urea [69], cholic acid [201], cryptands [202], and polymers of various structures [61, 68, 74, 79, 80, 82, 84, 86, 87, 98, 159, 160, 203–210] can be used as matrices.

A particular group among all kinds of organic and organoelement compounds is comprised by the matrices based on various silicon compounds. For example, silanes can be used as analogs of alkanes [42, 67, 169, 211–214]. The derivatives of di(1-pyrenyl)silane and di(1-pyrenyl)methane were obtained to study intramolecular excimerization [169]. These compounds demonstrated a high degree of intramolecular excimer emission

in polar organic solvents, such as DMSO, and the monomer–excimer fluorescence ratio strongly depended on the dielectric constant of the solvent in use. It was also shown that significant changes in optical properties do not occur when changing the carbon atom to the silicon one.

Oligosilanes were used as matrices to obtain naphthalene and anthracene dimers (Fig. 31) [42, 67]. The photophysical properties of (1-naphthyl)oligosilanes, which lead to the formation of excimers or charge transfer complexes (CTCs), strongly depend on the length of the silicon chain and the polarity of the medium. When using the oligosilane with $n = 3$, the emission involves π – π^* interaction between the fluorophores and σ – π^* interaction of the CTC. In a non-polar solvent, such as cyclohexane, the CTC is less stable, so the dinaphthyl derivative forms an excimer. However, in a polar solvent, the CTC is more stable, so its emission is observed at a longer wavelength (with lower energy) than the excimer emission. And in the case of oligosilanes with $n = 6$, the results showed that only σ – π^* interaction was observed in the emission spectra due to the higher degree of CTC formation for the longer silane chain [67].

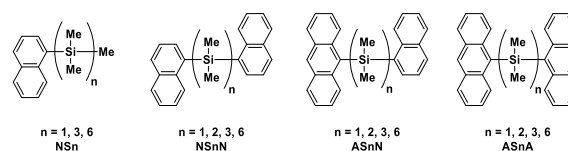


Figure 31. Structures of the oligosilanes with naphthalene and anthracene fragments.

Cyclic structures such as cyclohexasilane also allow multiple fluorophores to be arranged in close proximity to each other [214]. Intramolecular excimer emission was observed for *cis*-1,4-di(1-pyrenyl)decamethylcyclohexasilane in non-polar solvents (Fig. 32, left). The advantage of this matrix is that the fluorophores are coplanar. Time-resolved fluorescence spectroscopy and kinetic modeling showed that the driving force for excimer formation is very low and that the process is determined by the flexibility of the silicon ring. In a polar solvent, namely, acetonitrile, the photoinduced electron transfer occurs, with the cyclohexasilane ring acting as an electron donor and the pyrenyl group serving as an electron acceptor.

An example of another stereoregular structure is tetracyclosiloxane [215]. Compounds with intramolecular thiophene excimers were obtained on the basis of the siloxane framework (Fig. 32, right). In such a structure, the formation of excimers occurs extremely efficiently, and the new bound excited states are quite stable due to the siloxane ring and the close arrangement of the thiophene entities in space. A similar stereoregular matrix was also used for grafting boron dibenzoylmethanate derivatives and is shown in Fig. 27 [167].

Ganesan *et al.* [216] studied the photophysical properties of the compounds based on cyclosiloxanes and naphthalene diimide. The investigations performed showed that in dilute solutions of all three compounds, monomer (410 nm) and excimer (510 nm) emissions appear over the entire measured concentration range. This type of emission was observed even at concentrations of about 10^{-7} M, indicating that the emission at 510 nm is due to the intramolecular formation of excimers. At higher concentrations, the intermolecular interaction of fluorophores occurs.

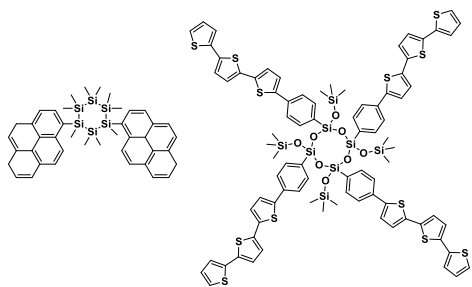


Figure 32. Structures of *cis*-1,4-di(1-pyrenyl)decamethylcyclohexasilane (left) and tetracyclosiloxane with thiophene units (right).

Linear siloxanes with various substituents are accessible reagents and convenient spacers/matrices for the preparation of dimers. A series of compounds were also obtained on their basis, where the derivatives of naphthalene diimide [216] and boron difluoride dibenzoylmethanate (Fig. 3, 22, 26) [161, 165, 166] were used as fluorophores.

Polyhedral oligomeric silsesquioxanes are hybrid materials with well-defined nanostructures and unique dual nature combining the properties of both inorganic (siloxane cage) and organic material (peripheral groups). The general formula is $[\text{RSiO}_{3/2}]_n$ or R_nT_n , where $n = 6, 8, 10,$ and 12 and R represents various polar or non-polar organic substituents, such as aryl, alkyl or any derivatives thereof covalently attached to silicon atoms. Among the various POSS molecules, octahedral silsesquioxane ($[\text{RSiO}_{3/2}]_8$ or R_8T_8) is the most commonly used. These compounds can be utilized as complex materials with multiple functions targeting therapeutic applications in controlled drug delivery, tissue engineering, *etc.* [217, 218]. Several studies reported the use of POSSs as matrices for grafting several fluorophore molecules [44, 101]. Polycyclic aromatic hydrocarbons such as naphthalene, anthracene, and pyrene were integrated into POSSs with flexible ionic linkers and their luminescent properties were explored (Fig. 33).

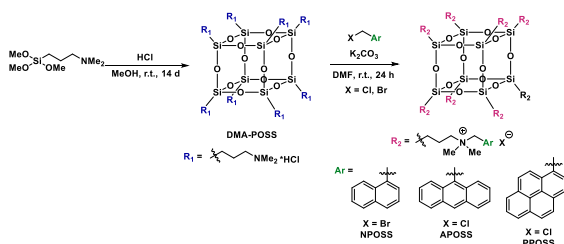


Figure 33. Synthesis of the POSSs with polycyclic aromatic hydrocarbons.

Polymeric siloxanes allow for obtaining the compounds with variable distribution of fluorophores along the chain, thus regulating their fluorescence properties. There are a number of works where the matrix is a siloxane chain and an excimer-forming fragment is phenyl [219, 220], naphthalene [46], pyrene [147], and derivatives of boron difluoride dibenzoylmethanate [168]. Nanoparticles such as SiO_2 nanoparticles [103, 149] can also be used as a matrix.

Conclusions

Based on the presented overview of the compounds capable

of forming intramolecular excimers in the excited state, it can be concluded that the most studied species in terms of photophysical properties and applications are polyaromatic compounds such as naphthalene and pyrene. The properties of these compounds are most often used for such applications as sensors for detecting various metals, including alkali and alkaline earth metals, iron, some rare earth elements, as well as for determining diamines (aromatic and aliphatic). For other aromatic compounds and their derivatives, there are currently few works on intramolecular excimerization and their application potential has not been widely disclosed and explored. These fluorophores are promising compounds from the viewpoints of synthesis and investigation of their properties, since they allow fine-tuning the structure, thus changing various properties (for example, fluorescence quantum yield and extinction coefficient), which can facilitate the use of the ensuing complexes in various fields of science. It is also worth mentioning that there is a wide variety of matrices that can be used to fix several fluorophores at close range with a high probability of their interaction with subsequent formation of intramolecular excimers in an excited state. Alkanes (methane, ethane, propane, butane, and others), silanes (linear and cyclic), diamines (aliphatic and aromatic), calixarenes, hexachlorotriphosphazene, polymers of various structures, and siloxanes are the compounds that are most frequently used as matrices.

Acknowledgements

The sections "Naphthalene" and "Boron difluoride β -diketonates" were prepared with financial support from the Russian Science Foundation (project no. 24-13-00443). The other sections were prepared with financial support from the Ministry of Science and Higher Education of the Russian Federation (agreement no. 075-00277-24-00).

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