Resonant Optical Nonlinearity and Fluorescence Enhancement in Electrically Tuned Plasmonic Nanosuspensions

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1. Introduction

Colloidal materials play an increasingly important role in many fields such as chemistry, life sciences, material sciences, and physics. Currently, one of the major scientific endeavors is to synthesize artificial materials via colloidal systems that exhibit desired optical properties but are not readily found in nature. It is also of great importance to control the flow of light in colloidal systems for fundamental and technological reasons. In the last decade, colloidal suspensions containing dielectric micro- or nanometer-sized particles have been shown to exhibit optical nonlinearities that lead to modulation instability and optical spatial solitons. Recently, plasmonic nanosuspensions are found to have superior performance, as compared with their dielectric counterparts, in demonstrating tunable optical nonlinearities that support guiding and self-trapping of light beams. Robust soliton-like propagation of light over a long distance of several centimeters has been observed due to an effective cubic–quintic-like plasmonic resonant nonlinearity, which would otherwise be impossible in such highly scattering media.

One established way to actively tune the properties of colloidal systems of rod-like particles is to apply an electric field (e-field). Although such alignment of long nanorods (aspect ratio > 10) in aqueous suspensions has been reported before, the alignment of short nanorods was demonstrated only recently in organic solutions, where absorption at the longitudinal surface plasmon resonance (LSPR) is shown to decrease with increasing e-field when light propagates along the direction of the field. In a similar experiment, the nonlinear refractive index of plasmonic suspensions is shown to be dynamically controlled in a Hartmann–Shack wavefront aberrometer. By changing the relative orientation between the probe light polarization and plasmonic nanorods, tunable polarizability can be realized, which in turn leads to changes in their optical properties such as effective refractive indices, scattering and absorption cross-sections, and the gradient and scattering optical forces. Meanwhile, metallic nanoparticles have been widely used in single-molecule detection techniques including surface-enhanced Raman scattering (SERS) and surface-enhanced fluorescence (SEF).
thanks to their near-field enhancement and large scattering cross sections as a result of the plasmonic resonance. It is thus quite natural to examine if electrically tuned plasmonic nanosuspensions could be explored for deep penetration of light and detection of faint signals from low-concentration fluorescent samples.

2. Absorption Modulation

The experimental setup used for our measurement is shown in Figure 1a. Organic gold nanorods (Part #E12-50-800, Nanopartz) are prepared by adding 50 μl of sample in 1 ml of toluene to have a concentration of around $3.7 \times 10^{10}$ ml$^{-1}$. The average length and diameter of the nanorods are 145 and 50 nm, respectively. The corresponding polarizability is simulated, as shown in Figure 1b, where red solid and dashed lines represent the real and imaginary parts of the polarizability parallel to the long axis of a nanorod ($\alpha_\parallel$), respectively. The imaginary part of the parallel polarizability $\alpha_\parallel$ has a peak at around 850 nm, corresponding to the LSPR. The real part of $\alpha_\parallel$ is negative for wavelengths below 850 nm and turns into positive at the LSPR peak. Likewise, blue solid and dashed lines represent the real and imaginary parts of the polarizability perpendicular to the long axis of a nanorod ($\alpha_\perp$), respectively, which is about one order of magnitude smaller than the parallel one. Both the real and imaginary parts of $\alpha_\perp$ are positive, whereas the peak in the imaginary part of $\alpha_\parallel$ around 550 nm corresponds to the transverse surface plasmon resonance (TSPR) along the short axis. The near-field patterns at TSPR and LSPR are shown in the insets of Figure 1b (the near-field strength at TSPR is enlarged 10 times for better visualization).

In the absence of any e-field, the gold nanorods are randomly oriented. Consequently, there are two maxima in the absorbance spectrum (black solid lines) of the nanosuspension, as shown in Figure 1c, corresponding to the TSPR at 550 nm and the LSPR at 850 nm. We first set the polarizer to be $P = 0^\circ$ such that the polarization of incident white light is parallel to the e-field. As nanorods tend to be aligned parallel to the field, the LSPR should dominate the absorption spectrum. As shown by the red solid curve in Figure 1c, the LSPR peak indeed increases and the TSPR peak decreases when the e-field is increased to $E = 0.54$ Vμm$^{-1}$. On the contrary, when the polarizer is set to be $P = 90^\circ$ so that the input polarization is perpendicular to the e-field, the TSPR should dominate the absorption spectrum. This is also confirmed by the blue dashed curve in Figure 1c, where the LSPR peak decreases but the TSPR peak increases when the e-field is increased to $E = 0.54$ Vμm$^{-1}$. Modulation depth (the difference between the absorptions with and without

![Figure 1](image-url)
an external field normalized by the one without external field) is used to quantify the tunability. It is $-0.067$ for the 550 nm peak and 0.14 for the 850 nm peak in the $P = 0^\circ$ case. It is 0.066 for 550 nm peak and $-0.24$ for the 850 nm peak in the $P = 90^\circ$ case, as shown in Figure 1c. As the e-field strength is increased to 1.3 V/μm$^{-1}$ (shown in Figure S1, Supporting Information), the modulation depth increases to $-0.24$ for the 550 nm peak in the $P = 0^\circ$ case and to $-0.46$ for the 850 nm peak in the $P = 90^\circ$ case. Hence, the strength of the LSPR and TSPR absorption peaks can be readily tuned by setting the polarization of the probe light.

3. Tunable Optical Nonlinearity

As established already for dielectric nanoparticles,[6,7,13] a suspended gold nanorod can be attracted toward or expelled away from high-intensity regions of an optical beam, depending on its tuned positive polarizability (PP) or negative polarizability (NP). This is due to the gradient force $F_{\text{grad}} = (1/4)\alpha V|E_0|^2$, where $E_0$ is the optical electric field amplitude and $\alpha$ denotes the real part of the polarizability of a single nanorod.[13] For both polarizability cases, the resulting change in the local refractive index is positive in the high-intensity region and hence a self-induced waveguide can be established in the colloidal nano-suspensions.[6,7,13] Clearly, increasing or decreasing the polarizability through plasmonic tuning should change the nonlinear response of the suspension. The polarizability also depends on nanorod orientation; thus, it should be possible to tune the optical nonlinearity by controlled orientation with an external e-field.

The setup for nonlinear propagation experiments is similar to that in Figure 1a, by just replacing the white light source and the spectrometer with a laser source and a camera, respectively. To increase the strength of the applied e-field and show appreciable diffraction of the propagating beam, a 4 cm-long and 5 mm-wide cuvette is used. The same organic gold nanorod suspension is diluted to a concentration of around $4.7 \times 10^9$ mL$^{-1}$. A laser beam with 790 nm wavelength is used, for which the polarizability is negative, with the largest absolute value of the polarizability marked by the black vertical dashed line in Figure 1b. Figure 2a,b show the transition from a linearly diffracted beam to a stable soliton observed at the exit facet of the cuvette as the beam power is increased from 1 to 40 mW. However, when the power is further increased, the soliton beam cannot sustain and tends to reshape into a ring-like pattern as a result of thermal convection.[16]

To study the dependence of transmission ratio and output beam size on the strength of the applied e-field, we set the input power to be 25 mW, where the transmittance is 1.5% and the output beam size is 89 μm as a reference. The focal spot is around 15 μm so the maximal electric field of the light at the focal spot is estimated to be around $E_{\text{light}} = 822$ V m$^{-1}$, which is around three orders of magnitude smaller than the external field. Hence, the alignment of the nanorods is mainly determined by the external field. However, the external field is uniformly distributed in the cuvette, but along the beam path, the nanorods are

![Figure 2](image-url)

*Figure 2. Experimental results showing output intensity patterns from a) linear diffraction at a pump power of 1 mW and b) nonlinear self-trapping at a pump power of 40 mW. c,d) Measured transmission and output beam diameters through a nanorod suspension as a function of the applied electric field strength at a probe beam wavelength of 790 nm. e,f) Transmission and output beam diameters at a probe beam wavelength of 930 nm. Both the transmission and the output beam sizes are normalized by their corresponding values obtained without any applied field. Unlike the monotonic behavior of the output beam diameter with increasing voltage at probe wavelength 790 nm, there is no obvious trend for the output beam diameter at wavelength 930 nm.*
moved radially by the optical force (which depends on the optical field). The latter is also the key to the formation of the soliton waveguide.\cite{2} The measured applied field increases as a function of the e-field strength is shown in Figure 2c. The transmission decreases as the applied field increases when the polarizer is set at $P=0^\circ$, whereas an opposite trend is observed for $P=90^\circ$. This is simply because the e-field can reorient the nanorods, so absorption around the LSPR peak changes. The normalized output beam size is shown in Figure 2d, which shows a significantly reduced output beam size (over 12%) for $P=0^\circ$ when the e-field increases to 1.0 V μm$^{-1}$. This apparent self-focusing of the beam results from the optical nonlinearity, as the e-field aligns more nanorods to exhibit parallel polarizability (NP) at 790 nm wavelength (see Figure 1b). The underlying physics can be explained by a nonlinear Schrödinger-like equation with an optical force-induced index potential,\cite{2} but here tuning the polarizability is assisted by the external e-field rather than just the optical field of the light beam itself.

For comparison, as shown in Figure 2e,f, a 930 nm laser was used for the same nonlinear experiment where Re($\alpha$) of nanorods is maximal and the optical force is the largest that attracts nanorods into the soliton channel. For an input power of 25 mW, the transmittance is 5.3% and the output beam size is 81 μm in this case. The change in transmission with increasing voltage is roughly the same for both wavelengths, as it is dominated by the external e-field. However, unlike the monotonic behavior of the output beam diameter with increasing voltage at probe wavelength 790 nm, there is no obvious trend for the output beam diameter at the wavelength of 930 nm. The intuitive explanation is as follows. For particles with NP at 790 nm, they are expelled out of the center region of the optical beam, resulting in an area with a higher effective refractive index but fewer particles. Then, if the optical force gets stronger, the center region will get more populated though the effective refractive index increases as well. Due to Ohmic heat, too many gold nanorods in the center region will dampen the power of the laser beam and reduce the optical force. This is a cycle of negative feedback. In this nonlinear process, the output beam diameter is a parameter that is more sensitive than the transmission, which could reveal more underlying dynamics. So in the PP case, the output beam diameter does not change monotonously with the voltage.

4. Fluorescence Enhancement

SERS and SEF are based on the fact that Raman or fluorescence signals can be greatly enhanced by metallic nanoparticles due to the SPR. While these methods are effective in single-molecule detection with a small excitation volume, it is also desirable to increase the detection sensitivity in large sample volumes, where a large total number of molecules are actually involved at the same concentration. As a proof of principle, we conduct a series of experiments in plasmonic nanosuspensions with and without adding the Rhodamine 6G (R6G) molecules using the setup in Figure 1a. The light source is replaced by a 532 nm green laser and a notch filter is added to filter out the laser beam before the detector. Unless otherwise stated, all our fluorescence experiments are done with a pump power of 200 mW and an integration time of 200 ms for the spectrometer. Figure 3a shows the typical absorption (red solid lines) and fluorescence (black solid lines, excited at 532 nm) spectra of R6G in an aqueous solution without any nanorods. The fluorescence spectrum (FS) can be fitted using a dual-emission model consisting of emission peaks at 560 and 590 nm (blue dashed lines), revealing that each spectrum consists of monomer and dimer spectra due to molecular aggregation.\cite{37} In addition, the FS of R6G in a methanol solution is also measured for comparison. Figure 3b shows the emission spectrum of 1 pM R6G in methanol. According to our fitting analysis, besides the dual R6G emission peaks, there are several experimental results: a) The typical absorbance (solid red curve) and FS (solid black curve) of R6G molecules measured in an aqueous solution without nanorods. Curve fitting of the FS reveals two peaks that are located at 560 and 590 nm (blue dashed curves). b) Corresponding FS of 1 pM R6G in a methanol solution. Besides the fluorescence peaks, there are several other emission peaks that correspond to the Raman resonances of methanol (red dashed curves).
other spikes located at 563, 577, 627, 631, and 646 nm. These spikes are related to the Raman signals of methanol. For instance, the spike at 646 nm is related to the O─H stretch (\(\approx 3400 \text{ cm}^{-1}\)), the spike at 631 nm is related to the C─H stretch (\(\approx 3000 \text{ cm}^{-1}\)), and the spike at 563 nm is related to the C─O stretch (\(\approx 1000 \text{ cm}^{-1}\)).[38] In such a transmittance-type volume detection scheme, more molecules are involved in the detection, so in principle even faint fluorescence signals as well as various Raman shifts of the solvent can be detected at the same time.

Then, the FS are measured as a function of the e-field strength, where 2 nM of R6G is mixed with the gold nanorod suspension with a concentration of \(7.4 \times 10^9 \text{ mL}^{-1}\) in methanol. The polarizer is first set at \(P = 0^\circ\), and the FS is shown in Figure 4a. Our results show that the fluorescence signal increases with the e-field. When the polarizer is set at \(P = 90^\circ\), however, we find that the fluorescence signal only increases slightly when the e-field is applied and then saturates when the e-field is further increased. Curve fitting is done to separate the fluorescence envelope from the Raman spikes, where we take the broad envelope peaking at 560 nm to represent the strength of R6G fluorescence and the two spikes at 562 nm and 630 nm to represent the strength of the Raman signal from methanol. The corresponding values are shown in Figure 4c for \(P = 0^\circ\), where the R6G fluorescence envelope increases almost linearly with the field while the Raman shifts do not show a clear trend with respect to the field. The fluorescence signal increases by 5.3 times as the e-field increases from 0 to 1.8 V/\(\mu\text{m}\). Meanwhile, as for the \(P = 90^\circ\) case, the R6G envelope does not change appreciably (with a maximal increase of around 1.3 times). In both cases, there is no obvious change in the Raman peak values at 562 and 630 nm. The nanorods could only affect nearby molecules, whereas the Raman signals are generated by molecules along the entire optical path. As the solvent molecules in the vicinity of the nanorods only constitute a small fraction of molecules residing in the entire optical path, the spectrum could not reflect the change in the Raman signal caused by the reorientation of nanorods due to the applied e-field.

5. Theoretical Analyses

As nanorods rotate along the direction of the external e-field, several optical properties change including the scattering and absorption cross sections and the fluorescence emission rate

![Figure 4](image-url)

**Figure 4.** Experimental results showing R6G fluorescence emission in a methanol solution mixed with a nanorod suspension as a function of the applied field strength when the polarization direction of the pump light is set to a) parallel and b) perpendicular to the applied field. Notice the different y-axis scales in (a) and (b). The fluorescence emission increases significantly with the applied field in (a) but saturates quickly in (b). c,d) Plots showing the normalized peak intensity of the R6G emission along with two Raman peaks of a methanol solution as a function of the applied electric field strength for excitation polarizations parallel (c) and perpendicular (d) to the applied field, respectively.
Figure 5. Theoretical results: a) The scattering and b) absorption cross sections of a single nanorod for \( \theta = 0^\circ \) (solid red curves), 45° (solid black curves), and 90° (solid blue curves). There is no appreciable difference at the fluorescence peak wavelength 560 nm (indicated by a vertical dashed line) for different rod orientations. In contrary, both the scattering and absorption cross sections around 800 nm vary dramatically from \( \theta = 90^\circ \) to \( \theta = 0^\circ \) due to the LSPR. c,d) Calculated FER \( \gamma_{em} \) at 560 nm as a function of molecule nanorod separation \( d \). The insets show four different molecule nanorod configurations: the molecule (indicated as \( p \)) is placed along the short or long axis of a rod oriented parallel or perpendicular to the incident polarization. Blue curves show the calculated ratio of \( \gamma_{em} \) between the perpendicular and parallel cases (left \( y \)-axis), and red curves are normalized to the FER in vacuum \( \gamma_{em}^0 \) (right \( y \)-axis). (e) and (f) are similar to (c) and (d) except that the excitation and emission wavelengths are assumed to be at 800 nm, and the blue curves are for the ratio of \( \gamma_{em} \) between the parallel and perpendicular cases (d) and (e) has the same \( y \)-axis label as (c) and (f), respectively.

(FER) of molecules. In Figure 5a,b, we show the calculated scattering and absorption cross sections of nanorods as a function of the angle between the pump-beam polarization and the long axis of a gold nanorod. It should be noted that the solvent is now methanol with a smaller refractive index as compared with toluene, and the LSPR peak blue shifts to around 800 nm. While both scattering and absorption cross sections increase around two orders of magnitude near the LSPR peak as the nanorod rotates from \( \theta = 90^\circ \) to \( \theta = 0^\circ \), the change in the cross sections around 560 nm is actually very limited, as shown by the vertical black dashed line. These results suggest that the increase in R6G fluorescence is not related to the scattering or absorption cross sections at the R6G emission wavelengths.

On the other hand, the fluorescence rate \( \gamma_{em} \) involves both the excitation rate \( \gamma_{exc} \) and the emission probability presented by the quantum yield \( q_a = \gamma_r / \gamma \), where \( \gamma_{exc} \propto |\mathbf{p} \cdot \mathbf{E}|^2 \) is dependent on the local excitation field \( \mathbf{E} \), \( \gamma_r \) is the radiative decay rate, and \( \gamma \) is the total decay rate. If the molecule has a high intrinsic quantum yield, the nonradiative decay rate is determined by Ohmic loss from the nearby gold nanoparticles. In principle, \( \gamma_{exc} \) should be evaluated at the excitation frequency and \( q_a \) at the emission frequency. The fluorescence rate can be written as

\[
\gamma_{em} = \gamma_{exc}[\gamma_r / \gamma] \tag{1}
\]

The orientation of a dipole emitter is also important. In general, the re-emitted fluorescence under the excitation of a linearly polarized pump light is partially polarized due to the random orientation of the molecular dipoles driven by Brownian motion. Still, the polarization of the fluorescence is mainly parallel to the polarization of the pump light (The polarization anisotropy of R6G in aqueous and methanol solutions is shown in Figure S2, Supporting Information).

The calculated \( \gamma_{em} \) for four typical molecule nanorod configurations is shown in Figure 5c,d (the calculation of \( q_a \), and \( \gamma_{exc} \) is shown in Figure S3, Supporting Information), where \( \gamma_{em}^0 \) is the FER in vacuum. Without any loss of generality, we consider a molecular dipole along the short or long axis of the nanorod as the polarization of the pump light is parallel or perpendicular to the long axis, as shown in the insets. In Figure 5c, we show \( \gamma_{em} \) as a function of the distance between the molecule and the rod along the short axis when the excitation polarization is perpendicular (case (i), red dashed lines) or parallel (case (ii), red solid lines) to the long axis. Because both the excitation and emission wavelengths are close to the LSPR peak, the perpendicular case shows larger FER, where the maximal ratio is around 16-fold for distances less than 5 nm. Similarly, in the cases of molecules along the long axis of the rod, as shown in Figure 5d, \( \gamma_{em} \) of the perpendicular case (case (iii), red dashed lines) is larger than that of the parallel case (case (iv), red solid lines), where the maximal ratio is about 23-fold for distances around 20 nm.

It may seem counterintuitive when one tries to explain our experimental results with these theoretical predications, as our calculations show that the perpendicular case should have larger...
FER. However, we note that for FER at 560 nm, $\gamma_{\text{em}}/\gamma_{\text{em}}^0$ is less than 1 in both cases, a result known as fluorescence quenching due to the damping of emitted fluorescence by the nearby gold nanorods. In addition, we need to take into consideration the nonlinear optical forces exerted on the nanorods. In the parallel case, the nanorod suspension at 532 nm exhibits an NP; thus, the rods tend to be expelled out of the soliton channel due to the gradient force. As a result, the emission property of the R6G molecules is less or not affected by the nanorods, and the soliton channel acts as an effective waveguide for the fluorescence signal. On the contrary, the nanorods in the perpendicular case are attracted to the center of the beam because of the PP. Consequently, R6G fluorescence will be quenched by the nanorods as their FERs are now smaller than that in free space. Hence, we expect that the overall fluorescence to increase steadily, as more nanorods become orientated parallel to the incident polarization, which is consistent with our observation. We note that other chemical effects like voltage-induced molecular structure changes and charge transfer excitations might also play roles in this process.

Finally, the FER for molecules with assumed excitation and emission wavelengths around 800 nm is calculated for both parallel and perpendicular cases, as shown in Figure 5e,f. As the emission wavelength is close to LSPR, the parallel case shows a much larger $\gamma_{\text{em}}$ this time. For cases where the molecules are placed along the short axis of the rod in Figure 5e (case (i) and case (ii)), the ratio $\gamma_{\text{em}||}/\gamma_{\text{em}\perp}$ sharply increases only when the distance is less than 8 nm. On the contrary, when molecules are placed along the long axis, due to the large contrast in the near-field distribution, the ratio $\gamma_{\text{em}||}/\gamma_{\text{em}\perp}$ could be 10^6 or more for distances less than 5 nm. Given that the scattering and absorption cross sections in the parallel case are more than two orders of magnitude larger than those in the perpendicular case, we expect that FS around 800 nm could be tuned with greater extent by changing the orientation of the nanorods through an applied e-field.

6. Conclusion

We have demonstrated tunable optical absorption and nonlinearity of plasmonic nanosuspensions by controlling the orientation of nanorods via an applied e-field. By mixing R6G with gold nanorod suspensions and setting the polarization of the incident light properly, more than fivefold fluorescence enhancement of R6G has been achieved. The e-field-induced orientation of the nanorods and the optical force-induced nonlinearity are found to be responsible for the enhancement, which can be further optimized, for example, by carefully choosing suitable nanorods that have appropriate resonant LSPR peaks. It might also be helpful to mix dielectric particles with plasmonic nanosuspensions to exploit tunable optical nonlinearity to generate soliton channels for efficient guiding and routing of fluorescence signals, which may find applications in biophotonics such as medical diagnosis and deep-tissue imaging. Given that our measurement is based on a transmittance-type volume detection method, more molecules can be involved, promising the detection of low-concentration (but a large total number of molecules as needed) fluorescent samples without the need of using a tightly focused laser beam. The concept and technique may be further adopted and developed for sensing and detection based on plasmonic materials.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

electric-field alignment, fluorescence enhancement, metallic nanosuspension, plasmonic resonance

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