Rapid Communication in Photoscience(2016), vol. 5, no. 2, pp. 21-26
http://dx.doi.org/10.5857/RCP.2016.5.2.21 ISSN 2288-4564(Online)

Review article

The Recent Current of Fluorescent Polypyridine Compounds Having Photofuctionality

Chang-Shik Choi

Department of Food and Fermentation, Far East University, Eumseong, Chungbuk 27601, Korea

ABSTRACT: Many kinds of fluorescent polypyridine compounds including bpy and dppz derivatives are described in understanding the recent current of fluorescent materials having photofuctionality. Those polypyridine compounds have the photofunctionality such as the fluorescence recognition and/or photo-switching. Furthermore, those compounds are applicated for the construction of long ranged photoinduced electron/energy transfer system. Various fluorescent π-conjugation systems connected by amide or imine bond as well as the simple fluorescent bpy derivatives are introduced in this review paper.

INTRODUCTION

Fluorescent artificial receptors are attracting considerable interest in various stages in sensory, biochemical, medical, photoelectronic, and other fields.[1] These receptors are composed of the host unit (binding site) and the signaling unit (fluorescent site), and communication between the two units is essential for guest specific response. Though various molecular designs have been proposed in order to develop efficient fluorescent receptors,[2] most of those receptors are designed by introducing molecular recognition site(s) into the known fluorophores.

Polypyridine compounds are useful molecular units as a binding site. They have multiple interaction sites, and the number of pyridine units is adjustable. 2,2'-Bipyridine (bpy) is most studied among them due to its excellent property as a bidentate ligand and as a hydrogen bond acceptor.[3] Moreover, rational receptor design using more than one bpy can reach selective and strong interaction with guests.[4] Therefore, many kinds of fluorescent polypyridine compounds including bpy and dppz derivatives are described in understanding the recent current of fluorescent materials having photofuctionality

Bpy and bpy derivatives

2,2'-Bipyridine (bpy) and its derivatives are the representative member of polypyridine compounds[5] (Fig. 1), which serve as the chelating agents for various metal cations, especially for transition metal cations because of their π-acid nature. Bpy was first prepared in 1888 by the dry distillation of the copper salt of picolinic acid.[6] Bpy and its derivatives are generally non-fluorescent,[5] and relatively little is known about their fluorescent properties. Only 3,3'-dihydroxy-bpy (**33'OH**)[7,8] has been known to show a moderate fluorescence (Φ = 0.2) among bpy derivatives. It was suggested[8] that the green fluorescence of **33'OH** was from the tautomeric form of **33'OH**, in which two hydroxy protons were transferred to the ring nitrogens.

***To whom correspondence should be addressed.**
E-mail: cschoi@kdu.ac.kr

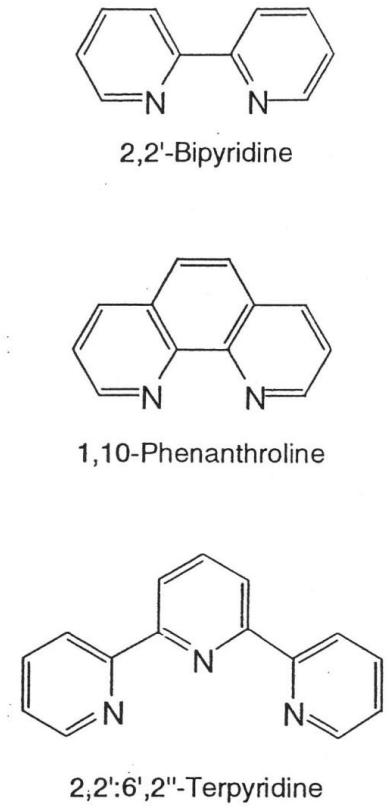
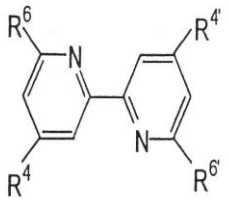


Fig. 1. The basic structures composing of polypyridine compounds

In my laboratory, as shown in Fig. 2, it was found that 6,6'-diamino-2,2'-bipyridine (**1a**) displayed a relatively strong fluorescence (λ_{max} = 404 nm; Φ = 0.45 in ethanol) in the near-UV region.[9] Based on this finding, other amino and/or chloro substituted bpys were synthesized and studied to find that only 6-amino-substituted derivatives exhibited efficient fluorescence. The fluorescence of 6-amino-6'-chloro-bpy (**3a**) was the strongest (λ_{max} = 429 nm; Φ = 0.78 in ethanol) among them. As it has already been stated in 1-1-2, alkyl derivative of **1a** (**1b**) shows excellent functionality as a fluorescent receptor for diphenyl phosphate derivatives.

1,10-Phenanthroline has more rigid framework compared to bpy, and is the attractive target for construction of tunable chromophores.[10] The parent 1,10-phenathroline possesses a low fluorescence quantum yield ($\Phi \leq 0.01$) and a rather short emission wavelength ($\lambda_{em} \approx 360$ nm).[11]



	R^4	R^6	$R^{4'}$	$R^{6'}$	Φ
6-Amino-					
1a	H	NH_2	H	NH_2	0.45
2	H	NH_2	H	H	0.42
3a	H	NH_2	H	Cl	0.78
4	H	NH_2	H	Br	0.48
4-Amino-					
5	NH_2	H	H	H	$\sim 10^{-2}$
6	NH_2	H	NH_2	H	0
7	NH_2	H	Cl	H	0
8	NH_2	H	H	Cl	0.05
Monochloro-					
9	Cl	H	H	H	0
10	H	Cl	H	H	0
Dichloro-					
11	Cl	H	Cl	H	0
12	H	Cl	H	Cl	0
6-Alkylamino-					
1b	H	NH(Hx)	H	NH(Hx)	0.26
1c	H	NPr_2	H	NPr_2	0.23
3b	H	NHPr	H	Cl	0.49
3c	H	NPr_2	H	Cl	0.34

Hx = 1-Hexyl

Fig. 2. The substituted 2,2'-bipyridines as a fluorescent organic compounds

Quite recently, H. S. Joshi *et al* reported interesting results.[12] Since the most intense electronic transition of the 1,10-phenanthroline skeleton is polarized along the 3-8 positions,[13] they hypothesized that increasing the conjugation along this axis may provide fluorescent derivatives with emission in the visible range, and have therefore synthesized 3,8-bis(arylethynyl)-1,10-phenanthrolines (**1**) as novel fluorescent chromophores (Fig. 3). The compounds **1** had red-shifted absorption and showed a strong fluorescence (λ_{max} = 384 - 416 nm; Φ = 0.49 – 0.87 in dichloromethane).[12]

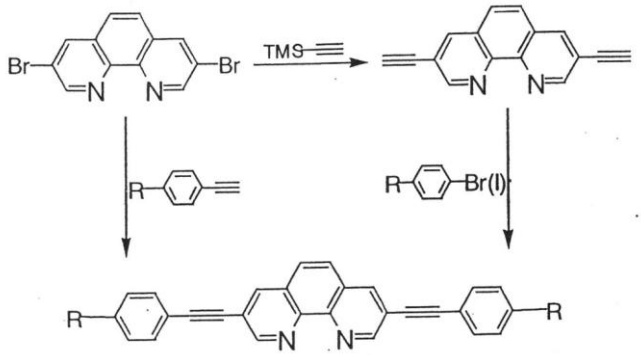
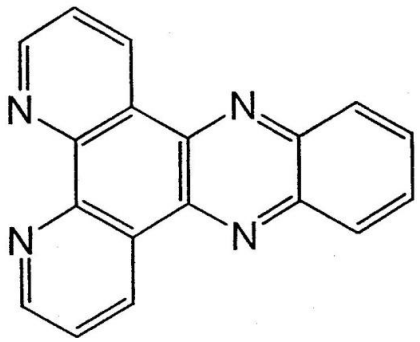


Fig. 3. Synthesis of conjugated 1,10-phenanthrolines 1 by Pd-mediated cross-coupling reactions. TMS = trimethylsilyl

The tridentate ligand 2,2':6',2''-terpyridine (terpy, Fig. 1) was first prepared over 60 years ago,[14] and its coordination chemistry, along with that of its substituted analogues, has been widely studied.[15] 4'-Aryl-terpys have been found to be useful for the colorimetric determination of iron(II).[16] Recently, the fluorescent derivatives of substituted terpys have been found in my laboratory. Among them, 6-amino-terpy, 6,6''-diamino-terpy and 4'-phenyl-terpy showed a relatively strong fluorescence.[17]

Dipyrido[3,2-*a*:2',3'-*c*]phenazine (dppz) and dppz derivatives

Preparation of dppz (Fig. 4) goes back to the beginning of 1970's, which was obtained by reaction of 1,2-diaminobenzene and 1,10-phenanthroline-5,6-dione.[18] The spectroscopic studies of dppz were done by Ackermann and Interrante,[19] and recently by Verhoeven and Reinhoudt *et al.*[20] from the middle of 1980's. According to the former study, lowest energy absorption maximum of dppz was in between 342 nm and 378 nm in ethanol at room temperature. While as to the fluorescent properties of dppz, fluorescence appeared at λ_{max} = 544 nm with the lifetime of <0.01 s in ethanol at room temperature, but no quantum yield was reported at all. However, Verhoeven and Reinhoudt *et al.* reported that at ambient temperature or even at 77 K virtually no fluorescence is observed for dppz, though phosphorescence was observed in the broad range between 540 nm and 650 nm at 77 K in an EtOH/MeOH (4:1) glass. We cloud not observe any fluorescence of dppz at all, supporting the results of Verhoeven and Reinhoudt *et al*.



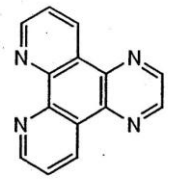
Dipyrido[3,2-*a*:2',3'-*c*]phenazine (dppz)

Fig. 4. Structure of dppz

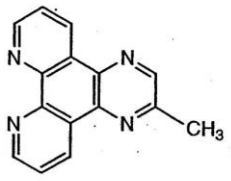
E. Amouyal *et al.*[21] investigated the redox potentials of dppz together with those of phenazine. They reported that dppz is more easily reduced than bpy by 1 V due to the lower energy π* orbital of the phenazine moiety, thus the π-accepting site in dipyridophenazine was localized on the phenazine portion of the molecule.

Phenazine constitutes a part of dppz, and chemistry of phenazine has a long history.[22] As to the fluorescent properties of phenazine derivatives, spectroscopic studies of their amino substituents were reported.[23] The influence of amino substituents on the excited π-π* singlet states of phenazine was investigated by absorption, fluorescence, and polarization spectroscopy at 77 K, and the Coulomb interaction of the free electron pair of the amino groups with the π-electrons of phenazine was shown to be responsible, essentially, for the drastic changes of the observed absorption spectra upon amine substitution. In particular, 2-aminophenazine is a good fluorophore according to Russian's report.[24] They reported that 2-aminophenazine showed relatively strong emission (Φ= 0.23 – 0.63) in the range between 518 nm and 596 nm in nonpolar to protic solvents.

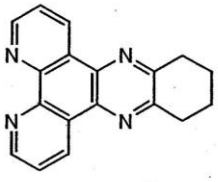
Metal complexes of dppz have been studied for more than a decade. As a photosensitizing unit, Ru(II) and Os(II) complexes of dppz were used for a construction of the photoinduced electron/energy transfer system from the beginning of 1980.[21,25,26] Those complexes were also used as DNA intercalators.[27,28] E. Amouyal *et al*. reported a detailed photophysical and electrochemical properties of $[Ru(bpy)_2(dppz)]^{2+}$.[21] According to their report, the orbital involved in the light-induced charge transfer (optical orbital) and the orbital involved in the first electrochemical reduction step (redox orbital) are separated, both in space and energy, within the complex $[Ru(bpy)_2(dppz)]^{2+}$; the optical orbital lies on the bpy moieties and the redox orbital on the phenazine portion of the molecule. Emission of $[Ru(bpy)_2(dppz)]^{2+}$ originates from an excited state localized on the bpy moieties, and not on the phenazine portion of the complex.



Dipyrido[3,2-*f*:2',3'-*h*]quinoxaline



2-Methyldipyrido[3,2-*f*:2',3'-*h*]quinoxaline



10,11,12,13-Tetrahydrodipyrido[3,2-*a*:2',3'-*c*]phenazine

Fig. 5. Structures of tetraazatriphenylenes as lanthanide ion sensitizers

Polyanionic double-stranded DNA can be the target of cationic metal complexes such as $[Ru(bpy)_2(dppz)]^{2+}$,[29] and $[Os(bpy)_2(dppz)]^{2+}$,[28] containing extended planar π-electron systems for intercalation between the base pairs. DNA intercalation prevents deactivation of the MLCT excited states of $[Ru(bpy)_2(dppz)]^{2+}$ and $[Os(bpy)_2(dppz)]^{2+}$ by water, and switches on their luminescence. The luminescence of $[Os(bpy)_2(dppz)]^{2+}$ showed in the far red region of the spectrum by DNA intercalation and its luminescence decay was observed as a multi-exponential when the complex intercalated into DNA, while it was observed as a mono-exponential in acetonitrile.[28] Protection from the detrimental effects of aqueous media can also be achieved by incorporating the sensitive MLCT excited state of $[Ru(bpy)_2(dppz)]^{2+}$ within anionic detergent micelles.[30] Conversely, $[Ru(bpy)_2(dppz)]^{2+}$ and $[Os(bpy)_2(dppz)]^{2+}$ serve as sensitive detectors for these important anionic assemblies. Yam *et al.* reported the syntheses of a series Ru(II) polypyridine complexes with a crown ether-containing ligand dipyrido[3,2-a;2',3'-c]phenazo-15-crown-5(dppzc).[31] The cation-binding properties of those complexes have been studied by electronic absorption spectroscopy and cyclic voltammetry and by electrospray ionization mass spectrometry. Verhoeven and Reinhoudt *et al* synthesized a series of tetraaza- triphenylene derivatives including dppz that constitutes a new and efficient class of sensitizers with significant complexing power for lanthanide ions. The tetraazatriphenylenes such as dipyrido[3,2-*f*:2',3'-*h*]quinoxaline, 2-methyldipyrido[3,2-*f*:2',3'-*h*]quinoxaline, or 10,11,12,13-tetrahydrodipyrido[3,2-*a*:2',3'-*c*]phenazine (Fig. 5) have the ability to sensitize the luminescence of different lanthanide ions within a practically suitable excitation window, and the 2:1 (ligand-ion) complexes are stable even at low concentrations in acetonitrile. High luminescence quantum yields were obtained for both Eu^{3+} and Tb^{3+} complexes of dipyrido[3,2-*f*:2',3'-*h*]quinoxaline, 2-methyldipyrido[3,2-*f*:2',3'-*h*]quinoxaline, or 10,11,12,13-tetrahydro dipyrido[3,2-*a*:2',3'-*c*]phenazine. However, no luminescence was observed at all for the dppz complexes.[20]

Though bpy is the well known chelating compounds and shows variety of functionality, only a few bpy derivatives show efficient fluorescence. The nonfluorescent bpy unit and the fluorescent aminophenazine unit is integrated into 7-amino-dppz (Fig. 6). Since it was pointed out that the bpy-localized and the phenazine-localized л* orbitals have relatively small overlap, this integration might not deteriorate the fluorescent properties of the aminophenazine

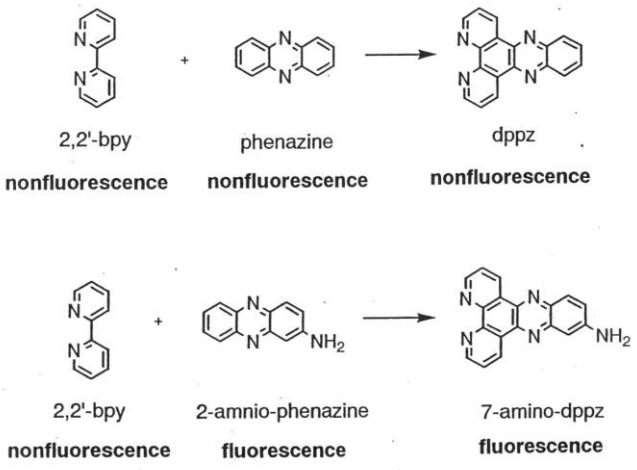


Fig. 6. The fluorescence properties of dppz and 7-amino-dppz compound

fluorophore unit, offering novel method for designing fluorescent bpy derivatives. Since the compound has the diimine-type metal coordination site and the amino group suitable for introducing additional functional units, fluorescent properties of the compound can be tuned by metal chelation and/or functional unit attached to the amino group if the compound shows efficient fluorescence

Absorption spectra of 7-amino-dppz and 2-aminophenazine were simulated using ZINDO (CI = 9) method after successive geometrical optimization by MM2 and MOPAC/AM1 calculations (Fig. 7). The simulated spectra were well consistent with the observed spectra.[32] The lowest energy absorption of both compounds were shown to arise from the transitions from HOMO (π) and HOMO-3 (n) to the LUMO (π*), and all were mostly localized on the phenazine moiety. The results thus suggest the phenazine unit to be mainly involved in the lowest-energy absorption of the dppz derivatives.

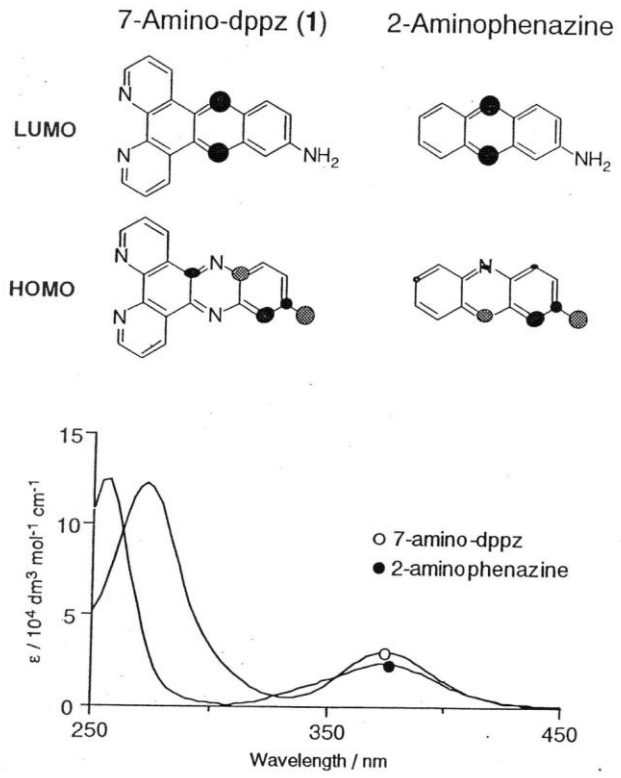


Fig. 7. Calculated molecular orbitals and simulated absorption spectra of 7-amino-dppz and 2-aminophenazine by ZINDO

As shown in Fig. 8, 7-Amino-dipyrido[3,2-*a*:2',3'-*c*]phenazine (7-amino-dppz) has a diimine coordination site, a rigid and extended π-conjugation system, and a reactive amino group within the molecule, and $[Ru(bpy)_2(dppz\text{-}NH_2)]^{2+}$ was synthesized as an useful photosensitizing unit for construction of photoinduced energy transfer systems. Anthraquinone, anthracene, and $[Os(bpy)_3]^{2+}$ having carboxylic acid function were used as the energy-accepting units and were successfully connected to the $[Ru(bpy)_2(dppz\text{-}NH_2)]^{2+}$ through amide bond. Electronic spectral and electrochemical studies of the resultant complexes were carried out, and it was shown that the effective excited energy transfer took place from the Ru(II) polypyridyl center to these units though the electron transfer could not be excluded for those having anthraquinone unit. In the case of the heterodinuclear Ru(II)/Os(II) complex, emission from the Ru(II) polypyridyl center was effectively quenched and that from the Os(II) polypyridyl center was increased compared to the reference Os(II) polyimine complex. Rate of energy transfer from the Ru(II) to the Os(II) polypyridyl center through the dppz-amide connector was estimated to be 1.0 x 10^8 s^{-1} in acetonitrile.[33]

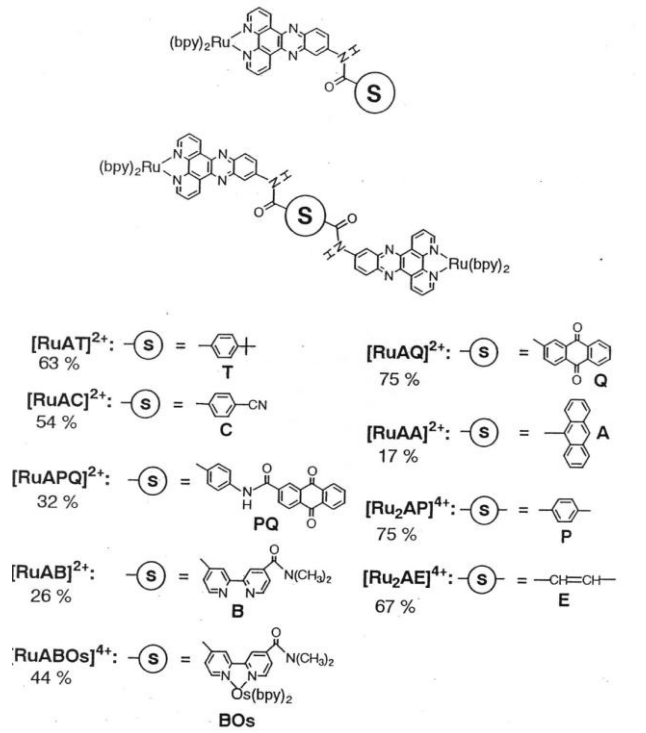


Fig. 8. Amide-type Ru(II) complxes from $[Ru(bpy)_2(dppz\text{-}NH_2)]^{2+}$

As shown in Fig. 9, by introducing benzoyl group to the dppz unit (dppz-CO-ph), dinuclear Ru(II) complexes having the extended π-conjugation system could be easily prepared by one-step reaction, and those complexes gave a novel synthetic strategy for the efficient energy or electron transfer in the molecular system. Our group presented useful and convenient method to prepare dinuclear Ru(II) complexes showing the photoinduced energy transfer or electron transfer from the Ru(II) polypyridyl center to the functional unit attached through imine bond. The emission from the excited Ru(II) center was efficiently quenched through the anthraquinone unit.[34]

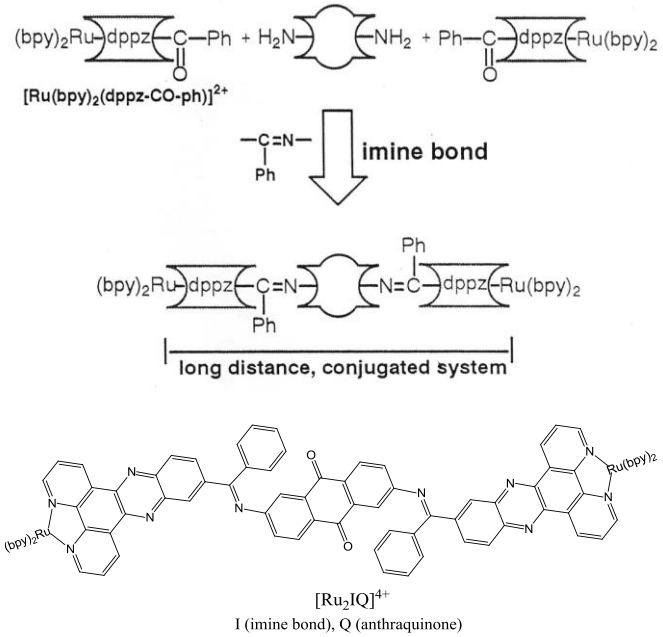


Fig. 9. Synthetic strategy for Imine-type Ru(II) complexes and typical dinuclear Ru(II) complex $[Ru_2IQ]^{4+}$ from $[Ru(bpy)_2(dppz\text{-}CO\text{-}ph)]^{2+}$

As shown in Fig. 10, the synthesized compound (7-(4-mba)dppz) with the methoxy electron-donating group showed a relatively high fluorescence quantum yield (Φ = 0.21 in dichloromethane), comparable to that of 7-amino-dppz, although the fluorescence maximum of the 7-(4-mba)dppz compound that appeared in the wavelength (440 nm) was shorter than that of 7-amino-dppz (517 nm). In addition, according to the investigation of time-resolved emission spectroscopy of the 7-(4-mba)dppz compound and 7-amino-dppz, the fluorescence emission curve of the 7-(4-mba)dppz compound showed a much shorter lifetime (τ = 0.3 ns) as a single component, while that of 7-amino-dppz showed a longer lifetime (τ = 5.6 ns) as a single component. [35]

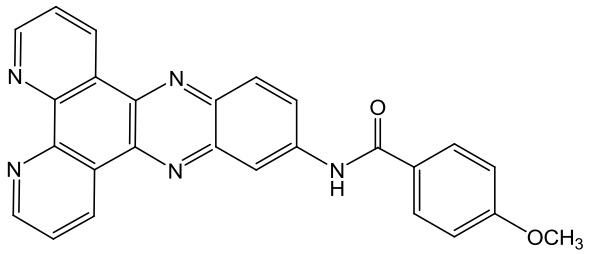


Fig. 10. The structure of fluorescent 7-(4-methoxybenzoylamino) dipyrido [3,2-a:2',3'-c]phenazine (7-(4-mba)dppz) compound

Furthermore, as shown in Fig. 11, when the change of the fluorescence emission intensity was plotted as the complexation ratio related to [Mg^{2+} ion]/[7-(4-mba)dppz] at the emission wavelength 475 nm, it was shown the complex structure of [Mg^{2+} ion]/[7-(4-mba)dppz] = 1 : 2. Also, when it was plotted as the complexation ratio related to [Zn^{2+} ion]/[7-(4-mba)dppz] at the emission wavelength 480 nm, the result was the same as the ratio [Mg^{2+} ion]/[7-(4-mba)dppz] = 1 : 2. Herein, we suggest that 7-(4-mba)dppz can have a potential application for the cation sensor material such as divalent Mg^{2+}, Ni^{2+}, Zn^{2+} and Cu^{2+} metal ions.[36]

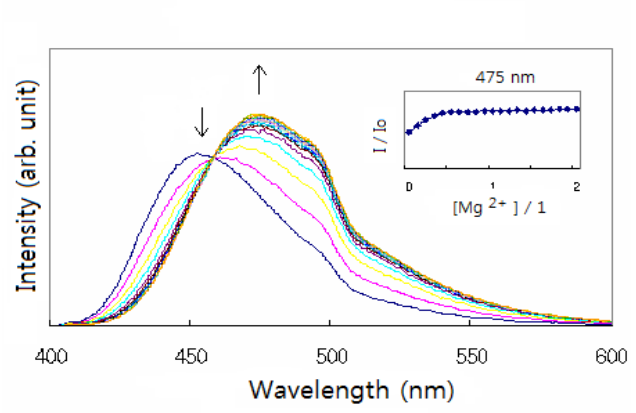


Fig. 11. The fluorescence spectral titration of 6.00 x 10^{-6} mol dm^{-3} 7-(4-mba)dppz by addition of Mg^{2+} ion in acetonitrile at room temperature

CONCLUSION

Polypyridyl compounds are useful molecular units as binding sites and have the photofunctionality such as the fluorescence recognition and/or photo-switching. In addition, the functional unit (-COOH or –NH_2 *etc*) of those compounds renders various building blocks for the construction of long ranged π-conjugation system. In particular, 7-amino-dipyrido[3,2-*a*:2',3'-*c*]phenazine (7-amino-dppz, dppz-NH_2) has a diimine coordination site, a rigid and extended π-conjugation system, and a reactive amino group within the molecule, in which $[Ru(bpy)_2(dppz\text{-}NH_2)]^{2+}$ is synthesized as an useful photosensitizing unit for the photoinduced energy transfer system. In the case of the fluorescent 7-(4-mba)dppz compound, we found that all the complexation ratio were the same as [M^{2+} ion]:[7-(4-mba)dppz] = 1:2 showing a potential application for the cation sensor material such as divalent metal ions.

Received December 19, 2016; Accepted December 27, 2016.

KEY WORDS: Fluorescent polypyridine compound, Bipyridine, DPPZ, Photofuctionality.

ACKNOWLEDGMENTS

This work was supported by Far East University.

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