

# Synthesis, characterization and nonlinear optical properties of laser-induced Au colloidal nanoparticles

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Received: 16 March 2012, Revised: 22 July 2012 and Accepted: 26 July 2012

## ABSTRACT

Gold (Au) colloidal nanoparticles (NPs) have been synthesized by direct irradiation of gold precursor ( $\text{HAuCl}_4$ ) solution in polyvinyl alcohol (PVA) matrix using nanosecond laser pulses at different irradiation fluencies and exposure times. Appearance of wine and dark wine colors indicated the formation of Au NPs. Transmission electron microscope (TEM) images exhibited spherical shaped particles and X-ray Diffraction (XRD) pattern confirms polycrystalline nature of Au NPs with cubic structure. Au NPs displayed negative nonlinearity and exhibited a shift from saturable absorption (SA) at lower intensities to reverse saturable absorption (RSA) accompanied by nonlinear scattering at higher excitation intensities in nanosecond regime at 532 nm. Two-photon absorption (TPA) is attributed to be responsible for the RSA behavior. Copyright © 2013 VBRI press.

**Keywords:** Gold colloidal nanoparticles; UV-visible absorption spectrum; surface plasmon; TEM, saturable absorption; reverse saturable absorption.



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## Introduction

In recent years the interest in the preparation and characterization of nanostructured materials has grown constantly because of their distinctive properties and potential use in technological applications [1]. Fundamentally new properties and functions are expected from these materials resulting from their small sizes and modified electronic structures. Gold (Au) nanoparticles (NPs) have drawn a great deal of attention in various areas of research including optics, electronics, catalysis, and biomedicine due to their unique optical [2], electrical [3] and chemical properties [4], as well as their biocompatible nature [5]. As these properties are strongly dependent on the structure (e.g., size and shape) of gold NPs, many synthetic strategies have been developed to control the size and the shape of the NPs [6-8]. The controlled synthesis of Au NPs of a desired size, in particular, has become an important aspect in the emerging basic field of colloid- and nano-based chemistry [6, 9, 10]. The optical properties of noble metal particles with their intense colors have fascinated scientists since the turn of this century [11]. For example, spherical Au NPs show a strong absorption band in the visible region of the electromagnetic spectrum at about 520 nm. This absorption, called the plasmon absorption, is absent for very small particles (2 nm and smaller) as well as for the bulk gold. It originates from the oscillation of the free electrons (6s-electrons of the

conduction band in the case of gold). Mie first described this phenomenon theoretically by solving Maxwell's equations for a radiation field interacting with a spherical metal particle under the appropriate boundary conditions [11]. Au NPs are known for their beautiful dark wine coloration which is ascribed to transverse oscillations of surface electrons of the particle on interaction with light of suitable wavelength, the surface plasmon resonance (SPR), whose position greatly depends on the shape and size of nanoparticles. Also, Au NPs have been known to exhibit high third-order nonlinear optical coefficient with an ultrafast time response [12] and are subjected to intensive studies involving optical nonlinearities [13]. Optical limiting, where the transmitted light through a medium is limited to a threshold, is usually attributed to effects such as reverse saturable absorption (RSA), nonlinear refraction and thermal nonlinearities and scattering. The self healing ability of solution based optical limiters makes them auspicious materials for real world applications, such as the protection of optical sensors from intense laser sources. These nascent technologies rely in part on the excellent inelastic light-scattering properties of Au NPs [14] and also on the shift of the plasmon band maxima with local dielectric constant [2]. Au NPs were prepared through different techniques in the past. Some of them are: chemical routes including the use of chemical reductants in both aqueous and nonaqueous solvents [6, 15], electrochemical [16], sonochemical [17], radiolytic [18] and photochemical methods [19] with different purpose of application. For the preparation of size-controlled colloidal gold particles, the radiolytic and photochemical methods which involve irradiation with light have recently aroused much attention because of the following advantages: (i) controlled reduction of metal ions can be carried out without using excess reducing agent; (ii) radiation is absorbed regardless of the presence of light-absorbing solutes and products; (iii) the reduction reaction is uniformly performed in the solution. In order to prevent the aggregation of the particles it is usually required to add protective agents such as soluble polymers, and surfactants in the photochemical methods [19].

Of all the types of stabilizers used in the synthesis, polymers hold great promise for effectively controlling the metal nanostructures. Generally speaking, polymer-stabilized metal NPs are synthesized by chemical or irradiated reduction of metallic precursor in the solution of polymer or monomer. Reduction of metal ions always involves the transfer of electrons from one species to another. Reducing agents, such as sodium borohydride [20] or hydrazine [21] are commonly used as electron sources in the chemical reduction. Transferring electrons involved in the irradiation reduction mainly come from solvated electrons or free radicals produced by irradiation of aqueous or alcoholic solutions with  $\gamma$  -rays [22] and ultraviolet or visible light [23]. In these cases, polymers added or prepared from certain monomers usually serve as protective agents or templates to both control the particle size and passivate the particle surface against agglomeration.

Here we report a remarkable "Laser irradiation" technique which is a bottom-up approach in comparison to the approach of laser ablation of bulk materials in solution

for producing NPs. The present syntheses involved in irradiation of metal salts in polyvinyl alcohol (PVA) matrix, where PVA acts as both reducing and stabilizing agent [24], thereby generating homogeneous distribution of the metal NPs. Moreover, this technique of synthesizing NPs has the following interesting features: very simple and convenient, synthesized NPs have regular spherical shapes and crystallized NPs can easily be obtained in one-step procedures without subsequent heat-treatments, because of the high energetic state of irradiated species without formation of by-products. Resizing and reshaping of colloidal NPs synthesized by other methods are also possible through melting and fragmentation technique by laser irradiation [25].

With all the above mentioned advantages, here we present the novel technique of synthesizing Au colloidal NPs through laser induced heating of precursor solution by directly irradiating Au precursor solution with pulsed nanosecond laser in the presence of PVA matrix. The NPs were found to be stable even after several weeks from synthesis. We also observed no variation in the optical properties of nanoparticle solutions with time. No significant changes were detected in the particle size distributions by TEM during this period as well. We observed very strong nonlinear absorption along with nonlinear scattering in ns (532nm) regime from the Au nanoparticles in PVA matrix.

## Experimental

Gold (III) chloride trihydrate ( $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ ) and Poly vinyl alcohol (PVA), were purchased from Aldrich and all the chemicals were used without further purification. Deionized water was purified to a resistivity of 18.2  $\text{M}\Omega\text{-cm}$  for use during the synthesis. All glassware were cleaned with an aqua regia solution (3:1,  $\text{HCl}:\text{HNO}_3$ ), and then rinsed with deionized water prior to use. 5 mM  $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$  in deionized water was added to 1 wt% PVA and the solution was vigorously stirred. A pulsed Nd:YAG laser (Spectra-Physics, USA) was used to irradiate the colloidal solutions at 532 nm of 6 ns pulse width, 8 mm beam diameter and a repetition rate of 10 Hz.

Measurement of size and surface morphology of colloidal Au nanoparticles were carried out by UV-Vis spectrophotometer (JASCO V-670), powder XRD (Philips PW 1830 X-Ray diffraction spectrometer with Co target) and FEI model TECNAI G<sup>2</sup>20 S-Twin TEM instrument. TEM pictures at accelerating voltage of about 200 kV have been taken by placing a drop of Au colloidal solution on a copper grid and drying under ambient conditions.

Nonlinear absorption studies were carried out with the ns laser (532 nm) using open aperture Z-scan technique for Au colloidal nanoparticles in PVA solution. A frequency doubled, Q-switched Nd: YAG (yttrium aluminum garnet) laser (Spectra-Physics, INDI 40, 532 nm, 6 ns, 10 Hz) was used as the excitation source. The general experimental set up on Z-scan technique has been described elsewhere [26]. Briefly, in a typical Z-scan experimental setup, a laser beam with a transverse Gaussian profile is focused on to the sample using a lens. The sample is then moved along the propagation direction of the focused beam. At the focal point, the sample experiences maximum pump intensity,

which gradually decreases in either direction from the focus. An  $f/40$  configuration was used for the present studies. Apertures were introduced in the path for beam shaping and calibrated neutral density filters were used to vary the laser intensity. Au nanoparticles solution was taken in a 1mm cuvette which is smaller than the Rayleigh range of the focused beam calculated to be  $\sim 4$  mm. Value of beam waist at the focus is  $25 \mu\text{m}$  and the peak intensities inside the sample vary from  $10^8$  to  $10^9 \text{ W cm}^{-2}$ . Data is then recorded by scanning the sample cell across the focus and collecting the data through a SRS boxcar averager (model SR250), output of which is given to a personal computer through an analog-to-digital (ADC) card. The nonlinear absorption coefficient ( $\alpha_2$ ) was obtained by fitting the nonlinear transmission expression of an open aperture Z-scan using the following relation [26].

$$T_{OA(2PA)} = 1 - \left[ \frac{\alpha_2 I_0 L_{\text{eff}}}{\frac{8}{2z} \left[ 1 + \frac{z^2}{Z_0^2} \right]} \right] \quad (1)$$

where  $Z$  is the sample position,  $Z_0 = \pi \omega_0^2 / \lambda$  is the Rayleigh range,  $\omega_0$  is the beam waist at the focal point ( $Z = 0$ ),  $\lambda$  is the laser wavelength,  $I_0$  is the intensity on the sample at focus, and effective optical path length in the sample of length  $L$  is given as  $L_{\text{eff}} = [(1 - e^{-\alpha_0 L}) / \alpha_0]$  with  $\alpha_0$  as the linear absorption coefficient at  $532 \text{ nm}$ .

## Results and discussion

Experimental set up for synthesizing Au colloidal NPs under laser irradiation is shown in Fig. 1. During irradiation a magnetic stirrer was used to prevent gravitational settling of the previously formed particles. Au NPs thus prepared show wine and dark wine colors. Also it was confirmed through UV-visible absorption spectrometer that the particles exhibited surface plasmon peak.

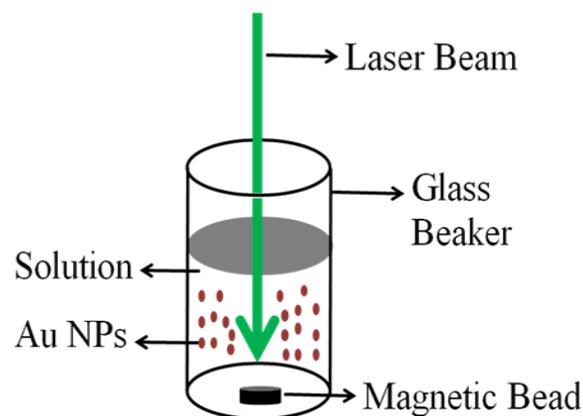


Fig. 1. Schematic Diagram of experimental set up used in synthesizing Au colloidal NPs.

Fig. 2(a) and 2(b) show the UV-Vis absorption spectra recorded from aqueous PVA-Gold (III) chloride trihydrate medium as a function of laser fluence and exposure time of ns laser pulse before and after irradiation at  $532 \text{ nm}$  wavelength, respectively. Plasmon peak was not observed

for pure solution before laser irradiation which can be noticed in Fig. 2(a). Au SPR peak has been observed to occur around  $525 \text{ nm}$  and steadily increased in intensity as a function of energy fluence without any shift in the plasmon peak wavelength with 45 minutes duration of exposure upon ns laser irradiation is shown in Fig. 2(a). Observed increase in plasmon peak intensity might be due to the increase in input irradiation fluence leading to the formation of larger number of nanoparticles. Fig. 2(b) shows UV-Vis spectra obtained as a function of exposure time at  $185 \text{ mJ/cm}^2$  fluence before and after irradiation. SPR band was observed around  $525 \text{ nm}$  and it steadily increased in intensity as a function of exposure time without any shift in the peak wavelength as in the former case. This indicates that the formed Au NPs are of same size for different energies and exposure times under study. Au colloidal NPs were seen to form within 15 minutes of exposure upon irradiation and this was confirmed by color change from pale yellow to wine and dark wine upon 60 minutes of exposure as shown in Fig. 3. Their absorption spectra are shown in Fig. 2(b).

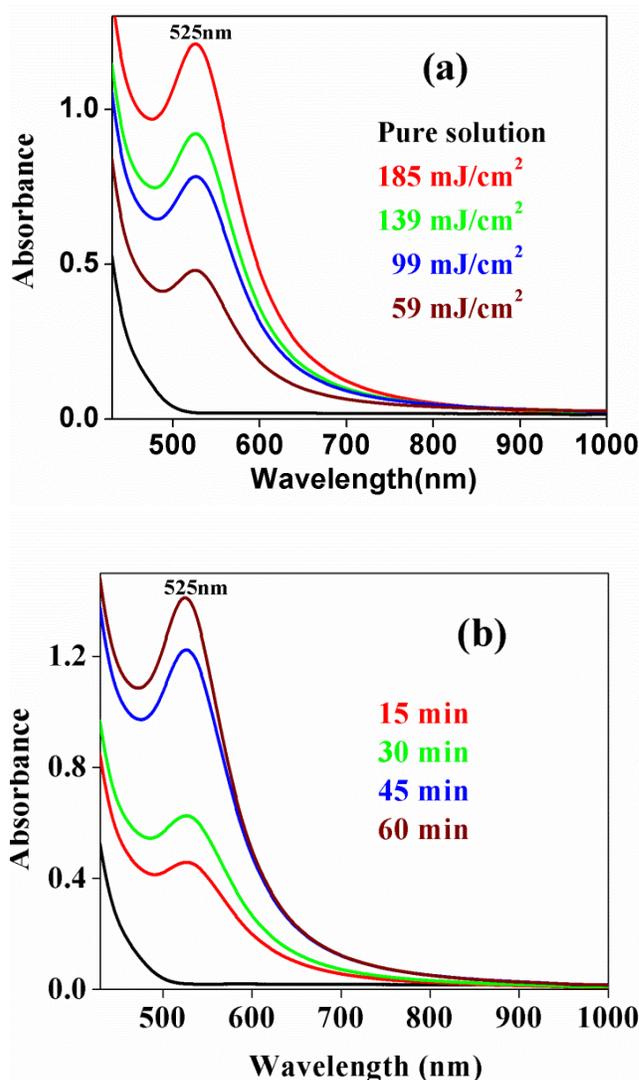
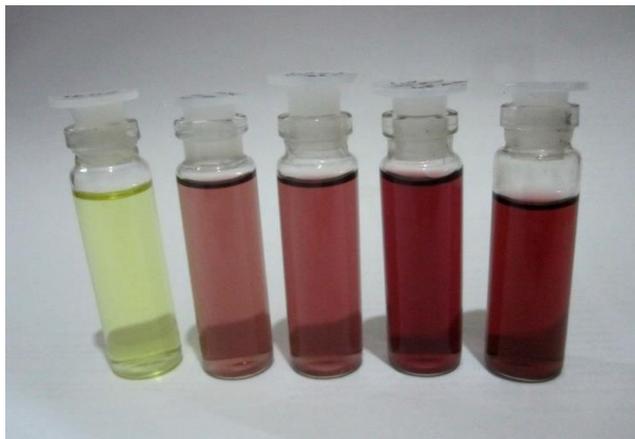
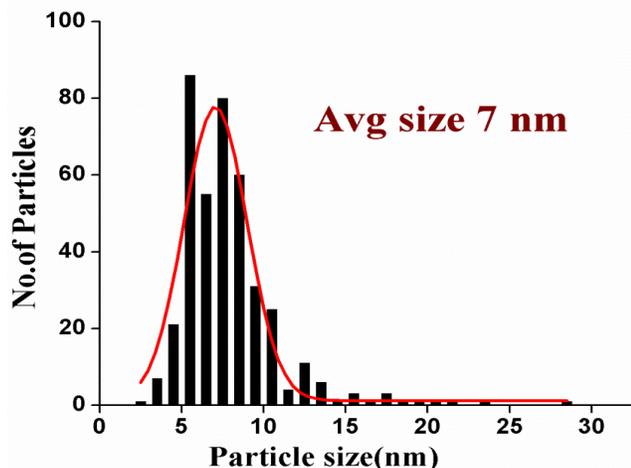
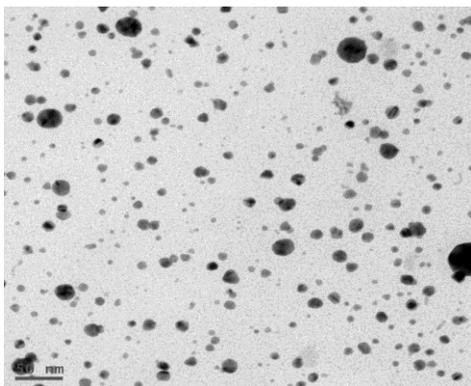


Fig. 2. UV-Vis absorption spectra of colloidal Au NPs with ns laser irradiation at  $532 \text{ nm}$  (a) at different fluences under 45 minutes duration of exposure (b) at different irradiation times with  $185 \text{ mJ/cm}^2$  fluence.

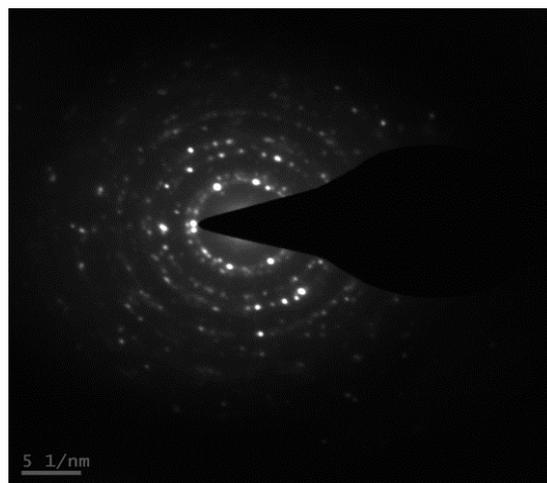
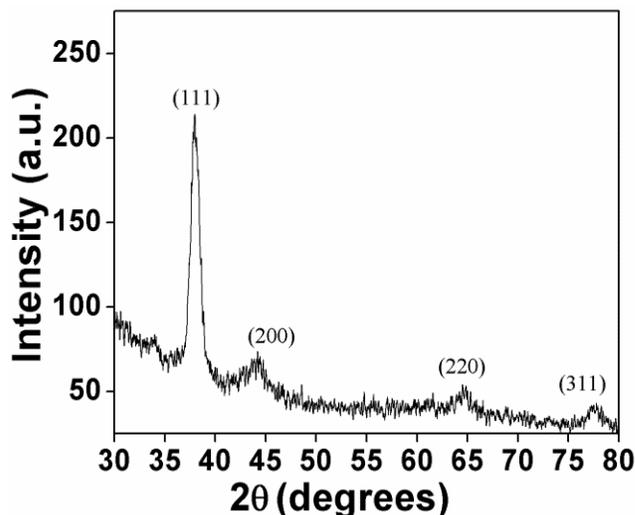


**Fig. 3.** Formation of Au colloidal NPs synthesized upon laser irradiation. Color change from pale yellow (unirradiated solution) to wine and dark wine can be observed from left to right with increase in exposure time from 15 to 60 minutes.

**Fig. 4** shows representative TEM images recorded from drop-coated copper grids of Au colloidal NPs synthesized under ns laser irradiation at  $185 \text{ mJ/cm}^2$  fluence for 60 minutes exposure time. The histogram taken from a large number of micrographs is also shown in Fig.4. It is evident that the diameter of particles ranges from 2 to 28 nm and average size has been estimated to be 7 nm.



**Fig. 4.** TEM images of Au colloidal NPs under ns laser irradiation at  $185 \text{ mJ/cm}^2$  fluence for 60 min exposure, and corresponding Histogram of Au NPs under study.



**Fig. 5.** XRD pattern and Electron diffraction pattern of Au colloidal NPs under study.

**Fig. 5** shows XRD pattern and electron diffraction patterns obtained for Au colloidal NPs synthesized upon ns laser irradiation with  $185 \text{ mJ/cm}^2$  fluence and 60 minutes exposure time. XRD pattern clearly shows that the Au NPs formed by the reduction of  $\text{AuCl}_4^-$  by ns laser irradiation are crystalline in nature with cubic structure. Electron diffraction patterns of Au NPs under study also exhibiting (111), (200), (220) and (311) rings indicating fcc structure of polycrystalline Au NPs which agrees well with XRD pattern.

As the Au NPs play a very important role in the nonlinear absorption [13], we carried out the Z-scan studies with ns laser. In ns regime we observed very interesting results that show a change in the absorption and scattering properties of Au colloidal NPs with laser irradiation. **Fig. 6(a)** and **6(b)** show the open aperture Z-scan data recorded with the ns laser. One can see from **Fig. 6(a)** that at low laser intensity  $I_0 = 164 \text{ MW/cm}^2$ , the data shows a saturation behavior and as the laser intensity is raised to  $I_0 = 326 \text{ MW/cm}^2$ ; one starts observing the strong two photon absorption (TPA) which is shown in **Fig. 6(b)**. Open circles represent experimental data and solid lines represent theoretical fits.

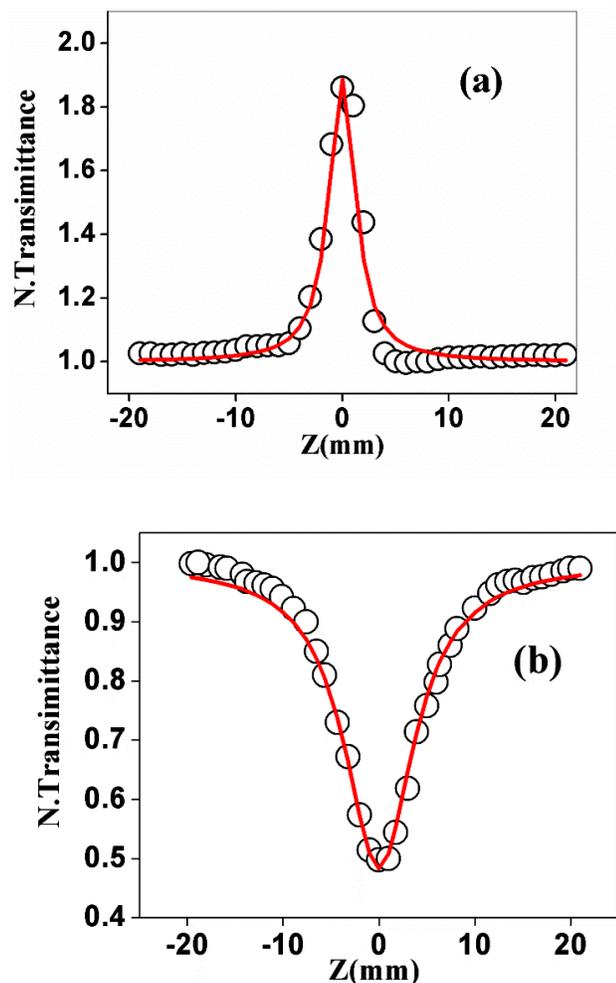


Fig. 6. Open aperture Z-scan curve (open circles) of Au NPs using ns laser at 532 nm at intensity of (a) 164 MW/cm<sup>2</sup> (SA) (b) 326 MW/cm<sup>2</sup> (RSA). Solid curves represent theoretical fits.

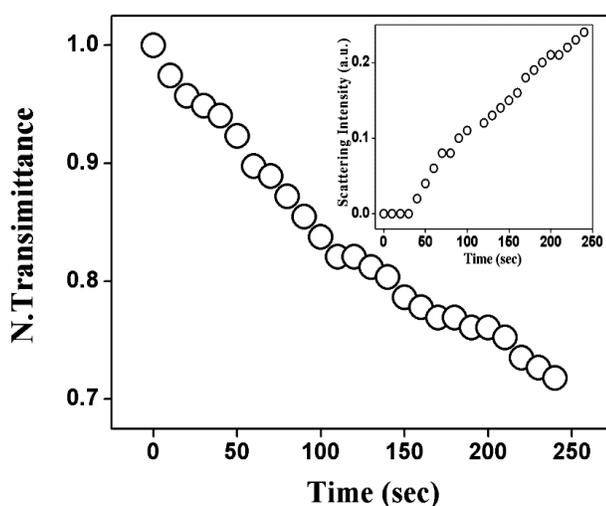


Fig. 7. Nonlinear transmittance data obtained for pure solution as a function of time and inset shows the scattering as a function of time.

We also recorded the transmission and scattering data with pure HAuCl<sub>4</sub>+PVA solution kept at focal point with

time for an incident intensity of  $I_0 = 326 \text{ MW/cm}^2$ . The data is shown in the Fig. 7, which clearly shows decrease in transmittance indicating the formation of NPs with time. Inset of Fig. 7 shows scattering recorded with time which was measured by placing the detector at an angle of 10° with respect to laser beam propagation. Scattering (inset of Fig. 7) starts appearing after ~ 40 sec which was due to the formation of NPs. Decrease in transmittance and increase in scattering with time as shown in the Fig. 7 indicate the formation of nanoparticles within few tens of seconds due to high intensity at the focus. From theoretical fit of transmittance curve using equation (1), the two-photon absorption coefficient ( $\alpha_2$ ) was estimated as 49 cm/GW at an intensity of 326MW/cm<sup>2</sup>. We have also collected scattering data at different intensities with detector placed at an angle of 10° with respect to the laser beam propagation, which is shown in Fig. 8. At lower intensities, where the sample exhibited SA behavior, we did not observe any scattering. However, at higher intensities where Au nanoparticles are exhibiting RSA behavior, we observed strong nonlinear scattering. Thus the gold nanoparticles in the PVA matrix can effectively be used as either saturable absorbers or as optical limiting materials by casting the solutions into thin films. The SA behavior is attributed to surface plasmon absorption bleaching and RSA is ascribed to excited state absorption, TPA and nonlinear scattering.

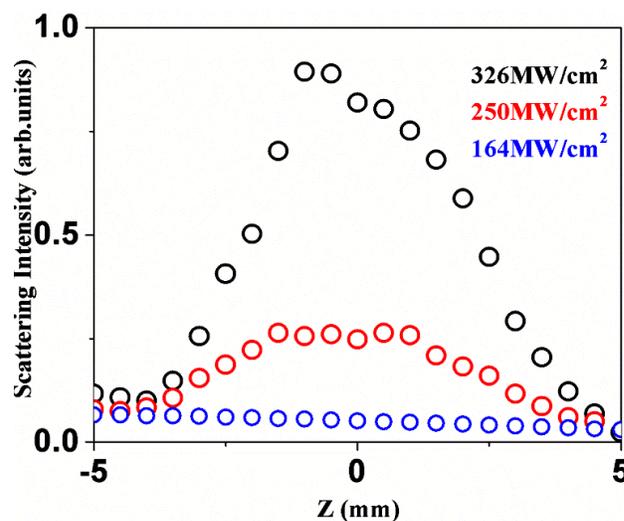


Fig. 8. Scattering of Au nanoparticles at different intensities.

Issues such as monodispersity and shape selectivity for obtaining phase pure monodisperse NPs are yet to be addressed and focused on. Hence, present study opens up a new possibility of very conveniently synthesizing metallic/bimetallic colloidal NPs using lasers. Achievement of such rapid time scales for synthesis of metallic nanoparticles in polymer matrix without agglomerations for a long period contributes to an increase in the efficiency of synthetic procedures using lasers as an alternative to other chemical synthesis protocols. This technique is simple and environmentally friendly because we do not use any chemicals except metal salt and PVA polymer and also it differs from other laser process such as the formation of

metal nanoparticles by pulsed laser ablation of a solid-state metal target immersed in a liquid.

## Conclusion

In conclusion, an innovative and convenient approach in synthesizing stable colloidal nanometer-sized Au particles in PVA matrix with a pulsed nanosecond laser has been demonstrated. The irradiation over extended periods leads to a color change from wine red to dark wine, observed physically and also through the surface plasmon peak around 525 nm confirmed the formation of Au NPs. TEM images reveal that the size of Au NPs are less than 30 nm predominantly with a spherical shape. Nonlinear absorption studies through Z-scan technique reveal that the Au colloidal NPs exhibited a switching behavior from SA to RSA with intensity along with nonlinear scattering at higher intensities.

## Acknowledgements

R. Kuladeep thanks University Grants Commission (UGC), India, for financial assistance. K. Shadak Alee acknowledges the senior research fellowship from Council of Scientific and Industrial Research, India. D. Narayana Rao acknowledges financial support from the Department of Science and Technology, India. This research is performed in the framework of ITPAR Phase II FaStFal 2007-2010 project.

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