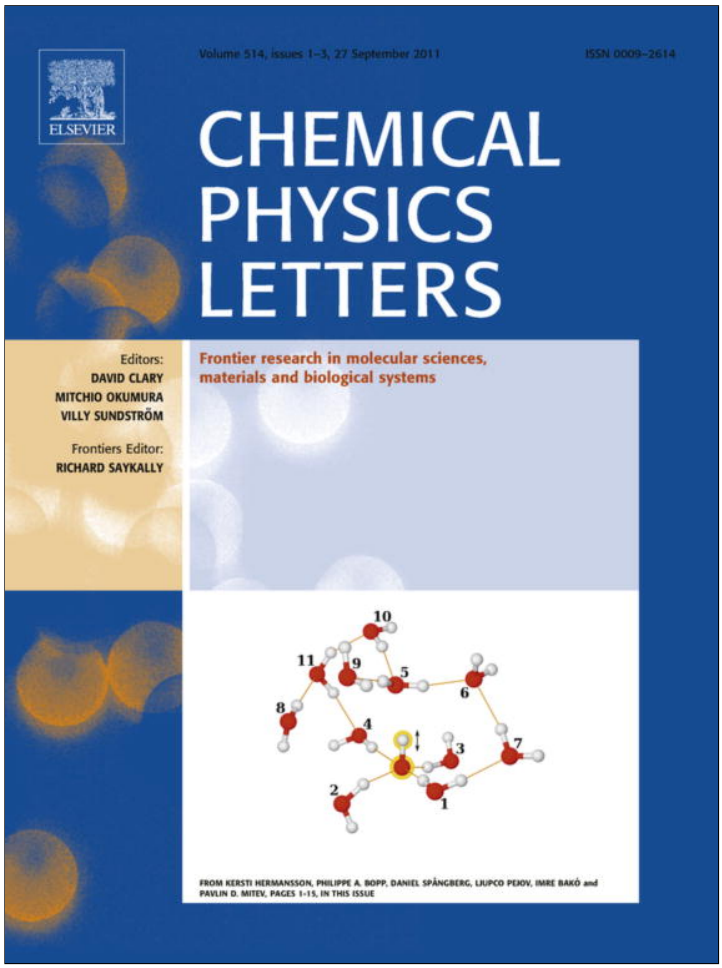

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Two-photon and three-photon absorption in dinaphthoporphycenes

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ABSTRACT

We present our results from the detailed investigations of both metal free-base and metal substituted dinaphthoporphycenes using the Z-scan technique with picosecond pulses. The compounds exhibited singular nonlinear absorption properties because of their molecular structure allowing us to access the two-photon and three-photon absorption states with different peak intensities when excited at the wavelength of 800 nm. This type of behaviour is explained using the unique energy level configuration of porphycene molecules. The orders of magnitude of both two-photon absorption and effective three-photon absorption coefficients and cross-sections were estimated along with sign and magnitude of the third order nonlinearity.

1. Introduction

Multi-photon absorption in a variety of materials, with organics in particular, find niche applications in the fields of imaging, lithography, memory based devices, optical limiting, and photodynamic therapy to name a few [1–6]. Porphyrins, phthalocyanines, their derivatives, and other molecules with large number of de-localized π electrons, are recognized to encompass large third-order nonlinearities enabling them for such applications [7–21]. In spite of a number of recent studies reporting molecules with large two-photon absorption (2PA) and three-photon absorption (3PA) coefficients/cross-sections, each of them using different pulses and at different wavelengths, further advances in this field are essential and beneficial. Furthermore, it is imperative to thoroughly appraise the structure–property relationship of such molecules, using pulses of varying duration and over a broad spectral range, for understanding their photo-physical behavior and enhancing their potential for practical applications [22–28]. Porphycenes, the constitutional isomers of widely researched porphyrins, were first synthesised by Vogel and co-workers in 1986 and subsequently many derivatives were reported [29–33]. Recently, large enhancement of two photon absorption (2PA) cross section ($\sigma^{(2)}$) was discovered in the case of conjugated porphyrin dimers, directly linked fused porphyrin dimers and even expanded porphyrins [17,18]. Particularly, aromatic core modified porphyrins are reported to have very large values of $\sigma^{(2)}$ [18]. To our knowledge only one report on 2PA studies of porphycenes [34] have been communicated indicating that these organic systems need to be explored further for their tremendous potential in nonlinear optical (NLO) applications. Arnbjerg et al. [34] have established that in the spectral domain of 750–850 nm the 2PA cross sections for two porphycenes (TPPo and PdTPPo) were large compared to that from the porphyrin analogue. These observations are attributed to the fact that, for the porphycenes, the two-photon transition is nearly resonant with a comparatively intense one-photon Q-band transition. In case of benzosapphyrins the extension of π-systems, by fusing the bipyrrole moiety with aromatic ring, has more dramatic effect on the electronic properties of the macrocycle than multiple fusion of benzene rings through the β-positions of respective pyrrole units. We have recently synthesized and studied a new class of porphycenes with interesting photo-physical and NLO properties [35]. In these molecules bipyrrole and naphthalene moieties are involved lending them the name dinaphthoporphycenes [36]. We observed that the metallation of the complex leads to significant change in the photo-physical properties of the complexes. Nonlinear optical studies using Z-scan technique with ~2 ps pulses at 800 nm have been performed to characterize the dinaphthoporphycenes. Closed and open aperture Z-scans were performed to estimate the third order nonlinearities of the molecules.

2. Experimental details

Different porphycenes (Po1–Po5) were synthesized and purified using the methods reported by us [35]. The common names of the compounds are as follows: Po1 is Tetra *n*-propyl dinaphthoporphycene, Po2 is Tetra iso-propyl dinaphthoporphycene, Po3 is Tetra *n*-pentyl dinaphthoporphycene, Po4 is Nickel substituted Tetra isopropyl dinaphthoporphycene, and Po5 is Nickel substituted

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Tetra *n*-pentyl dinaphthoporphycene. The compounds were dissolved in chloroform to obtain solutions of 0.25 mM concentration corresponding to a linear transmittance in the range of 70–80% near 800 nm. The Z-scan measurements were performed using ~2 ps (FWHM), 800 nm pulses with a repetition rate of 1 kHz from an amplified Ti:sapphire system (Legend, Coherent). The amplifier was seeded with pulses of duration ~15 fs (FWHM, spectral bandwidth of ~50–60 nm) from the oscillator (Micra, Coherent). The pulses were nearly transform limited and this was confirmed from the bandwidth and pulse duration measurements performed using an external auto-correlation experiment using a 2-mm thick BBO crystal in the non-collinear geometry. A quartz cuvette (1 mm thick) containing the sample solution was traversed in the focusing geometry enabled by an achromat lens of 200 mm focal length. The beam waist ($2\omega_0$) at focal plane was estimated to be 60 ± 4 μm ($FW1/e^2M$) with a corresponding Rayleigh range (Z_r) of 3.5 ± 0.4 mm ensuring the validity of thin sample approximation. The Z-scan was performed over a distance of 10 Z_r on a high-resolution linear translation stage (Newport ILS250PP) by recording the sample transmittance using a power sensor (Coherent PS19). A LabVIEW program was designed and used for automating the data acquisition of the Z-scan experiments. Typical energies in the range of 1–10 μJ, corresponding to peak intensities in the range of 70–400 GW/cm^2, were sued for all the experiments. The closed aperture scans were performed at peak intensities <100 GW/cm^2.

3. Results and discussion

Absorption spectra of all the molecules are shown in the Figure 1 along with their respective molecular structures (inset). In the metal free dinaphthoporphycenes, *n*-propyl, iso-propyl and *n*-pentyl groups are the different variations on the periphery of the core. The nickel coordinated dinaphthoporphycenes have only two variations with isopropyl and *n*-pentyl groups on the periphery of the core. The absorption spectra of metal free porphycenes (Po1, Po2, Po3) have large red shifted absorption bands, lowest energy Q-band (640–750 nm) appearing near 715 nm. These porphycenes demonstrate well-defined Soret or B-bands (350–430 nm) peaking near 400 nm. The lowest energy Q-band is relatively more intense than the Soret-band in comparison to other porphycenes [30]. The presence of few UV bands with maxima at 265 nm is attributed to the naphthalene moieties in the macrocycle. The absorption spectra of Po4 and Po5 show a general trend of red-shifted B band and blue-shifted Q-bands. The absorbance at 800 nm was negligible for most of the compounds with 80% transmittance.

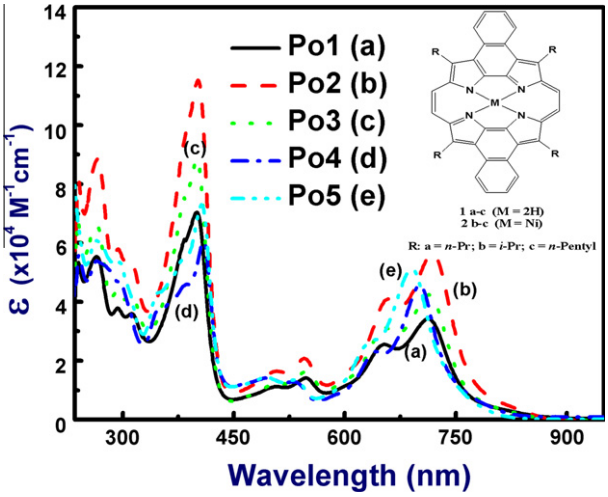

Figure 1. UV–visible absorption spectra of the compounds studied. Inset shows the structure of compounds.

Figure 2 shows the typical closed aperture data along with their corresponding fits for solvent chloroform and Po1–Po5 performed at incident intensities of 75 GW/cm^2. The data were fitted using the standard equations [37,38] to extract the nonlinear refractive index ($\mathbf{n_2}$). The samples exhibited large nonlinearities with the formation of annular rings near focus. This distorted the typical Z-scan curve near the valley reflecting in a slightly asymmetric curve which has been previously reported by Kouhski et al. [39] and Chen and co-workers [40]. However, the estimated nonlinear phase shift ($\Delta\phi$) was less than π for the data presented in Figure 1. All the samples possessed negative nonlinearity with the magnitudes in the range of 2–3 × 10^{-15} cm^2/W and are summarized in Table 1 along with their imaginary $\chi^{(3)}$ values. The reproduction of the typical closed aperture valley-peak signature for the solvent (chloroform) ruled out the possibility of any misalignment in the setup. Pure solvent exhibited a positive nonlinearity of ~0.55 × 10^{-15} cm^2/W. The solvent contribution being positive clearly suggests that the

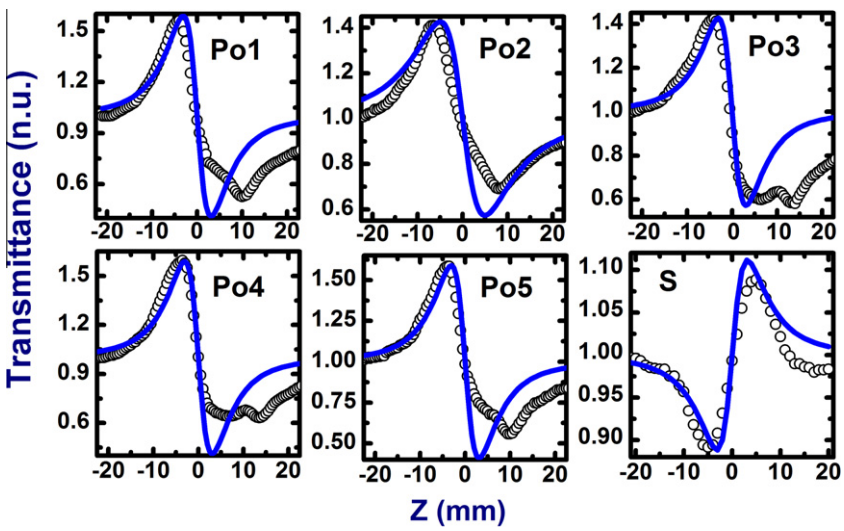

Figure 2. Closed aperture Z-scans of Po1–Po5 and chloroform recorded with peak intensities of 75 GW/cm^2. Open circles represent the experimental data whereas the solid lines are fits.

Table 1
Summary of the NLO coefficients obtained for Po1–Po5.

Sample	I_∞ (GW/cm^2)	β (cm/W) $\times 10^{-11}$	σ^2 (GM)	γ (cm^3/W^2) $\times 10^{-21}$	σ^3 (cm^6 s^2/photon^2) $\times 10^{-76}$	n_2 (m^2/W) $\times 10^{-19}$	Re$\chi^{(3)}$ (m^2/V^2) $\times 10^{-21}$	α_2 (m/W) $\times 10^{-13}$	Im$\chi^{(3)}$ (m^2/V^2) $\times 10^{-22}$	$\lvert\chi^{(3)}\rvert$ (m^2/V^2) $\times 10^{-21}$	$\lvert\chi^{(3)}\rvert$ (e.s.u.) $\times 10^{-13}$	W	T
Po1	78	5	8260	–	–	−3.8	4.3	5.0	3.5	4.3	3.1	1.9	1.0
	118	–	–	3.5	14.4								
Po2	98	7	11 600	–	–	−2.6	2.9	7.0	4.9	2.9	2.1	1.3	2.1
	132	–	–	9	36.9								
Po3	74	8.8	14 500	–	–	−2.7	3.0	8.8	6.2	3.0	2.2	1.3	2.6
	117	–	–	25	103								
Po4	74	13	21 500	–	–	−3.7	4.1	13.0	9.2	4.2	3.0	1.8	2.8
	120	–	–	19.5	80.1								
Po5	120	4.8	7930	–	–	−3.5	3.9	4.8	3.4	3.9	2.8	1.8	1.1
	407	–	–	11	45.2								

nonlinearity of the solute (porphycene) is higher than the values quoted here.

Figure 3a–j comprise open aperture Z-scan measurements performed at different peak intensities on the samples. The shape of Z-scan curves obtained at lower peak intensities when compared to those obtained at higher intensities were different indicating two different NLO processes occurring at those corresponding intensities. The presence of two resonances in the absorption spectra of these molecules, one near 400 nm (due to porphycene core) and other near 270 nm (due to naphthalene moiety), could explain the uncharacteristic nonlinear absorption behaviour of the molecules. 3PA in naphthalene has been ascertained earlier through several earlier studies [41,42]. For the 800 nm photon these two resonances can act as two-photon (2P) and three-photon (3P) states, respectively. Fakis et al. [43] reported similar data in pyrylium chromophores with femtosecond pulses at 760, 790, and 840 nm. In their case higher order effects appeared beyond a certain threshold of peak intensity which resulted in creating a critical population in the first excited state by 2PA process. Sutherland et al. [44] and Anémian et al. [45] also observed similar results and modelled their data using an effective 3PA coefficient. The difference between the *instantaneous* 3PA and *effective* 3PA is the process through which three photons are absorbed by the molecule. In the former case three photons are simultaneously absorbed (via virtual levels of the molecule) whereas in the latter case two photons are absorbed and the molecules is then in an excited state (real state). Depending on the lifetime of the excited state and

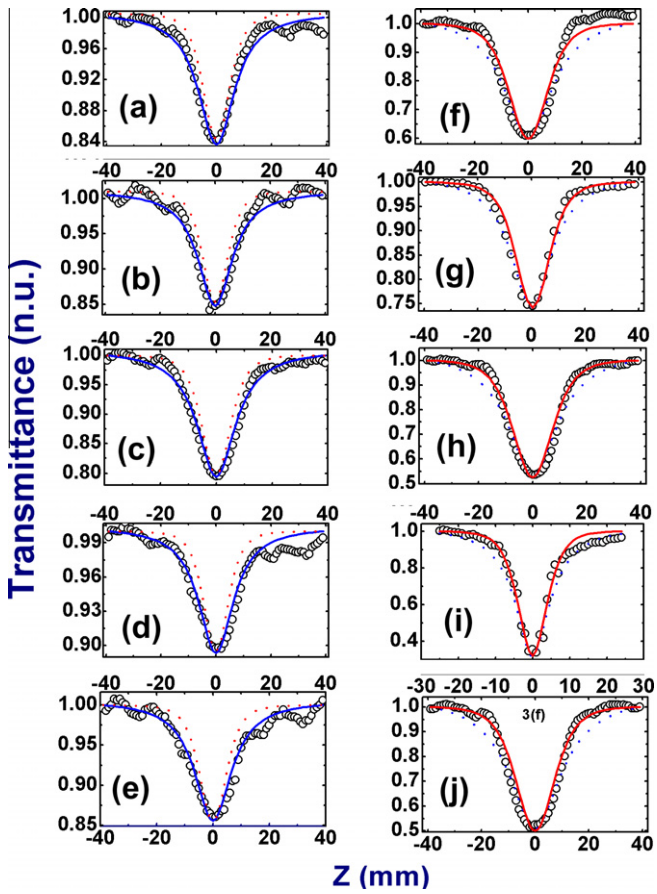

Figure 3. (a–j) Open aperture Z-scans of Po1–Po5 at different peak intensities (see Table 1). For low peak intensities the 2PA was best fit [blue solid lines are for 2PA and red dotted lines are for 3PA, (a–e)] while for high peak intensities 3PA was the best fit [red solid lines are for 3PA and blue dotted lines are for 2PA (f–j)]. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

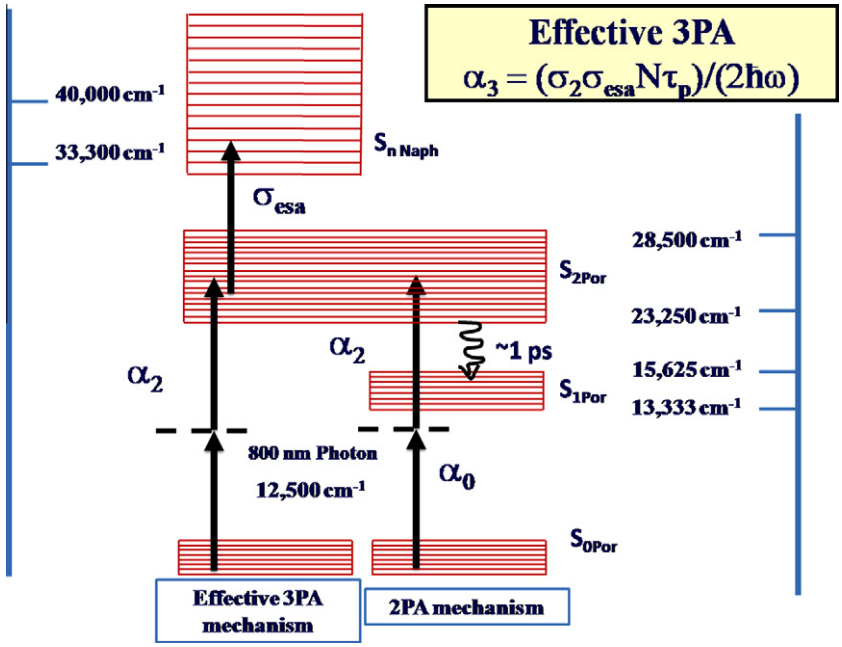

Figure 4. Energy level structures in porphycenes explaining the two resonances for 2PA and 3PA.

Table 2
Summary of comparison of NLO coefficients of Po1–Po5 with others reported in literature.

Compounds	Sample	σ^2 (GM)	τp	Ref.
Dye	Cytochrome *C*	100	150 fs	[16]
Heteroaromatic quadrupolar dyes	PEPEP	3130	130 fs	[28]
A-pi-A polymethine dyes	G37	2900	140 fs	
	G38	8800		[22]
	G74	15 000		
	G152	17 000		
Congeneric pentapyrrolic expanded porphyrins:	Pentaphyrin	3300	130 fs	
	Sapphyrin	2900		[25–27]
	Isosmaragdyrin	2700		
	Orangarin	1200		
Porphyrin-Squaraine-Porphyrin	PoR- SQA-Por	11 000	100–140 fs	[23]
Self-assemblies of butadiyne-linked bis(imidazolylporphyrin)	Polymer Zn Substituted Bis(Imidazolylprophyrin)	440 000	120 fs	[24]
Water soluble porphyrin dimers	P_2C_2-NMeI	17 000	300 fs	
	P_2C_2-CO_2NH_4	14 000		[25]
	P_2-Suc	10 000		
Tetraphenylporphycenes (TPPo)	TPPo	2280	120 fs	
	PdTPPo	1750		[34]
	meso TPPo	24		
Dinapthoporphycenes	Po1	8260	~2 ps	
	Po2	11 600		This work
	Po3	14 500		
	Po4	21 500		
	Po5	7930		

pulse duration there could be absorption to even higher excited states using another photon (2 + 1) provided the excited state cross-section is significant. The effective 3PA can also be termed as two-photon resonant three-photon absorption.

Figure 4 clearly illustrates the possible excitation mechanism for our molecules causing the singular nonlinear absorption behaviour of the molecules. At lower peak intensities the 2PA could be from the S_2 states (23 250–28 500 cm^{-1} with single photon corresponds to 12 500 cm^{-1}) of porphycene molecule while the effective 3PA is a result of 2PA and excited state absorption from S_2 state to the S_n states (33 300–40 000 cm^{-1}) of the porphycene molecule. Typical lifetimes of S_2 states in such molecules are reported to be <1 ps [46]. The open aperture Z-scan data have been fitted by time integration of sample transmittance assuming a Gaussian temporal profile [37,38,44,47]. The 2PA fitting was done using the Eq. (1) while the effective 3PA fitting was done using Eq. (2).

$$T(z,S=1) = \frac{1}{\sqrt{\pi}q_0(z,0)}\int_{-\infty}^{\infty}\ln[1+q_0(z,0)e^{-\tau^2}]d\tau \quad (1)$$

$$T(z,S=1) = \frac{1}{\sqrt{\pi}p_0(z,0)}\int_{-\infty}^{\infty}\ln\left[\sqrt{\ln(1+p_0^2\exp(-2\tau^2))} + p_0(z,0)\exp(-\tau^2)\right]d\tau \quad (2)$$

where $T(z)$ is the normalized transmittance as a function of z, $q_0 = \alpha_2 L_{eff} I_0$, $p_0 = (2\alpha_3 L'_{eff} I_0^2)^{1/2}$, α_2 = 2PA coefficient and α_3 = effective

3PA coefficient, I_0 is the peak intensity at focus. Effective path lengths in the sample of length L for 2PA, 3PA are given as $L_{eff} = [1 - \exp(-\alpha_0 L)/\alpha_0]$, $L'_{eff} = [1 - \exp(-2\alpha_0 L)/2\alpha_0]$, respectively, and α_0 is the linear absorption coefficient. The effective 3PA coefficient is defined as $\alpha_3 = \frac{\tau_p \sigma^{\beta}_{esa}}{2h\nu}$ [41–43].

At lower incident intensities (typically < 100 GW/cm^2), the data presented in Figure 3a–e was best fitted to 2PA (Eq. (1)) indicating that 2PA was the dominant mechanism. At higher incident intensities (typically > 110 GW/cm^2), the data presented in Figure 3f–j was best fitted to effective 3PA (Eq. (2)) indicating its dominance in this regime. For Po5 2PA and 3PA were observed for peak intensities of 120 GW/cm^2 and 400 GW/cm^2, respectively. $\chi^{(3)}$ (TPA, Kerr effect etc.) and $\chi^{(5)}$ effects proceed from the combination of the same kind of excited states. There are no different excited schemes separately for $\chi^{(3)}$ and $\chi^{(5)}$. Interference effects related to the phase of the nonlinearity (it is a complex number) can make them appear with different thresholds [48]. In the present case the S_2 state is responsible for both $\chi^{(3)}$ process (2PA) and $\chi^{(3)}$ process (effective 3PA). The values of ground state absorption cross-section measured for Po1-Po5 were 1.8, 2.4, 2.3, 2.4, 2.7 ($\times 10^{-17}$ cm^2) whereas the excited state cross-sections (σ_{ex}) obtained from the fits were 3.9, 6.5, 14.4, 7.4, 11.6 ($\times 10^{-17}$ cm^2), respectively. These values were obtained using 1 ps [46] as the lifetime of S_2 state in Eq. (2) and the values of β used were obtained from the fits to low intensity data. Table 1 furnishes the 2PA and 3PA cross-sections at different incident intensities for all the samples. Our measurements were repeated at very low peak intensities (~50 GW/cm^2) for the samples Po2, Po3, and Po4 and the data again confirmed the presence of only 2PA and not 2PA + ESA. However, the magnitude of 2PA coefficients could have been over-estimated [49] using this technique and other complementary techniques are required to arrive at the exact values. The random experimental errors (arising from estimation of spot size at focus, concentration measurements, input power measurements, data fitting etc.) result in an overall error of ± 20% in our calculations. The magnitudes Im [$\chi^{(3)}$] and $\chi^{(3)}$ were calculated from the nonlinear coefficients. For examining the potential of these molecules in photonic applications the figures of merit (FOMs) T and W were evaluated and the data is again shown in Table 1. $T > 1$ suggest large nonlinear refraction values suggestive of the potential application of dinaphthoporphycenes in photonic devices. However, the corresponding FOM for nonlinear absorption, W, is >1. $W < 1$ is generally desirable for photonic devices.

The 2PA absorption cross-sections are also compared with some of the previously reported molecules and summary of the data is presented in Table 2. Cytochrome *C* [16], PEPEP [28] dyes exhibited low $\sigma^{(2)}$ values of 100 and 3130, respectively. Recently, A-π-A polymethine dyes have demonstrated large cross-sections of the order of 10^4 GM [22]. The porphyrins with expanded rings [26,27] exhibit $\sigma^{(2)}$ values of 1200–3300 GM whereas Porphyrin-Squaraine-Porphyrin assembly has been reported to have $\sigma^{(2)}$ of 11 000 GM [21]. For water soluble porphyrins [25], the maximum values reported are of the magnitude ~10^4 GM. However, in comparison to previous molecules, dinaphthoporphycenes have large $\sigma^{(2)}$ values with Ni substituted Po4 having $\sigma^{(2)}$ = 21 500 GM. But self assembled porphyrins reported by Ogawa et al. [24], have larger $\sigma^{(2)}$ magnitude (~440 000 GM) than present work. The nonlinear coefficients presented in this work are obtained with ~2 ps pulses and assuming the magnitudes could be larger (due to longer pulses) by one order of magnitude when compared to 100 fs pulses data, the order of magnitudes for our samples (single monomer molecules) are still comparable to many of the recently investigated molecules (either oligomers or polymers). Our preliminary femtosecond pump probe data suggest that these molecules have very fast excited state (S_1) life times of <100 picoseconds. Therefore, we strongly feel that these molecules find potential applications in the fields of photonics and imaging. Our future efforts will focus on (a) evaluating the nonlinear coefficients with shorter pulses (100 fs or less) (b) examine the nonlinearities at different spectral regions to ascertain the nonlinear absorption mechanisms and their figures or merit (c) undertaking the ultrafast pump–probe studies to establish the excited state dynamics in these molecules.

4. Conclusions

From the first level investigations using Z-scan technique we have evaluated nonlinear absorption coefficients, cross-sections and refractive indices for the dinaphthoporphycenes in solution form. The unusual nonlinear absorption behaviour is accounted by the presence of two resonances in the absorption spectrum which allows one to access the two-photon and three-photon states using different peak intensities. The magnitudes of nonlinear coefficients are superior or comparable to some of the recently reported molecules with large nonlinearities.

Acknowledgments

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