

S₂- and S₁-states properties of zinc porphyrin polypeptides

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Abstract

S₂- and S₁-states properties of zinc porphyrin polypeptides were investigated using various fast spectroscopic techniques. The B band of zinc porphyrin polypeptides showed broadening and shift to the longer wavelength side with the polymerization degree, indicating the interaction between the porphyrin units. The lifetime of S₂-fluorescence became shorter with increasing the polymerization degree. This phenomenon can be explained by applying the energy gap law to the S₂–S₁ internal conversion. From the picosecond transient absorption measurements, singlet–singlet annihilation was confirmed, indicating the intrachain singlet energy migration in the polypeptides. The annihilation rate became faster with the polymerization degree because of the shorter distance between the zinc porphyrin units of longer polypeptides.

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1. Introduction

Inspired by the natural light-harvesting system, various kinds of porphyrin arrays have been demonstrated [1–26]. Recently developed synthetic methods realize the various porphyrin arrays utilizing the covalent bonds or coordination bonds among the porphyrin units. Polymerization of a monomer bearing a porphyrin unit is also an important method to construct porphyrin arrays, because the polymerization provides the large porphyrin arrays. To date, various polymers bearing porphyrin units in a linear or face-to-face conformation have been reported [14–26]. For construction of the porphyrin arrays aiming at the light-harvesting system, the polymerization method that controls the polymerization degree and sequence precisely will be useful.

Some of the present authors and their co-workers have already synthesized L-lysine-based polypeptides bearing porphyrin pendants (porphyrin polypeptides), in which the central metals can be varied [17–19]. The porphyrin polypeptides are expected to form a helical structure, in which the porphyrin units are aligned

in a favorable conformation for the energy migration. From the results of fast spectroscopic methods, we revealed the singlet energy migration process in the free-base porphyrin polypeptides [26].

It is reported that the S₂ state of the porphyrin is an important precursor of the various reactions [27–31]. For example, Mataga et al. reported the intramolecular electron transfer from the S₂ state of porphyrin [27,28]. Furthermore, contribution of the S₂ state of chlorophyll in the natural photosynthesis system is well known [30–31]. Thus, the polymer-structure dependence of the S₂-state properties is an interesting subject to be cleared.

Herein, we report the S₂- and S₁-states properties of the zinc porphyrin polypeptides (**ZP1**, **ZP2**, **ZP4**, and **ZP8**, Fig. 1) based on various fast spectroscopic techniques. The S₂-state properties dependent on the polymerization degree and the singlet energy migration were observed.

2. Experimental

2.1. Materials

Zinc porphyrin polypeptides were synthesized according to a previously reported procedure [17–19]. All other chemicals were of the best commercial grades available.

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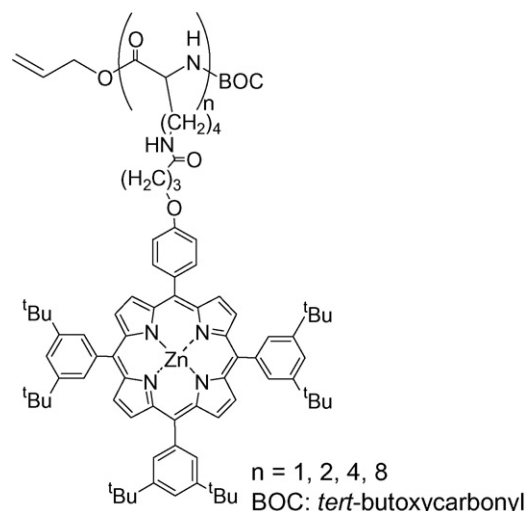


Fig. 1. Molecular structure of zinc porphyrin polypeptides (**ZPn**, $n = 1, 2, 4, 8$).

2.2. Apparatus

Transient absorption spectra in the picosecond region were measured by the pump and probe method employing the second harmonic oscillation (532 nm) of a picosecond Nd:YAG laser (Continuum, RGA69-10LD, fwhm 30 ps, $1\text{--}16\text{ mJ pulse}^{-1}$) as the excitation source. The probe pulse generated by focusing the fundamental light of the Nd:YAG laser on a $\text{D}_2\text{O}:\text{H}_2\text{O}$ cell was detected with a MOS linear image sensor (Hamamatsu Photonics, M2493-40) equipped with a polychromator (Hamamatsu Photonics, C5094) after passing through the sample (Optical path: 2.0 mm).

The time-resolved fluorescence spectra were measured by the single photon counting method using a streakscope (Hamamatsu Photonics, C4334-01) equipped with a polychromator (Acton Research, SpectraPro150). The ultrashort laser pulse was generated by a Ti:sapphire laser (Spectra-Physics, Tsunami 3941-M1BB, fwhm 80 fs) pumped with a diode-pumped solid state laser (Spectra-Physics, Millennia VIIIs). For excitation of the sample, the output of the Ti:sapphire laser was converted to the second harmonic oscillation (430 nm) with a harmonic generator (Spectra-Physics, GWU-23FL).

The fluorescence lifetime in the sub-picosecond regime was measured using fluorescence up-conversion method. The second harmonic oscillation (420 nm) of the output of the femtosecond laser (840 nm) was used to excite the sample in a cell with a 1.0 mm optical path length. The residual fundamental and the fluorescence were focused in a BBO type I crystal to generate a sum-frequency oscillation, which was detected by a photomultiplier tube (Hamamatsu Photonics, H8259) and the photon counter (Stanford Research Systems, SR400) after passing through the monochromator (Nikon G250). The cross correlation time of the apparatus was 200 fs fwhm.

The steady state absorption and fluorescence spectra were measured using a Shimadzu UV-3100PC and a Hitachi 850, respectively.

Spectroscopic measurements were carried out after the bubbling with Ar gas.

3. Results and discussion

3.1. Steady state absorption of zinc porphyrin polypeptides

The absorption spectrum of **ZP8** showed peaks at 429.3 nm (B band, $\lambda_{\text{abs}}(\text{B})$) and 556.2 and 600.0 nm (Q bands, $\lambda_{\text{abs}}(\text{Q})$). For comparison, the absorption peak positions of other compounds are listed in Table 1. It was revealed that the peak position of the B band shifted to the longer wavelength side with the number of the porphyrin unit in the polypeptides. In addition, B band showed broadening. The distance between the porphyrin units is expected to become shorter with the degree of the polymerization. Thus, the red shift and broadening of the B band can be attributed to the exciton coupling of the transition dipole moment of the porphyrin units. From the observed shift and broadening, the dipole moments of the porphyrin rings are considered to be in the oblique configuration. This consideration is supported by the MM calculation, from which the oblique configuration of porphyrins due to α -helix structure of the L-lysine backbone is indicated [26]. Actually, **ZP8** showed the induced CD pattern in the B band region supporting the α -helix structure.

3.2. S_2 -fluorescence of zinc porphyrin polypeptides

Zinc porphyrin shows S_2 -fluorescence while free base porphyrin does not show clearly. In Fig. 2, S_2 -fluorescence spectrum of **ZP8** is shown. In Table 1, fluorescence bands due to S_2 - and S_1 -fluorescence ($\lambda_f(S_2)$ and $\lambda_f(S_1)$, respectively) are also summarized. Both fluorescence bands show shift to the longer wavelength side in accordance with the absorption peak shift. The S_2 -fluorescence of **ZP8** decayed according to the single exponential function, from which the lifetime of the S_2 -fluorescence ($\tau(S_2)$) of **ZP8** was estimated to be 0.84 ps (inset of Fig. 2), which is much shorter than that of **ZP1** (1.49 ps). As summarized in Table 1, the $\tau(S_2)$ became shorter as the polymerization degree. Furthermore, the S_2 -fluorescence intensity of longer polypeptides is smaller than that of **ZP1**: The relative intensities of S_2 -fluorescence band are 1.0, 0.23, 0.38, and 0.38 for **ZP1**, **ZP2**, **ZP4**, and **ZP8**, respectively. Since the quantum yields of S_2 -fluorescence of zinc porphyrins are reported to be 0.0037–0.016 [32–35], the quantum yields of S_2 -fluorescence

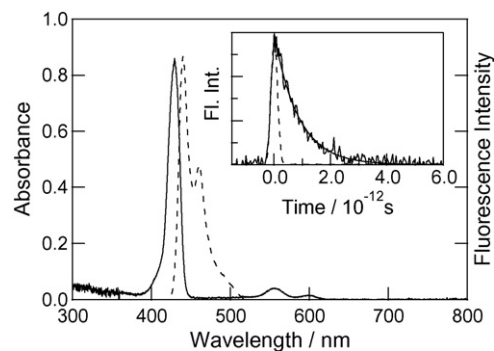


Fig. 2. Steady state absorption (solid line) and S_2 -fluorescence spectra (dashed line) of **ZP8** in toluene. Inset: Decay profile of S_2 -fluorescence (465 nm) of **ZP8**. Solid and dashed lines are fitted curve and irf, respectively.

Table 1
Absorption and fluorescence properties of zinc porphyrin polypeptides in toluene

	$\lambda_{\text{abs}}(\text{B})$ (nm) (fwhm) ^a	$\lambda_{\text{f}}(\text{S}_2)$ (nm)	$\tau(\text{S}_2)$ (ps)	$\lambda_{\text{abs}}(\text{Q})$ (nm)	$\lambda_{\text{f}}(\text{S}_1)$ (nm)	$\Phi_{\text{f}}(\text{S}_1)$ ^b	$\tau(\text{S}_1)$ (ns) ^c
ZP1	425.8 (663)	436, 460	1.49	552.0, 592.6	600, 650	0.050	1.99
ZP2	428.4 (848)	438, 462	1.03	556.1, 599.3	606, 655	0.050	1.97
ZP4	429.1 (854)	438, 462	0.91	557.2, 600.8	607, 656	0.058	2.14
ZP8	429.3 (926)	440, 461	0.84	556.2, 600.0	608, 656	0.052	2.18

^a In cm^{-1} .

^b Estimated using zinc tetraphenylporphyrin in toluene as a standard ($\Phi_{\text{f}}(\text{S}_1) = 0.040$) [40].

^c Lifetime measurements were made at excitation densities low enough to eliminate singlet–singlet annihilation.

of **ZP2**, **ZP4**, and **ZP8** are in the order of 10^{-3} to 10^{-4} . The smaller S_2 -fluorescence quantum yields of **ZP2**, **ZP4**, and **ZP8** than **ZP1** can be attributed to the smaller oscillator strength of oligomers due to the exciton coupling.

The main deactivation process of the S_2 state of zinc porphyrin should be the internal conversion from the S_2 to the S_1 state, since the quantum yield of the S_2 -fluorescence is quite small as mentioned above. Furthermore, other deactivation channels, such as charge transfer and intersystem crossing, can be negligible because any transient species indicating such processes was not observed during the femtosecond laser flash photolysis employing 400 and 550 nm laser pulses for the S_2 and S_1 state excitation, respectively. In the presence of the exciton coupling, the internal conversion process can be attributed to the deactivation from the lower exciton state of the S_2 to the upper exciton state of the S_1 as indicated in Fig. 3. The rate of the internal conversion (k_{IC}) is usually governed by the energy gap law (Eq. (1)) [36],

$$k_{\text{IC}} \propto \exp(-\beta \Delta E) \quad (1)$$

where β and ΔE are a constant and an energy gap between the states, respectively. In the case of **ZP1**, the energy gap between the S_2 and S_1 states ($\Delta E(\text{S}_2-\text{S}_1)$) can be evaluated from the state energies estimated from the corresponding fluorescence peaks. For **ZPn** ($n = 2, 4, 8$), the energy gap between the lower

exciton state of the S_2 and the upper exciton state of the S_1 can be evaluated by taking the fluorescence peak shift into the consideration as indicated in Fig. 3. The linear relation between the $\ln(k_{\text{IC}})$ values and the $\Delta E(\text{S}_2-\text{S}_1)$ values was confirmed as shown in Fig. 4. The β value was estimated to be 11.0 eV^{-1} . Thus, it is suggested that the present S_2 – S_1 internal conversion of zinc porphyrin polypeptides is governed by the energy gap law.

Song et al. reported that the S_2 state lifetimes of *meso*, *meso*-linked zinc porphyrin dimer, trimer, and tetramer were largely reduced because of participation of exciton split B band as a ladder type deactivation channel [7]. Compared with the *meso*, *meso*-linked zinc porphyrin arrays, the exciton coupling in the present zinc porphyrin polypeptides is rather small. Furthermore, other deactivation channels such as charge transfer and intersystem crossing is negligible. Thus, the S_2 state energy decayed according to the energy gap law.

The estimated β value is about a half of the previously reported value for tetraphenylporphyrins bearing various metal ions such as Al, Zn, and Cd [35]. The difference in the central metal will cause changes in the deactivation processes of the excited state, for example, the heavy atom effect is well known. Therefore, the previously reported value seems to include not only the effect of ΔE but also other factors. On the other hand, the present estimated β value is similar to that reported for azulene derivatives [37], supporting above consideration.

3.3. S_1 -state properties of zinc porphyrin polypeptides

The S_1 -fluorescence properties (fluorescence quantum yield ($\Phi_{\text{f}}(\text{S}_1)$) and lifetime ($\tau(\text{S}_1)$)) of zinc porphyrin polypeptides do not show significant change with the polymerization degree

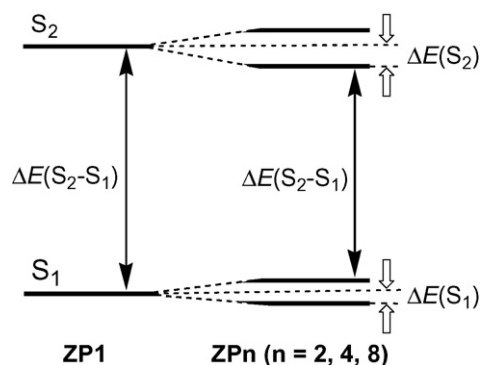


Fig. 3. Schematic energy diagram for S_2 and S_1 states of **ZP1** and **ZPn** ($n = 2, 4, 8$). The energy level of the lower exciton state of S_2 -excited **ZPn** can be estimated from the peak position of the S_2 -fluorescence. Energy difference between the S_2 state of **ZP1** and the lower exciton state of S_2 -excited **ZPn** is $\Delta E(\text{S}_2)$ in this figure. Similarly, energy difference between the S_1 state of **ZP1** and the lower exciton state of S_1 -excited **ZPn** is $\Delta E(\text{S}_1)$. $\Delta E(\text{S}_2-\text{S}_1)$ of **ZPn** can be calculated from $\Delta E(\text{S}_2)$ and $\Delta E(\text{S}_1)$, because the difference between the S_1 state of **ZP1** and the upper exciton state of S_1 -excited **ZPn** is equal to $\Delta E(\text{S}_1)$.

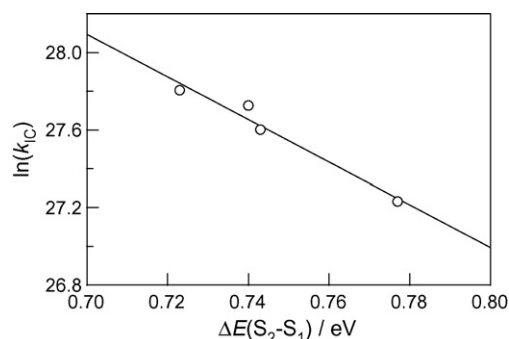


Fig. 4. Relation between $\ln(k_{\text{IC}})$ and S_2 – S_1 energy gap.

when compared to the S_2 -fluorescence properties, probably due to the weak excitonic coupling of the Q bands with smaller transition dipole moment.

Further S_1 -state properties of the porphyrin polypeptides were investigated using picosecond laser flash photolysis. The transient absorption spectrum of **ZP8** at 20 ps after the laser flash (532 nm, fwhm 30 ps) can be attributed to the singlet excited state of the **ZP8**, because a large absorption band around 455 nm and bleaching due to Q-band (556 nm) are characteristic features of the porphyrin units in the singlet excited state. As shown in the inset of Fig. 5(a), the singlet excited state of **ZP8** decayed according to the two steps decay in the picosecond region. The slowly decaying component can be attributed to the decay of the singlet excited state by intersystem crossing, fluorescence, and internal conversion processes, because the deactivation rate agreed well with the S_1 -fluorescence lifetime ($\tau(S_1) = 2.18$ ns). The deactivation by these processes does not cause the substantial decay in the 0–600 ps time region. On the other hand, the decay rate constant of the fast decaying part was $(1.8 \pm 0.1) \times 10^{10} \text{ s}^{-1}$. The amplitude of the fast decaying component depends on the laser fluence, i.e., the fast decaying component almost vanished when the excitation laser fluence was low (inset of Fig. 5(a)). From these findings, the fast decaying component can be attributed to the deactivation of the singlet excited state by the intramolecular singlet–singlet annihilation process resulted from the singlet energy migration among the porphyrin units [9–11,15,26,38,39]. This assignment is supported by the fact that the fast decaying component was not observed in the kinetic trace of $\Delta O.D.$ at 455 nm of **ZP1** (Fig. 5(b)). In the case of **ZP4**, the singlet–singlet annihilation was also confirmed (Fig. 5(b)), although the decay

rate $((1.0 \pm 0.2) \times 10^{10} \text{ s}^{-1})$ was slower than that of **ZP8**. This finding indicates that the singlet–singlet annihilation becomes less efficient when the distance between the porphyrin units becomes large. Furthermore, the rate-determining step of the present deactivation can be attributed to the singlet energy migration, because the rate depends on the distance between the chromophores. Further slower singlet–singlet annihilation rate $((3.2 \pm 0.4) \times 10^9 \text{ s}^{-1})$ was estimated for **ZP2**, which has a longer porphyrin–porphyrin distance. Under the present experimental condition, the influence of the number of the excited porphyrins in one molecule can be excluded from the discussion on the annihilation process, since the averaged number of the excited porphyrins in one molecule at immediately after the laser irradiation was calculated to be 1.6 from the kinetic trace of **ZP8** (Fig. 5(a)).

In the previous paper, we reported the excited state properties of free-base porphyrin polypeptides [26]. The singlet–singlet annihilation process was similarly observed with the free-base porphyrin polypeptides, although the rate constants were smaller than those of the present zinc porphyrin polypeptides, i.e., observed rates were $(1.1 \pm 0.1) \times 10^{10}$, $(7.3 \pm 0.4) \times 10^9$, and $(3.5 \pm 0.2) \times 10^8 \text{ s}^{-1}$ for the octamer, tetramer, and dimer of free-base porphyrin polypeptides, respectively. As a factor, which enhances the singlet energy migration in the porphyrin polypeptides, stronger interaction between porphyrin pigments of the zinc porphyrin polypeptides can be pointed out. Because the absorption band of the zinc porphyrin polypeptides show a shift due to the exciton coupling (Table 1), on the other hand the free-base porphyrin polypeptides did not show such spectral change. The metallation of the porphyrin units enhanced the interaction between the porphyrin units. These facts support the fast singlet energy migration in the present zinc porphyrin polypeptides.

4. Conclusion

In the present paper, the S_2 - and S_1 -states properties of the zinc porphyrin polypeptides were investigated using various fast spectroscopic techniques. The lifetime of S_2 -fluorescence became shorter with increasing the polymerization degree. This phenomenon can be explained by applying the energy gap law to the S_2 – S_1 internal conversion. From the picosecond transient absorption measurements, singlet–singlet annihilation was confirmed, indicating the intrachain singlet energy migration in the polypeptides. The annihilation rate became faster with the polymerization degree because of the close distance between the zinc porphyrin units. Therefore, the exciton coupling among the porphyrin units plays an important role in the deactivation process of the S_2 - and S_1 -states.

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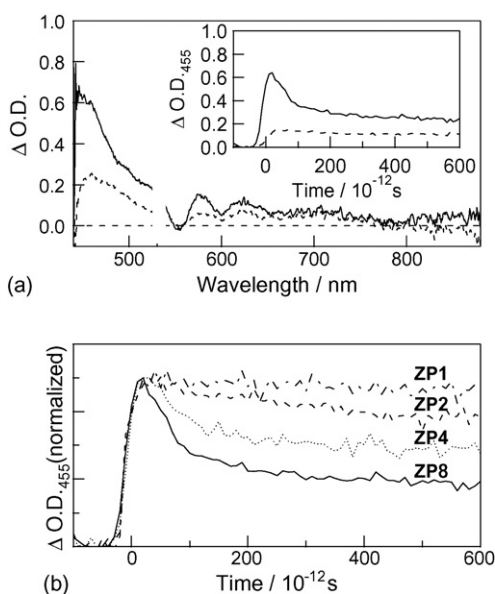


Fig. 5. (a) Transient absorption spectra observed at 20 (solid line) and 500 ps (broken line) after the laser flash (532 nm, fwhm 30 ps, 16 mJ pulse^{-1}) during the laser flash photolysis of **ZP8** in toluene. Inset: kinetic traces of $\Delta O.D.$ at 455 nm (laser fluence: (solid line) 16 mJ pulse^{-1} , (broken line) 1 mJ pulse^{-1}). (b) Kinetic traces of **ZP1**, **ZP2**, **ZP4**, and **ZP8** at 455 nm. Absorbance of the samples was matched at the excitation wavelength (532 nm).

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