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Theoretical Analysis on Optical Limiting Properties of Newly Synthesized Graphene Oxide-Porphyrin Composites

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Optical limiting (OL) properties and two-photon absorption (TPA) of a series of covalently linked graphene oxide-porphyrin composite materials have been investigated by numerically solving the rate equations and field intensity equation with an iterative predictor-corrector finite-difference time-domain technique in nanosecond time domain. Our results show that graphene oxide-porphyrin composites exhibit enhanced OL behavior and possess larger TPA cross section compared with individual porphyrins. Interestingly, unlike the previous result that porphyrin with heavier central metal shows better nonlinear abilities than that without metal substitute, graphene oxide-metal free porphyrin composite has stronger nonlinear absorption properties compared with graphene oxide-metal porphyrin composite. The computational results are in reasonable agreement with the experimental ones. Special attention has been paid to the influence of thickness of the medium and pulse width on TPA cross sections, which presents that larger TPA cross sections are obtained as the medium is thicker or the pulse duration is wider.

Key words: Optical limiting, Two-photon absorption, Graphene oxide, Porphyrin

I. INTRODUCTION

Currently, materials exhibiting strong nonlinear optical (NLO) absorption are of much interest as optical limiters for potential applications in the protection of eyes and optical sensors against damage exposed to undesired high-intensity radiation [1−6]. One mechanism is reverse saturable absorption (RSA) that is especially effective in producing good optical limiting (OL) behavior. The RSA is mainly contributed by two-photon absorption (TPA) [7, 8]. Looking for optical limiters with good RSA ability is a hot research topic during the last decade [9, 10].

Graphene has attracted great attention because of its extraordinary electric, thermal, mechanical properties, and processability [11−17]. Unfortunately, the poor dispersibility of graphene limits its application. However, this problem can be overcome by introducing various organic molecules covalently or non-covalently into graphene sheets. Moreover, porphyrin (Por) is widely used as the attachment on account of its special structures of two-dimensional highly conjugated delocalized π-electron system and its ability of bonding with metal. During the past few years, many experimental groups have explored properties of graphene or its functional compounds. Feng *et al.* investigated the

properties of graphene families, including graphene oxide nanosheets, graphene nanosheets (GNSs), graphene oxide nanoribbons (GONRs), and graphene nanoribbons (GNRs) with a nanosecond laser pulse at 532 and 1064 nm, showing that these compounds exhibited broadband NLO as well as good OL performance, and TPA had strong effects on the NLO and OL responses of the graphene nanostructures [18]. Liu *et al.* studied the NLO properties of two novel graphene nanohybrid materials covalently functionalized with Por and fullerene using the *Z*-scan technique at 532 nm in the nanosecond and picosecond time scale, which presented that covalently functionalized graphene with Por and fullerene can enhance the NLO performance in nanosecond regime [19]. Very recently, a series of metal (Sn and VO) or metal free (H_2) porphyrins (Sn-Por, VO-Por and H_2-Por) and graphene oxide (GO) composites functionalized by them (GO-Sn-Por, GO-VO-Por and GO-H_2-Por) were synthesized and their photophysical properties were investigated, suggesting that the GO and Por composites can be used as good reverse saturable absorbers [20].

In comparison with abundant experimental research, theoretical studies on the mechanism of enhanced nonlinear optical abilities for graphene composites at *ab initio* level are necessary. However, electronic structures of the materials are quite difficult to be obtained at *ab initio* level mainly due to the limitation of computational capacity. Moreover, dynamical parameters of laser field greatly influence the NLO properties, thus interaction between laser field and the medium should

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257

be considered for exploring the NLO properties [21, 22]. In order to get reasonable explanation on the experiment measurements and deepen understanding the enhancement mechanism of these complex composite systems, we need to simplify the composite with a few energy levels and consider the interaction between laser and molecule. In this work, we present a dynamical theory of sequential TPA for long pulse, and theoretically study the OL behavior of a series of GO-Por compounds by numerically solving field intensity equation together with rate equations in local time frame with the iterative predictor-corrector finite-difference time-domain (FDTD) technique method [23].

II. THEORETICAL METHODS AND COMPUTATIONAL DETAILS

Because the time of intersystem crossing (ISC) transition between the singlet and triplet excited states of porphyrins can be ignored compared with the pulse duration, we simply take the composites as a generalized three-level model as suggested by Rao *et al.* [20], which includes the ground state S_0, the excited states S_1 and S_2 (Fig.1). Thus, two sequential TPA channels $(S_0 \rightarrow S_1) \times (S_1 \rightarrow S_2)$ would play a dominating role for a long pulse.

A. Rate equations and field intensity equation for a three-level system

On basis of the rotating wave approximation (RWA) and slowly varying envelope approximation (SVEA), rate equations for the populations of the S_0, S_1, S_2 states can be written as

$$\frac{\partial}{\partial t}\rho_{S_0} = -\gamma_{S_0 S_1}(\rho_{S_0} - \rho_{S_1}) - \gamma_{S_0 S_2}(\rho_{S_0} - \rho_{S_2}) + \Gamma_{S_1}\rho_{S_1} \tag{1}$$

$$\frac{\partial}{\partial t}\rho_{S_1} = \gamma_{S_0 S_1}(\rho_{S_0} - \rho_{S_1}) - \gamma_{S_1 S_2}(\rho_{S_1} - \rho_{S_2}) - \Gamma_{S_1}\rho_{S_1} + \Gamma_{S_2}\rho_{S_2} \tag{2}$$

$$\frac{\partial}{\partial t}\rho_{S_2} = \gamma_{S_0 S_2}(\rho_{S_0} - \rho_{S_2}) + \gamma_{S_1 S_2}(\rho_{S_1} - \rho_{S_2}) - \Gamma_{S_2}\rho_{S_2} \tag{3}$$

where Γ_{S_0}, Γ_{S_1}, and Γ_{S_2} are decay rates of the states S_0, S_1, and S_2 respectively. The total populations are normalized as $\sum_{n=0}^{2} \rho_{S_n} = 1$. $\gamma_{S_0 S_1}$, and $\gamma_{S_1 S_2}$ are rates of the one-photon induced transitions $S_0 \rightarrow S_1$ and $S_1 \rightarrow S_2$, which can be expressed through the corresponding one-photon absorption (OPA) cross sections $\sigma_{S_0 S_1}$, $\sigma_{S_1 S_2}$ or dipole moments $\mathbf{d}_{S_0 S_1}$, $\mathbf{d}_{S_1 S_2}$ in the RWA

$$\gamma_{S_0 S_1}(t) = \frac{|\mathbf{d}_{S_0 S_1}|^2 I(t)}{\hbar^2 c \varepsilon_0} \frac{\Gamma}{\Omega_{S_0 S_1}^2 + \Gamma^2}$$
$$= \frac{\sigma_{S_0 S_1} I(t)}{\hbar \omega} \frac{\Gamma^2}{\Omega_{S_0 S_1}^2 + \Gamma^2} \tag{4}$$

$$\gamma_{S_1 S_2}(t) = \frac{|\mathbf{d}_{S_1 S_2}|^2 I(t)}{\hbar^2 c \varepsilon_0} \frac{\Gamma}{\Omega_{S_1 S_2}^2 + \Gamma^2}$$
$$= \frac{\sigma_{S_1 S_2} I(t)}{\hbar \omega} \frac{\Gamma^2}{\Omega_{S_1 S_2}^2 + \Gamma^2} \tag{5}$$

$$\Omega_{S_0 S_1} = \omega - \omega_{S_0 S_1} \tag{6}$$
$$\Omega_{S_1 S_2} = \omega - \omega_{S_1 S_2} \tag{7}$$

where $\Omega_{S_0 S_1}$ and $\Omega_{S_1 S_2}$ are detuning of the light frequency ω from the resonant frequency. It is important to note that $\sigma_{S_1 S_2}$ here is the effective excited state absorption (ESA) cross section for the composites, including $\sigma_{S_1' S_2'}$ and $\sigma_{T_1' T_2'}$ (OPA cross section from the lowest triplet state T_1' to higher triplet state T_2') of porphyrin as well as $\sigma_{S_1'' S_2''}$ of graphene oxide. The homogeneous broadening of the spectral line Γ is set as $\hbar\Gamma$=0.1 eV.

Similarly, the rate of two-photon induced transition $S_0 \rightarrow S_2$ can be defined through the TPA cross section $\sigma_{S_0 S_2}$ as

$$\gamma_{S_0 S_2} = \frac{\sigma_{S_0 S_2} I^2(t)}{2\hbar\omega} \frac{\Gamma^2}{(2\omega - \omega_{S_0 S_2})^2 + \Gamma^2} \tag{8}$$

As for the electromagnetic field, the absorption of the field can be described by the following field intensity equation within RWA and without considering the change of refraction and transverse of the field

$$\left(\frac{\partial}{\partial z} + \frac{1}{c}\frac{\partial}{\partial t}\right) I(t) = -N[\sigma^{(1)} I(t) + \sigma^{(2)} I^2(t)] \tag{9}$$

where N is the molecular density, $\sigma^{(1)}$ and $\sigma^{(2)}$ are the total OPA and TPA cross sections, respectively, which can be expressed by

$$\sigma^{(1)} = \sigma_{S_0 S_1}(\rho_{S_0} - \rho_{S_1}) + \sigma_{S_1 S_2}(\rho_{S_1} - \rho_{S_2}) \tag{10}$$
$$\sigma^{(2)} = \sigma_{S_0 S_2}(\rho_{S_0} - \rho_{S_2}) \tag{11}$$

B. Calculation of the dynamical TPA cross section

Theoretical details are referred to Ref.[24]. Here, we just list the main formulations. Inverse transmission $1/T_z$ is a quadratic function of the input field intensity I_0,

$$\frac{1}{T_z} = \frac{I_0}{I_z} = \exp(\alpha z) + \frac{[\exp(\alpha z) - 1]\beta}{\alpha} I_0 - \frac{[\exp(\alpha z) - 1]\xi}{\alpha} I_0^2 \tag{12}$$

We can determine the absorption coefficients α and β by fitting the input-output pulse peak intensity through the above equation, and the TPA cross section can thus be obtained using

$$h\nu\beta = \sigma_{tp} N \tag{13}$$

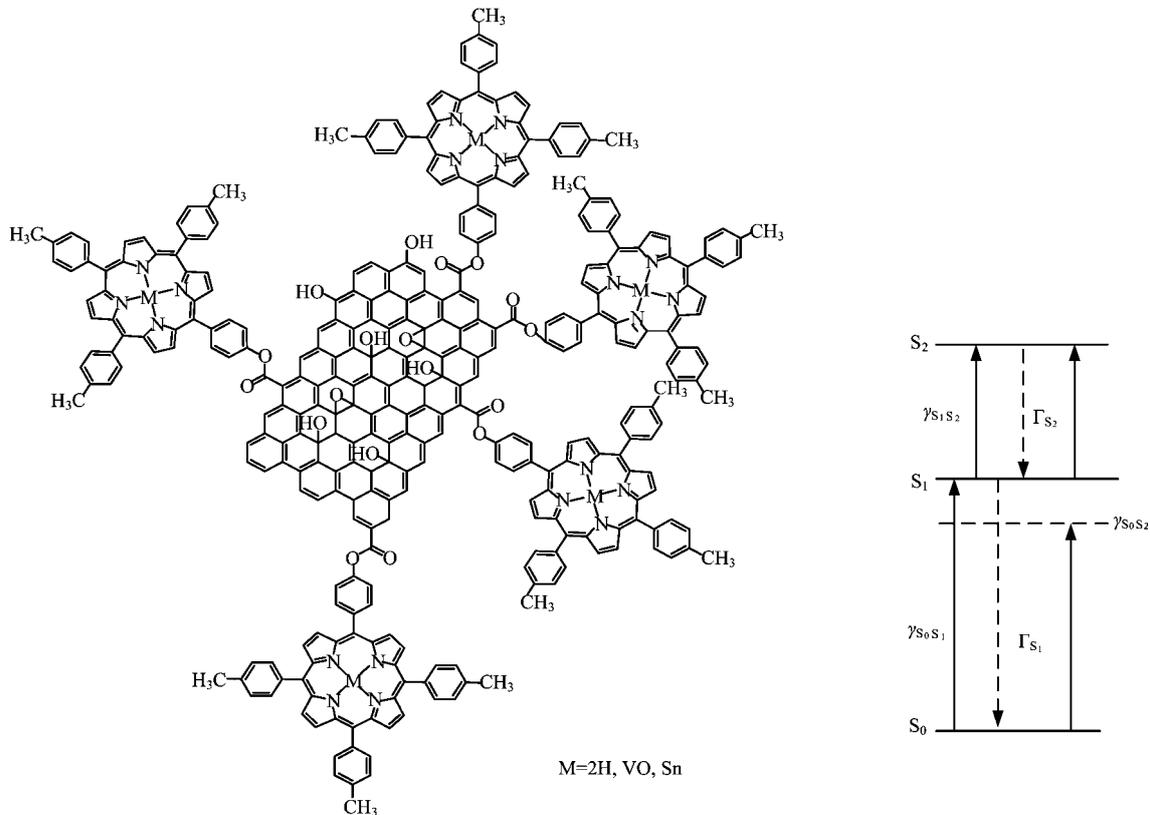where $h\nu$ is the incident photon energy.

FIG. 1 The structures of composites (left) and the scheme of transitions (right).

TABLE I Photophysical parameters of porphyrins and their graphene oxide composites [20]. $\sigma_{S_0S_1}$ and $\sigma_{S_1S_2}$ are in unit of 10^{-23} and 10^{-22} m^2, respectively.

Molecule	$E_{S_0S_1}$/eV	$E_{S_1S_2}$/eV	$\sigma_{S_0S_1}$	$\sigma_{S_1S_2}$	$\sigma_{S_1S_2}/\sigma_{S_0S_1}$	$\Gamma_{S_1}/(10^8/s)$	$\Gamma_{S_2}/(10^{11}/s)$	$\beta/(nm/W)$
VO-Por	2.25	0.63	4.8	8.4	17.5	7.7	4.5	1.42
GO-VO-Por	2.24	0.62	8.7	26.2	30.1	8.3	18	37.6
Sn-Por	2.20	0.67	4.5	10.2	22.7	2.4	4.3	1.68
GO-Sn-Por	2.19	0.67	8.5	24.8	29.2	2.5	17	36.5
H$_2$-Por	2.40	0.56	4.7	9.8	20.9	1.1	3.6	1.59
GO-H$_2$-Por	2.41	0.52	8.8	41.2	46.8	1.6	15	49.2

C. Computational details

Structures of three different porphyrin molecules (VO-Por, Sn-Por and H$_2$-Por) and graphene oxide composites (GO-VO-Por, GO-Sn-Por and GO-H$_2$-Por) are shown in Fig.1. For performing numerical simulations, the resonant transition energies, OPA cross sections and relaxation rates of these compounds are extracted from the experiments [20]. The corresponding values are collected in Table I. The previous works found that numerical results can be compared with the measurements according to the scaling relation $N_{theo}L_{theo}=N_{exp}L_{exp}$ [5]. Therefore, our simulations are performed for relatively high concentration and short distance compared with the experimental condition. Concentrations

N_{theo} for porphyrin and composite are thus chosen as 3.52×10^{23}/m^3 and 2.26×10^{23}/m^3, respectively.

The input electric field is chosen to be a hyperbolic secant shape incident pulse

$$E(z,t=0)=F_0 \sec h\left[\frac{1.76}{\tau}\frac{(z+z_0)}{c}\right]\cos\left[\frac{\omega(z+z_0)}{c}\right] \tag{14}$$

where τ is the full width at half maximum (FWHM) of pulse intensity profile. F_0 is peak amplitude, which is related with field intensity I by:

$$I(t) = \frac{c\varepsilon_0 |F_0(t)|^2}{2} \tag{15}$$

The choice of z_0 ensures that the pulse penetrates neg-

ligibly into the medium at $t=0$. In order to match the experimental parameters, duration and wavelength of pulse are set to 6 ns and 532 nm, respectively.

III. RESULTS AND DISCUSSION

For exploring OL performance of the compounds, transmittances of intensity are shown in Fig.2. It reveals that all the studied porphyrins and its graphene oxide composites exhibit outstanding nonlinear absorption ability, which is influenced by the central metals. One can see from Fig.2(a) that all the media show constant transmittances at low intensity, indicating a linear absorption process. However, with the increase of the input intensity, transmittance decreases remarkably. The mechanism can be explained using the simplified three-level scheme (see Fig.1). Molecules at S_0 state are excited to S_1 state by absorbing the first photon, and then they will absorb another photon to reach higher excited state S_2 when the input intensity is strong enough. If absorption cross section of the excited state excesses that of the ground state, RSA occurs and transmittance reduces significantly. It is also clear that the transmittances of graphene oxide-porphyrin composites are much smaller than that of single porphyrins, indicating a better OL ability. The covalently linked graphene oxide not only provides more delocalized electrons but also opens an additional channel for porphyrin to transfer energy between them. Figure 2(b) displays the transmittance as a function of the medium thickness with the field intensity $I_0=5\times10^8$ W/cm². It is obvious that OL behavior is better for longer propagation distances.

In order to get understanding of the dynamical process of OL, H_2-Por and its graphene oxide composite are selected to study the pulse propagation owing to their outstanding OL properties as shown in Fig.2. Temporal evolution of the pulse intensity envelope and the population of different states at different propagation distances are demonstrated in Fig.3. The peak intensity is set to be $I_0=5\times10^8$ W/cm². Figure 3(a) displays that pulse intensities decrease rapidly during pulse propagation since the energy transfer from the field to molecules. Moreover, more energy is transferred for the composite than H_2-Por on account of the larger ESA cross section (see Table I). Figure 3(b) shows that molecules are mainly excited from the ground state S_0 to the OPA state S_1, while the TPA state S_2 is populated slightly. The main reason is that, in long pulse regime, incoherent two-step TPA plays a dominative role compared to the coherent one-step TPA process. Population of the S_2 state decays rapidly back to the S_1 state due to rather short lifetime of S_2.

We further present the curves of output fluence versus input fluence for different compounds at the propagation distance of 0.48 mm in Fig.4. OL behaviors of the compounds are clearly demonstrated. One can see that the composites show enhanced OL ability compared
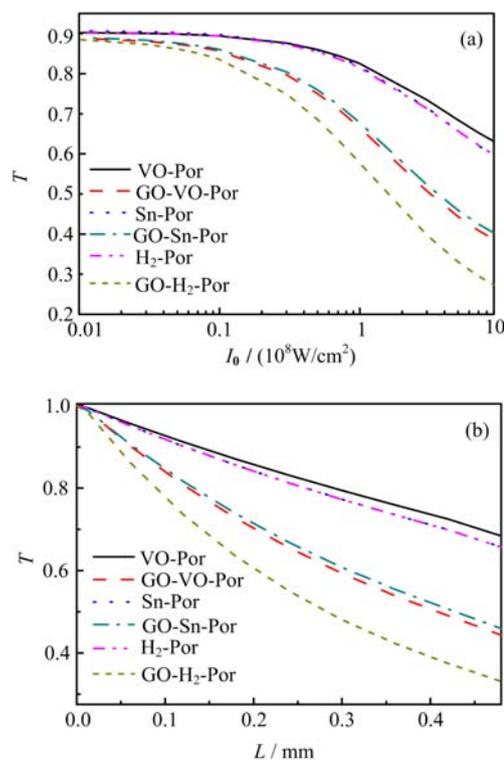

FIG. 2 Transmittance as functions of (a) the peak intensity of incident field ($L=0.48$ mm) and (b) the thickness of the medium ($I_0=5\times10^8$ W/cm²). $\tau=6$ ns and $N=N_{\text{theo}}$.

with individual porphyrins. As value of FOM in Table I increases, the OL ability turns better, which agrees well with the experimental results. It is noteworthy that different central metals have different influences on the OL behavior. Graphene oxide-metal free porphyrin owns the best nonlinear absorption behavior compared with the others, while previous studies showed that porphyrin with heavier central metal has better OL performance as a result of the faster ISC caused by the enhanced spin-orbit coupling [25]. In the composite, graphene oxide acts as electron acceptor, and peripheral porphyrin shows as electron donor. Because metal free porphyrin possesses stronger electron giving capacity than metal porphyrin, electrons are easily transferred between metal free porphyrin and graphene oxide, which further leads to preferable ESA for graphene oxide-metal free porphyrin. It is interesting that, when porphyrins are attached to the graphene oxide, their nonlinear absorption abilities are largely modulated. That is to say, OL ability for single porphyrin molecules takes the order of Sn-Por>H_2-Por>VO-Por, while for graphene oxide-porphyrin composites the order turns into GO-H_2-Por>GO-VO-Por>GO-Sn-Por. This phenomenon suggests an effective approach for improving optical properties of individual porphyrins.

By fitting input-output peak intensity curves in the OL region, we get TPA coefficients β of the molecules at propagation distance of 0.48 mm, which
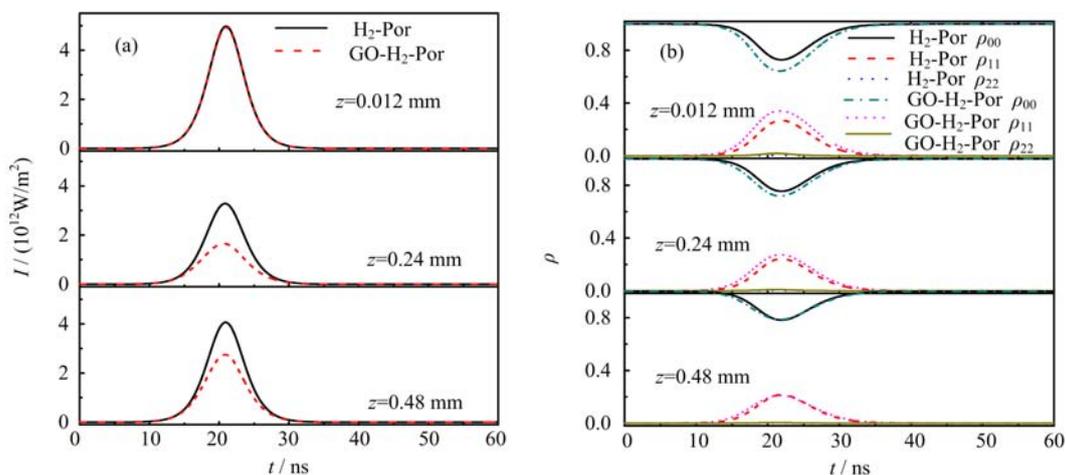
FIG. 3 (a) Temporal evolution of the profile of the pulse intensity and (b) dynamics of the populations of different energy levels at different propagation distances of 0.012, 0.24, 0.48 mm. The input peak intensity $I_0=5\times10^8$ W/cm^2, $\tau=6$ ns and $N=N_{\text{theo}}$.

TABLE II Values of TPA coefficient β (nm/W) and TPA cross section σ_{tp} (10^6 GM) at different propagation distances of 0.24, 0.36, and 0.48 mm.

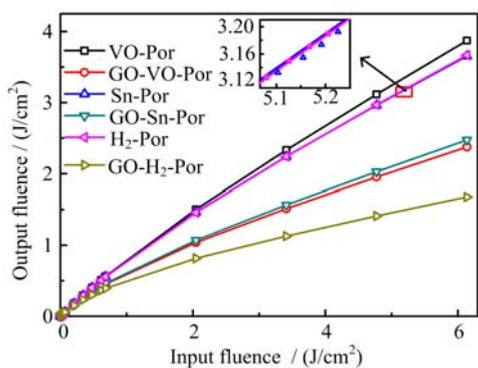| Molecule	0.24 mm		0.36 mm		0.48 mm	
	β	σ_{tp}	β	σ_{tp}	β	σ_{tp}
VO-Por	0.187	1.990	0.191	2.025	0.193	2.042
H$_2$-Por	0.218	2.317	0.222	2.358	0.224	2.377
Sn-Por	0.219	2.323	0.223	2.365	0.225	2.387
GO-Sn-Por	0.562	9.284	0.585	9.675	0.603	9.964
GO-VO-Por	0.598	9.885	0.624	10.320	0.644	10.644
GO-H$_2$-Por	0.950	15.693	1.000	16.578	1.043	17.245

FIG. 4 Output fluence versus input fluence for different compounds at the propagation distance of 0.48 mm. $\tau=6$ ns and $N=N_{\text{theo}}$.

are 0.193, 0.224, 0.225, 0.603, 0.664, 1.043 nm/W for VO-Por, H$_2$-Por, Sn-Por, GO-Sn-Por, GO-VO-Por, GO-H$_2$-Por, respectively. These values are consistent with the experimental measurement in trend. Dynamical values of the TPA cross section are thus obtained as 2.042×10^6, 2.377×10^6, 2.387×10^6, 9.967×10^6, 1.064×10^7, 1.725×10^7 GM

(1 GM=1×10^{-50} cm^4·s/photon) for VO-Por, H$_2$-Por, Sn-Por, GO-Sn-Por, GO-VO-Por, GO-H$_2$-Por, respectively, which are several orders of magnitude larger than that of other organic molecules [3, 5]. From the results, we can find that introducing graphene oxide into porphyrin can effectively enhance nonlinear absorption ability of the porphyrin because GO has rich delocalized π-electrons.

The values of TPA coefficient β and TPA cross section σ_{tp} at different propagation distances in the medium are given in Table II. It is seen that longer propagation distance results in stronger TPA because more energy can be stored into the medium for longer distance. For GO-H$_2$-Por as an example, dynamical values of TPA cross section are obtained as 1.57×10^7, 1.66×10^7, 1.73×10^7 GM at propagation distances of 0.24, 0.36, and 0.48 mm, respectively. Thus, TPA cross sections are dependent on thickness of medium. We also investigate the influence of pulse widths on β and σ_{tp}. As shown in Table III, one can see that TPA becomes stronger with the increase of pulse duration. For instance, dynamical TPA cross sections of GO-H$_2$-Por are 3.04×10^6, 1.41×10^7, and 1.73×10^7 GM when the pulse durations are 0.3, 3, and 6 ns, respectively. For

TABLE III Values of TPA coefficient β (nm/W) and TPA cross section σ_{tp} (10^6 GM) with different input pulse width at the propagation distance of 0.48 nm.

Molecule	0.3 ns		3 ns		6 ns	
	β	σ_{tp}	β	σ_{tp}	β	σ_{tp}
VO-Por	0.035	0.367	0.151	1.604	0.193	2.042
H_2-Por	0.040	0.429	0.174	1.849	0.224	2.377
Sn-Por	0.041	0.430	0.176	1.868	0.225	2.387
GO-Sn-Por	0.110	1.818	0.502	8.301	0.603	9.964
GO-VO-Por	0.117	1.927	0.536	8.863	0.644	10.644
GO-H_2-Por	0.184	3.039	0.855	14.129	1.043	17.245

pulses with longer duration, populations of the S_1 state have more opportunities and enough time to be excited to the S_2 state through sequential transition $S_1 \rightarrow S_2$. Thus, two-step sequential TPA becomes the main absorption mechanism in nanosecond domain. It is indicated that broadening the pulse width is a useful way to enhance TPA of the compounds.

IV. CONCLUSION

We have studied the NLO properties of a series newly synthesized metal and metal free porphyrins with their graphene oxide composites utilizing a dynamical nonlinear absorption theory of laser pulse with duration on the scale of nanoseconds. A simplified three-level scheme is introduced to illustrate the mechanism of achieving OL. It is found that graphene oxide-porphyrin composites possess enhanced nonlinear absorption compared with individual porphyrins on account of the abundant delocalized charge transfer from porphyrin to graphene oxide, while the OL performance of the compounds is influenced by the central substituent of the porphyrin. Moreover, the dynamical TPA cross sections are obtained by numerical fitting the output-input peak intensity curves in the OL region, which indicates that thickness of the absorber and pulse duration should be taken into consideration when the nonlinear optical properties of medium are investigated. Our results suggest an efficient way to enhance the NLO properties of compounds by introducing graphene oxide.

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