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Effect of aggregation on nonlinear optical properties of a naphthalocyanine

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Abstract

The magnitude and dynamic response of the third-order optical nonlinearity of silicon 2,3-naphthalocyanine bis(trihexylsilyloxide) (SiNc) were measured by femtosecond degenerate four-wave mixing (DFWM) technique under resonant conditions. The temporal profiles of the DFWM signal were obtained with a time resolution of 0.3 ps (FWHM), and were found to consist of at least two components, i.e., the coherent instantaneous nonlinear response (electronic response) and the slow response due to the excited state population grating. The present SiNc in neat film shows small red-shift compared to monomer absorption because of the large distance between adjacent naphthalocyanine rings, which results in weak delocalization of Frenkel exciton in the aggregated state. However, time-resolved DFWM measurements indicate that the SiNc films show fast nonlinear optical response and large third-order nonlinear susceptibility. The effective $\chi_e^{(3)}$ value of the neat SiNc film was evaluated to be as high as 4×10^{-7} esu, and the figure of merit of third-order nonlinearity F ($F = \chi^{(3)}/\alpha$), was calculated to be about 4.1×10^{-13} esu cm.

Keywords: Degenerate four-wave mixing; Aggregation; Third-order nonlinear susceptibility; Naphthalocyanine

1. Introduction

Phthalocyanines are one of the major types of tetrapyrrole derivatives showing a wide range of applications in materials science, medicine and catalysis [1–4]. Over the past two decades, phthalocyanines have been extensively studied as an important class of third-order nonlinear optical materials because of their extensively delocalized two-dimensional 18π-electron system, their structural flexibility, their exceptionally high thermal and chemical stability, and their potential for use in photonic applications such as optical swiching and optical limiting [5–10]. Owing to the extended π system, it is well known that these macrocyclic compounds exhibit a high aggregation tendency forming dimeric and oligomeric species in solutions [11–13]. It has been shown that this molecular association greatly influences the intrinsic nature of macrocycles including their spectroscopic, photophysical, electrochemical and nonlinear optical properties [14–18]. A substantial number of investigations have been focussed on the aggregation behavior of substituted phthalocyanines in various solvent systems in which the aggregation number and some thermodynamic parameters have been determined [19–21].

Naphthalocyanines are phthalocyanine-related compounds with further extended π-electron delocalization, which are expected to have an even higher aggregation tendency. Studies in this area, however, have been extremely rare [22]. Silicon 2,3-naphthalocyanine bis(trihexylsilyloxide) (hereafter referred to as SiNc) was first synthesized by Wheeler et al. [23] in 1984 and has been studied for various applications. The structural formula of SiNc is shown in Fig. 1. The bulky trialkylsiloxy groups on the central Si atom provide SiNc relatively high solubility in common organic solvents. SiNc exhibits a strong absorption in the near-infrared region, with a molar extinction coefficient as high as *ca.* $4 \times 10^5\,\mathrm{mol^{-1}\,l\,cm^{-1}}$ at the Q-band absorption peak [23]. In the present study, the resonant third-order opti-

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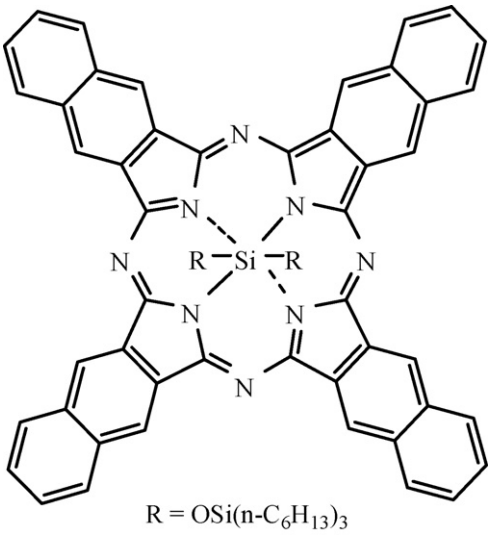

Fig. 1. Structural formula of silicon 2,3-naphthalocyanine bis(trihexylsilyloxide).

cal nonlinearities of SiNc both in solution and in solid films were measured by femtosecond degenerate four-wave mixing (DFWM). The probable decay mechanisms of DFWM response and the effect of aggregation on third-order optical nonlinearities of SiNc were discussed.

2. Experimental

SiNc was purchased from Sigma–Aldrich and used as received. SiNc was dissolved in tetrahydrofuran (THF) at a concentration of 1.0×10^{-5} mol l^{-1} for measurements. Poly(methyl methacrylate) (PMMA) films doped at various concentrations of SiNc and a neat SiNc film were prepared by dip-coating tetrahydrofuran (THF) solutions onto slide glass plates. Because of excellent solubility for SiNc in THF solvent, all the films fabricated have high optical qualities with good surface flatness, relative homogeneous thickness and low light scattering for DFWM measurement. Thickness of samples was measured to be *ca.*130–200 nm for the SiNc/PMMA films and *ca.* 50 nm for SiNc neat film by a surface profiler (ULVAC, Dektak^3), respectively.

Absorption spectra of all samples were measured by a UV–vis spectrophotometer (Jasco, V550). Time-resolved fluorescence lifetime measurements were carried out using a combination of a Ti:sapphire laser (Spectra-Physics, Tsunami) and a streak camera (Hamamatsu, Streak Scope C4334).

Third-order nonlinear susceptibility $\chi^{(3)}$ was measured at the Q-band absorption peak of each sample using the forward DFWM technique. The system used in our experimental study of the DFWM is the same as that described in the previous papers [24,25]. The folded box-CARS type geometry with three linearly polarized beams of the fundamental output of a Ti:sapphire regenerative amplifier laser (Spectra-Physics, Spitfire, 130 fs, 1 kHz, 1 W) was used for the DFWM experiment. Two of the three forward beams are used for the generation of a refractive index grating in sample, while the third beam is used for the detection of this grating, i.e., the light-induced change of the refractive index. The laser beam was largely attenuated by ND filters in order to avoid saturation. The energies of beam 1 and beam 2 were less than 100 nJ/pulse, and that of beam 3 was less than 10 nJ/pulse. The intensity of the laser radiation on samples was about 1.0 GW/cm^2. A half-wave plate was positioned in beam 2 and rotated 45° in order to measure $\chi^{(3)}_{xyxy}$ (perpendicular configuration). The sample was simultaneously irradiated with beams 1 and 2, and then irradiated with beam 3 after a suitable delay time. The three collinear beams were focused onto the sample by a lens with a 500 mm focal length. Beam 4, the produced DFWM signal, was detected by a CCD after passing through a monochromator. The output of the CCD was acquired using a personal computer. The time resolution of the system was *ca*. 0.3 ps (FWHM). The intensity of the DFWM signal due to the solvent and quartz cuvette was completely negligible. The experimental uncertainty was estimated to be within ±20%. The dependence of the DFWM signal intensity on the laser power was measured. The power dependence was almost cubic which proves no saturation under the measured conditions.

The effective third-order nonlinear susceptibility, $\chi^{(3)}$ was calculated by comparison with the signal of a reference sample CCl_4 ($\chi^{(3)} = 4.01 \times 10^{-14}$ esu) [26] measured under the same conditions, and was corrected for the absorption losses according to the following relationship:

$$\chi^{(3)}_{\text{sample}} = \left(\frac{n_{\text{sample}}}{n_{\text{ref}}}\right)^2 \left(\frac{I_{\text{sample}}}{I_{\text{ref}}}\right)^{1/2} \left(\frac{L_{\text{ref}}}{L_{\text{sample}}}\right) \times \alpha L_{\text{sample}} \times \exp\left(\frac{\alpha L_{\text{sample}}}{2}\right) [1 - \exp(-\alpha L_{\text{sample}})]^{-1} \chi^{(3)}_{\text{ref}} \quad (1)$$

where I is the DFWM signal intensity, α is the linear absorption coefficient, n is refractive index, and L is interaction length. The subscript ref refers to the reference. Instead of using CS_2, we used CCl_4 as a reference. The reason is that the molecular anisotropy of CS_2 makes it undergo molecular reorientation under the action of the laser pulses and give rise to orientation nonlinearity. The rise and fall times for this orientational contribution to DFWM signal are 200 and >600 fs, respectively [27,28]. Therefore, it is not appropriate in femtosecond regime to use the known effective $\chi^{(3)}$ value of CS_2 as the reference to evaluate the effective $\chi^{(3)}$ value of sample. On the other hand, the isotropic structure of CCl_4 and its instantaneous response make CCl_4 an excellent reference material for femtosecond DFWM.

The second-order molecular hyperpolarizability γ is related to $\chi^{(3)}$ by

$$\gamma = \frac{\chi^{(3)}}{T^4 N_0} \quad (2)$$

where N_0 is the number density of the dye molecules, and T is the local field factor. For the calculation, the formula for the spherical molecules, $T = (n^2_{\text{sample}} + 2)/3$, was used. The $\chi^{(3)}$ contribution from the solvent is negligible in comparison to the solute. In our calculations, we consider only the electronic component to the DFWM signal, therefore the evaluated $\chi^{(3)}$ and γ are electronic and they will be represented by $\chi^{(3)}_{\text{e}}$ and γ_{e}, respectively.

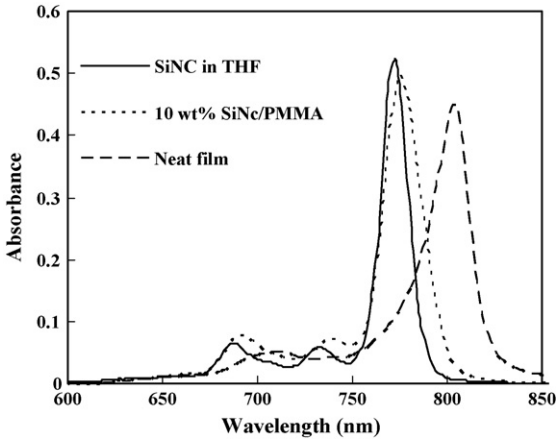

Fig. 2. Absorption spectra of the SiNc in THF solution (solid line), a 10 wt% SiNc/PMMA film (dotted line) and a neat SiNc film (broke line).

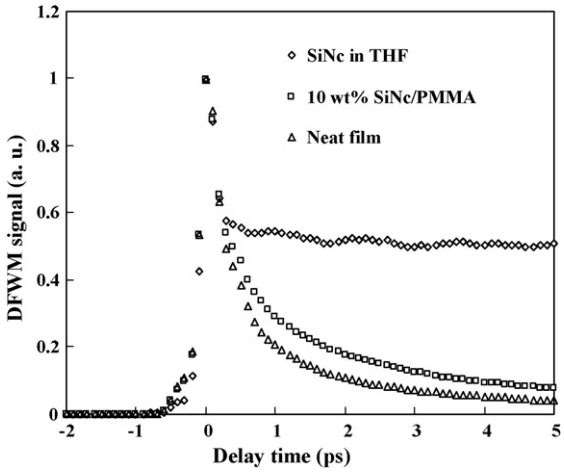

Fig. 3. Temporal profiles of DFWM signal of the SiNc in THF solution, a 10 wt% SiNc/PMMA film and a neat SiNc film.

3. Results and discussion

3.1. Absorption spectra

Fig. 2 shows the near-infrared absorption spectra of SiNc in THF solution, 10 wt% SiNc/PMMA film and a neat SiNc film. SiNc in THF solution shows a characteristic monomeric absorption of naphthalocyanine, with the Q-band absorption maximum (λ_{max}) at 772 nm assigned to the $S_0(0)$–$S_1(0)$ transition [23], and full width at half maximum (FWHM) is *ca.* 17 nm. The 10 wt% SiNc/PMMA film exhibit a broad and red-shifted absorption spectra (λ_{max}: 775 nm, FWHM: *ca.* 33 nm) compared with the monomeric spectrum. The Q-band absorption spectrum of the neat SiNc film is slightly broad (FWHM: *ca.* 25 nm) and shows a *ca.* 32 nm red shift from the monomeric Q-band absorption maximum. In the present study, it was found that films with concentration of SiNc higher than 20 wt% have similar absorption maxima near 802 nm, and films with concentration of SiNc larger than 40 wt% have absorption spectra very similar to that of the neat film. It suggested that SiNc molecules have similar packing arrangement resembling the slipped stacked structure of the J-type aggregate in these films [29]. It seems that molecular stacking of SiNc in the films is very sensitive to the concentration of SiNc, and aggregation degree becomes large as the concentration of SiNc increases. Using the exciton model, the red-shifted solid-state absorption spectra have been well elucidated for a series of axially substituted naphthalocyanines which have slipped stacked molecular arrangement in thin solid films [30].

3.2. Temporal profile of DFWM signal

The temporal profiles of DFWM signal were measured as a function of the delay time of beam 3. The results are illustrated in Fig. 3. Each temporal profile has at least two components, a rapidly decaying component and a slow component whose decay rate varies with concentration of SiNc. The fast component is primarily determined by the laser pulse, and can be attributed to the contribution from the electronic response. The slow component appears to be associated with the excited state population grating. Compared with the DFWM response of SiNc in THF, the DFWM response of SiNc in films decays very fast. This is because the DFWM intensity due to the population grating is expected to be proportional to the square of the concentration of SiNc dye in an excited state, and the excited SiNc monomer in the films will be quenched by dimer or aggregates.

3.3. Simulation of temporal profile of DFWM signal

To analyze the temporal profiles of a DFWM signal of SiNc in THF solution and in films, they were simulated. The observed DFWM temporal profile $I(t)$ is assumed to be expressed by the following convolution integral:

$$I(t) = \int_0^t L(t')R(t-t')\,dt' \tag{3}$$

where L(t) is the temporal profile of the laser and $R(t)$ is the DFWM response of samples irradiated by a laser with an ideal delta function shape pulse. For SiNc in THF, $R(t)$ is assumed to be a biexponential function

$$R(t) = \alpha_1 \exp\left(-\frac{t}{\tau_1}\right) + \alpha_2 \exp\left(-\frac{t}{\tau_2}\right) \tag{4}$$

The slow response of a DFWM signal of the films was not exponential for SiNc in the films. The excited monomer will be quenched by dimer or aggregates. Förster's theory [31] states that the temporal behavior of the concentration of excited donor molecules quenched by energy acceptor molecules randomly distributed in a three-dimensional space can be expressed by the following equation:

$$\rho(t) = \exp\left[-\frac{t}{\tau_D} - \gamma_A\left(\frac{t}{\tau_D}\right)^{1/2}\right] \tag{5}$$

where τ_D is the lifetime of the donor without an acceptor, γ_A is a constant related to energy transfer rate.

The DFWM signal intensity of a population grating is proportional to the square of the concentration of excited molecules.

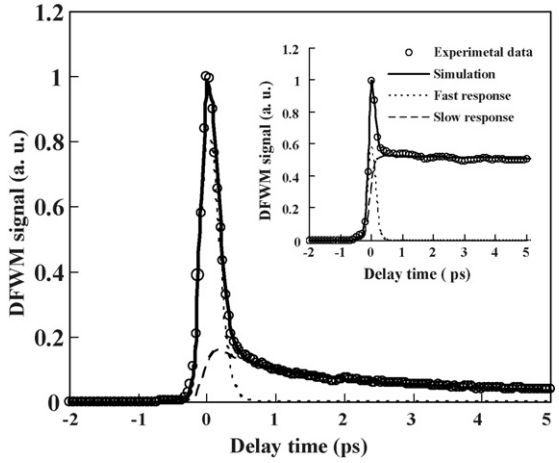

Fig. 4. Simulation for a temporal profile of DFWM signal of a neat SiNc film by assuming Förster type energy transfer, open circles, solid line, dotted line and broke line represent experimental data, simulation, fast component and slow component, respectively. Inset: a simulation for a temporal profile of DFWM signal of SiNc in THF solution by a biexponential function.

Therefore, $R(t)$ for SiNc in the PMMA films and SiNc neat film can be assumed to be the sum of an exponential function (electronic component) and a slow response (population grating), and expressed as follows:

$$R(t) = \alpha_1 \exp\left(-\frac{t}{\tau_1}\right) + \alpha_2 \rho(t)^2 = \alpha_1 \exp\left(-\frac{t}{\tau_1}\right) + \alpha_2 \exp\left[-\frac{2t}{\tau_D} - 2\gamma_A\left(\frac{t}{\tau_D}\right)^{1/2}\right] \quad (6)$$

All the parameters in Eqs. (4) and (6) and the time difference between the experimental and simulated DFWM temporal profiles, Δt, were determined using a nonlinear, least-squares iterative convolution method based on the Marquardt algorithm [32,33]. The DFWM temporal profile of a glass plate is used as $L(t)$.

Fig. 4 shows the simulation of temporal profiles for the DFWM signal of SiNc in THF (inset of Fig. 4) and a neat SiNc film, which were simulated in accordance with above refered method by a biexponential function and assuming Förster type energy transfer, respectively. It was found that the simulated curve reproduced the experimental values very well. The observed decay of DFWM signals consist of at least two components, i.e., fast component induced by the contribution from the coherent instantaneous nonlinear response (electronic response) and slow component due to the contribution from the population grating of excited state. The contribution from the fast component becomes dominant for the DFWM signal of the neat SiNc film because of aggregation effect on the DFWM response.

3.4. Time-resolved fluorescence

Our time-resolved fluorescence measurements for SiNc in THF solution and in solid films reveal that the fluorescence lifetimes of SiNc in solid films are much shorter than that of the SiNc monomer, as shown in Fig. 5, indicating that nonradiative processes are dominant in solid films. Sakakibara et al. [34] reported that solid films of magnesium, chloroaluminum, and metal-free phthalocyanines have much smaller fluorescence quantum yields (10^{-4} to 10^{-5}) than the corresponding monomers (>0.5) due to large intermolecular interactions. Within nonradiative decay processes, exciton coupling and energy transfer between SiNc molecules probably account for the fast decay of the S_1 state. Since the measured DFWM response showed very little dependence on the laser intensity, nonexponential bimolecular exciton–exciton annihilation is considered to be less important in the decay process. This may be due to the fact that the large out-of-plane substituents induced strong steric repulsion between the SiNc molecules obstructs sufficient exciton motion which is a prerequisite prior to exciton–exciton annihilation. Therefore, we considered that, for the films with low SiNc concentration,

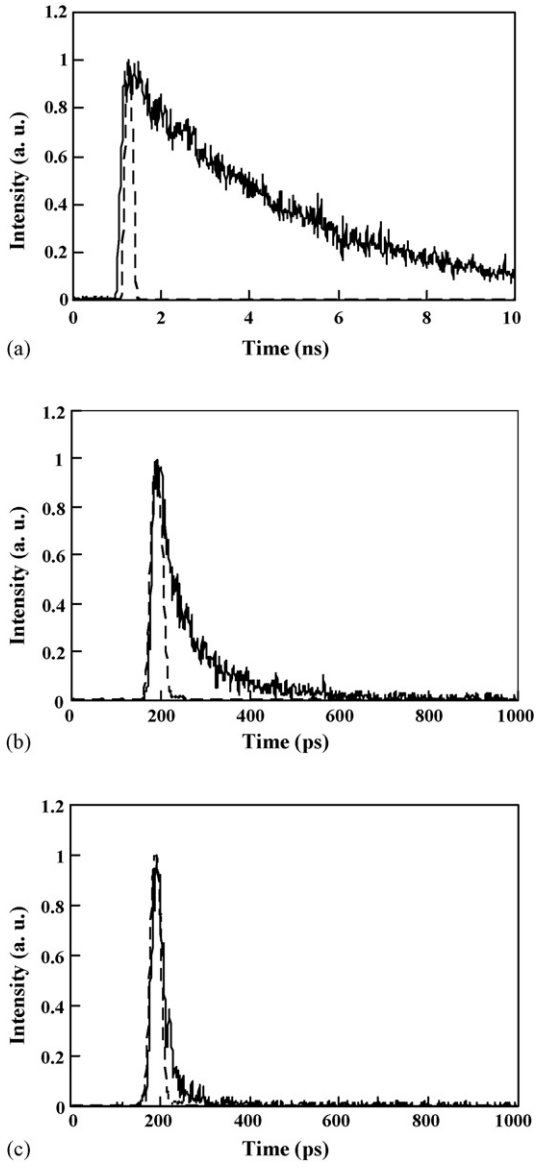

Fig. 5. Fluorescence decay curves of SiNc in different media. (a) SiNc in THF solution; (b) a 10 wt% SiNc/PMMA film; (c) a neat SiNc film. Excitation wavelength was 400 nm. Broken lines represent the response function to laser excitation pulse.

the observed fast relaxation of the DFWM response is due to the efficient excitation energy transfer from the S_1 exiton of SiNc monomer to dimers or higher aggregates. While, for the films with high SiNc concentration, which primarily consist of J-type aggregates, energy transfer from site to site within the J-type aggregates gives rise to the shortening of the S_1 state lifetime.

3.5. Third-order nonlinear susceptibility and molecular hyperpolarizability

We evaluated $\chi_e^{(3)}$ by putting the intensity of the fast component calculated by the corresponding simulation into the Eq. (1) as I_{sample}. Then, the γ_e was calculated using Eq. (2). The values of the resonant γ_e was determined to be about 1.6×10^{-28} esu for SiNc in THF solution. The electronic component of the nonlinear susceptibilities of the SiNc films were also calculated using Eq. (1) assuming that the refractive index of thin films is same as that of PMMA ($n = 1.49$). It was found that $\chi_e^{(3)}$ was further enhanced in the films with more aggregated molecular stacking. The $\chi_e^{(3)}$ of the neat SiNc film (4.0×10^{-7} esu) was approximately 30 times greater than that of a 10 wt% SiNc/PMMA film ($\chi^{(3)} = 1.3 \times 10^{-8}$ esu). In addition to the effect of concentration and resonance on third-order nonlinear susceptibility, the exceptionally enhanced $\chi_e^{(3)}$ can be considered to be induced from the J-type aggregates formed in the neat film. Furthermore, the figure of merit of third-order nonlinearity F ($F = \chi^{(3)}/\alpha$), was evaluated to be about 4.1×10^{-13} esu cm.

Fu et al. [35] have studied the third-order optical nonlinearities of sulfonated zinc and copper 2,3-naphthalocyanines (ZnNcS and CuNcS), metal-free 5,9,14,18,23,27,32,36-octabutoxy-2,3-naphthalocyanine, and its zinc and copper complexes (Nc–BuO, ZnNc–BuO and CuNc–BuO) by DFWM measurement in the previous paper. The $\chi_e^{(3)}$ value of 20 wt% ZnNc–BuO/PMMA film was determined to be 8.9×10^{-9} esu, which was about two times greater than those of Nc–BuO and CuNc–BuO, and 15 times greater than that of ZnNcS due to resonance enhancement. They have also reported the third-order optical nonlinearities of a silicon 2,3-naphthalocyanine dioctyloxide [36], which has similar structure to SiNc of the present study, and the $\chi_e^{(3)}$ value of its polystyrene films was determined to be on the order of 10^{-9} esu. Although the polystyrene film containing silicon 2,3-naphthalocyanine dioctyloxide (30 wt%) showed a larger red-shift, its third-order nonlinear susceptibility was much samller than that of present neat SiNc film due to broad absorption band and poor solubility.

Nalwa and Kobayashi [37] reported a $\chi^{(3)}$ value of 5.0×10^{-7} esu at 800 nm from the DFWM technique for the thin films of a silicon naphthalocyanine derivative. However, the dynamical response of the derivative was not mentioned, presumably its $\chi^{(3)}$ value involves a contribution of population grating.

Previous experimental and theoretical works have shown that molecular J-aggregates are self-organized systems that combine a strong third-order optical susceptibility in the exciton absorption band (J-band) with a short response time [38–42]. Although enhancement of $\chi^{(3)}$ in phase II, which has red-shifted molecular stacking similar to the J-type aggregates, has been reported in evaporated and spin-coated phthalocyanine films [43,44], this enhancement in naphthalocyanine films has been seldom reported. The enhancement of the nonlinear optical properties results from the delocalization of Frenkel exciton over the aggregates [42]. Considering the extent of delocalization of Frenkel exciton is determined by the largeness of intermolecular dipole–dipole interaction which has a strong dependence on intermolecular distance, the present SiNc in neat film shows small red-shift compared to absorption band of monomer because of large distance between adjacent naphthalocyanine rings. Katayose et al. [30] had reported the same result and they concluded that the extent of shift significantly increases with decreasing alkyl length of axial substituents for the [bis(trialkylsiloxy)]silicon tetrakis(alkylthio)-2,3-naphthalocyanines. Though the small red-shift can result in weak delocalization of Frenkel exciton in the aggregated state, the present SiNc in neat film shows the large third-order nonlinear susceptibility. Therefore, it is of potential ultility as material of optoelectronics, optical switching devices and integrated optics. Furthermore, facile introduction of the axial substituents with short alkyl length to decrease distance of adjacent naphthalocyanine rings, should further enhance the third-order optical nonlinearity.

4. Conclusion

In summary, the magnitude and dynamic response of the third-order optical nonlinearity of silicon 2,3-naphthalocyanine bis(trihexylsilyloxide) were measured by femtosecond DFWM technique under resonant conditions. Though delocalization of Frenkel exciton is weak in the aggregated state of present SiNc, time-resolved DFWM measurements indicate that the neat SiNc film show fast nonlinear optical response and large third-order nonlinear susceptibility. The effective $\chi_e^{(3)}$ value of the neat SiNc film was evaluated to be as high as 4×10^{-7} esu, and the figure of merit of $\chi^{(3)}/\alpha$ was calculated to be about 4.1×10^{-13} esu cm. The present SiNc has significant potential for applications in third-order nonlinear optics.

Acknowledgements

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