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PHOTOTHERMAL CONVERSION CHARACTERISTICS OF GOLD NANOPARTICLE DISPERSIONS

Hui Zhang[a,b], Hui-Jiuan Chen[b], Xiaoze Du[a], Dongsheng Wen[b,c*]

[a] School of Energy and Power, North China Electric Power University, China

[b] School of Engineering and Materials Science, Queen Mary University of London, U.K

[c] School of Process Environmental and Materials Engineering, University of Leeds, U.K.

Correspondent author: Dongsheng Wen

Correspondent Address: School of Process Environmental and Materials Engineering, University of Leeds, LS2 9JT, U.K. Email: d.wen@leeds.ac.uk; Tel: 0044 113 3431299

ABSTRACT

This work proposes and validates a novel idea of using plasmonic nanoparticles (PNP) to improve the solar thermal conversion efficiency. Gold nanoparticle (GNP) is synthesized from an improved citrate-reduction method, and used as an example to illustrate the photothermal conversion characteristics of PNPs under a solar simulator. The experimental results show that GNP has the best photo-thermal conversion capability comparing to other reported materials. At the lowest particle concentration examined (i.e., 0.15 ppm), GNP increases the photo-thermal conversion efficiency of the base fluid by 20% and reaches a specific absorption rate (SAR) of ~10 kW/g. The photo-thermal conversion efficiency increases with increasing particle concentrations, but the SAR shows a reverse trend, which is unexpected as all GNPs should be still in the independent scattering regime.

Keywords: Plasmonic nanoparticle, gold nanoparticle, nanofluids, solar energy, photo-thermal conversion, solar collector

1 INTRODUCTION

Developing sustainable energy technologies, especially solar energy related, becomes extremely important in securing our energy future. Nanoparticle-based direct absorbing solar energy collector (DASC) is a recent development, which employs nanoparticles to convert light energy into thermal energy directly. Comparing to conventional solar thermal collectors that rely on the transfer of heat through the wall, nanoparticles absorb solar energy directly within the fluid volume. Such an idea transfers the surface heat transfer limitation associated with conventional solar collectors into a volumetric absorption phenomenon. An optimized DASC system could not only simplify the conventional system, i.e. replacing metal pipes with transparent glass tubes, but also increase the absorption efficiency through proper engineering the absorption spectrum at the nanoscale.

Significant advance has been achieved in this field since the concept was first proposed. The photothermal conversion efficiency of a range of nanomaterials were experimentally investigated [1-6] and a few theories were proposed to maximize the conversion efficiency under optimized particle concentrations [2,7,8]. These studies show that certain nanoparticle can absorb sun light directly, which may pave the way for a new generation of solar thermal energy system. However there are still a number of issues that requires special attention: i) only limited nonmaterials have been investigated and it is still unclear about the optimized material and particle concentration; ii) it is unclear about the photothermal conversion mechanisms, especially regarding the interaction between nanoparticles, the solar light and the base fluid, and iii) a lot of studies were based on the optical properties and characterized by the extinction coefficient [2,3,4,8,9], with limited direct photothermal conversion experiments. The extinction coefficient is a key factor to illustrate the wavelength-selective absorbing feature of nanoparticles; however it may not represent the real photo-thermal energy conversion process. For any application in solar thermal systems, it is the temperature rise that decides the overall efficiency. In addition, the lack of proper control in nanomaterials (i.e., size, shape and concentration) makes it is difficult to study the particle morphology effect and to compare the results among different research groups. Most of the nanoparticles were purchased in the commercially with wide size distribution in the form of agglomerations. The presence of surfactant / dispersant in the dispersion may influence the absorption process significantly.

It is known that commonly-used fluids for solar thermal applications such as water and ethane glycol have good absorption efficiency in the infrared regime. Consequently particles that have strong absorption in the visible light, not much in completion with the base fluid, should be considered more favourably. Plasmonic particles have been widely used the medical and life science fields, ranging from gene therapy, controlled drug delivery, enhanced imaging and diagnosis, and non-invasive thermal therapies [10-13]. Significant heat can be induced through the surface plasmon resonance process (SPR), where the absorption from the nanoparticle could be greatly enhanced via the coupling of the incident radiation with the collective motion of electrons in metal. As the plasmon resonance frequency is typically comparable with the visible light spectrum, it appears that plasmonic particles could be good candidates for direct photothermal conversion to enhance the solar thermal efficiency especially in the visible light spectrum. Using gold nanoparticle as an example, this paper will investigate the direct photo-thermal conversion characteristics of plasmonic particles. To elucidate clearly the effect of particles, GNPs will be synthesized based on an improved citrate-reduction method followed by a rigorous purification procedure. The photothermal conversion study of various GNP concentrations will be performed under a solar simulator. The specific absorption rate of GNPs will be calculated and compared with other published studies.

2 EXPERIMENTAL APPROACH

2.1 Gold nanofluids formulation and characterization

In this work, gold nanoparticle dispersions are formulated through simultaneous production and dispersion of nanoparticles in situ. GNPs were synthesized by the citrate reduction method with the aid of ultrasonification for particle morphology control [14]. In brief, a mixture of 190 ml deionised water (DI) and 5.0×10^{-6} mol of $HAuCl_4$ was heated until boiling, and it was subjected to heating and stirred using a magnetic blender. After 10 ml of 0.5% sodium citrate was added and the solution's colour changed to wine-red, the preparation was finished after the solution was placed in the ultrasonic bath at 80°C for 30 min. Gold nanoparticle dispersions were purified by the membrane dialysis method. In this process, 100 ml of GNP dispersion was put in a membrane tube with pore size of 2-3 nm in diameter, which allows the smooth diffusion of ions but keeps GNPs inside. The

membrane was located in flask of DI water ~ 2000 ml, stirred by a magnetic stirrer. The DI water was changed twice per day and the purification process lasted for 5 days. During this process, the concentrations of various impurities diminished exponentially with the times of DI water change, which resulted in a negligible impurities presence in the dispersion and confirmed by the UV-Vis spectrum measurement. This ensures that the photothermal conversion effect we will study below is solely due to the effect of gold nanoparticles. .

Atomic absorption spectrometer (Varian 220FS SpectrAA Atomic Absorption Spectrometer & GTA 110) was used to measure the concentration of obtained gold dispersions. The concentration of the original GNP dispersion was determined as 32 mg/L based on the flame and electrothermal atomization techniques. For both unpurified and purified samples, the original dispersion was diluted into four different concentrations: 150.0, 75.0, 33.3, and 11.1 μmol/L and with 3 minutes ultrasonification. The size and shape of gold nanoparticles were identified by a transmission electron microscopy (TEM) equipped with an Energy Dispersive X-ray spectroscope (EDX). In the operation, the TEM was performed with a Jeol JEM-2010 electron microscope at a bias voltage of 200 kV. The particle size distribution in liquid was identified by a dynamic light scattering (DLS) device (Malvern nanosizer). The resulting dispersion of gold nanoparticles presents in clear red-wine color and TEM images of gold nanoparticles are shown in Fig.1. The gold nanoparticles are spherical with diameters in the range of 15 nm to 20 nm. Fig.2 presents the size contribution of GNPs in DI-water, which is in the range of 10~30 nm. The DLS measured value is slightly larger than the TEM results, which is due to the nature of the DLS measurement. A nearly full dispersed particle status in the liquid is presented in the DLS result.

2.2 Photothermal conversion experiment

The photothermal conversion experimental setup is shown in Figure 3. In order to minimize the experimental uncertainties under direct sun light, a solar simulator (Newport Co. Oriel Xenon Arc lamp) was used as the light source. It provides a close spectral match to the solar spectra, and can vary the radiation intensity between one and two AM. The performance parameters of the solar simulator are based on the ASTM standard [15], including spectral match (fraction of ideal percentage) of 0.7~1.25, non-uniformity of irradiance of up to 5% and temporal instability of up to 5%. To minimize the temperature gradient inside the fluid, a thin layer of gold nanoparticle

dispersion (~3mm) was put in a Petri dish (3.5mm diameter), which was located in the centre spot of the solar simulator. A uniform radiation from the simulator can be assumed. The top of the Petri dish was covered by a glass plate to reduce the radiation from the sample. The centre sample temperature was measured by a K-type thermocouple (Omega 5TC-TT-K-36-36), whose head was fixed on the bottom centre of the Petri dish. The data was recorded in a PC though a data acquisition hardware (thermocouple input devices, NI, USB-9211, 4-Channel, 24-bit) under the Labview environment. Preliminary tests showed that the space variation of the sample temperature is small. The uncertainty of the temperature was estimated as ±0.25°C. Sample fluid was injected slowly through a cylindrical injector into the Petri dish before each experiment.

3 RESULTS ANALYSIS

The absorption spectrum of gold dispersion at different concentrations was firstly measured using an UV/Vis spectrometer (PerkinElmer, lambda 35). The ratio of radiant power transmitted (P) by a sample to the radiant power incident (P0) on the sample is defined as the transmittance, i.e., P/P0. The absorbance is defined as the logarithm (base 10) of the reciprocal of the transmittance, Log (P0/P), through the Beer's law,

$$\log\left(\frac{P0}{P}\right) = \varepsilon \emptyset L \tag{1}$$

in which ε is the extinction coefficient, $\emptyset$ is volume fraction, and L is the distance of light traveling through the sample. The absorbance of different concentrations of gold dispersions is shown in Figure 4. All samples exhibit similar absorption pattern. The peak absorption wavelength is about 520 nm, and is independent on the particle concentration. Such a value is consistent with the result of spherical gold nanoparticles in the size range of 10~30nm. The absorption peak can be shifted to longer wavelength by engineering gold particles into shapes with large aspect ratios, a practice used frequently in the medical field to shift the absorption peak to the biological infrared window.

An air mass of 1.5, (AM =1.5), which represents the solar spectrum at mid-latitudes, is used in the photothermal conversion experiments. At AM=1.5, the solar intensity (I) is 1000 W/m^2 according to

the standard of ASTM G-173. The sample fluids were heated under the fixed solar intensity for 300 seconds. The transient temperature curves were shown in Figure 5a, which clearly shows that low concentrations of gold nanoparticles can significantly increase the bulk temperature. For example, the bulk temperature increased by ~13 K for a GNP concentration of 0.0112% after 300 seconds irradiation, whereas the temperature increase is ~8 K for the pure water. The bulk temperature increases with the increase of particle concentrations, but exhibiting a non-linear feature. At the initial heating stage, the temperature increases nearly linearly with the radiation time, but deviates slightly from the linear feature at the late stage due to the heat dissipation to the surroundings .

Under low continuous radiation conditions (as in this work), the temperature difference between the particle and the fluid is very small. The temperature of the gold nanoparticle can be assumed to the same as the surrounding fluid, measured by the thermocouple. As a simple estimation, it can be assumed that the fluid have the same temperature in space under the current experimental setup (i.e., uniform solar radiation and small fluid depth). The photothermal conversion efficiency (η) can then be calculated by:

$$\eta = \frac{(c_w m_w + c_n m_n)\Delta T}{IA\Delta t} \tag{2}$$

in which c_w and c_n are the specific heat of water and gold respectively; m_w and m_n are the mass of water and gold respectively; ΔT is the temperature rise in the Δt time interval, and A is the illumination area of fluid in the experiment. As the particle concentration is so small, i.e, $\frac{c_n m_n}{c_w m_w} \sim 0$, the photothermal conversion efficiency can be simplified as:

$$\eta \approx \frac{c_w m_w}{IA} \cdot \frac{\Delta T}{\Delta t} \tag{3}$$

The efficiency is directly proportional to the temperature rise gradient. Using the initial temperature rise gradient as an example (i.e. the first 60 second when the heat leak is negligible), the photothermal conversion efficiency is calculated, shown in Figure 6. Remarkable performance of gold nanoparticles is observed even under very small particle concentrations. Comparing with the base fluid, the efficiency increases by nearly 10% for the smallest gold nanoparticle concentration (0.00028%, or ~ 0.15 ppm). The photothermal conversion efficiency increases nearly linearly with

the nanoparticle concentration up to 0.0014%, and then levels off at higher particle concentrations. The maximum photothermal conversion efficiency reaches 78% at 0.01% particle loading (~ 6 ppm).

It should be noted that very dilute gold nanoparticle dispersions were used in the experiment, i.e., below 6 ppm (max volume fraction), and the gold nanoparticles were uniformly dispersed in the base fluid. A simple estimation shows that the l/d value, i.e., the ratio of the particle-particle distance (l) to the particle diameter (d), is in the range of 10~100. It is reasonable to consider that the particle–particle interaction should not be important. Gold nanoparticles should act independently in absorbing solar energy according to the scattering regime map [16]. Within such a dilute concentration range, it should be reasonable to predict that the enhancement in the photothermal conversion efficiency, defined as $(\eta_n-\eta_w)/\eta_w \cdot 100$ (%), shall increase linearly with the particle concentration. However Figure 6b shows a different story. Significant enhancement is observed at the lowest particle concentration, and the enhancement ratio increases with the particle concentration linearly up to 0.0014%. The linearity is broken at higher particle concentrations, implying that the photothermal conversion capability per nanoparticle decreases at higher particle loadings.

The specific absorption rate (SAR) describes the particle's capability in absorbing energy per unit mass, which can be calculated as:

$$SAR = \frac{(c_w m_w + c_n m_n)\Delta T_n - c_w m_w \Delta T_w}{1000 \cdot m_n \Delta t}, \text{ [kW/g]} \quad (4)$$

in which ΔT_n and ΔT_w was the temperature rises at the same Δt time interval for water and nanofluids respectively. Again, as $(\frac{c_n \Delta T_n}{1000 \cdot \Delta t} \sim 0)$, the SAR calculation can be simplified as:

$$SAR \approx \frac{c_w m_w}{1000 \cdot m_n}\left(\frac{\Delta T_n}{\Delta t} - \frac{\Delta T_w}{\Delta t}\right) \quad (5)$$

Clearly SAR is proportional to the temperature rise gradient. Again using the initial temperature as an example, Figure 7 shows SAR at different particle concentrations. The highest SAR is observed at the lowest particle concentrations, reaching 10 kW per gram of gold nanoparticle. The SAR value decreases from 10 to about 1 kW/g (i.e. a factor of 10) when the particle concentration increases

from 0.00028% to 0.0112%. The value of SAR for gold nanoparticle under such a low heat flux is larger than many magnetic nanoparticles under high magnetic fields, which typically in the order of one hundred Watt per gram of magnetic nanoparticle [17-19].

Clearly one interesting question to ask is why the conversion efficiency of individual gold nanoparticle decreases with the increase of particle concentration in a region that the particle–particle interaction is negligible? One possibility is the particle agglomeration. It would be conveniently to consider that the number of individual particles may not increase proportionally with the increasing particle concentrations. This is, however, unlikely the case as the highest concentration used in the experiments is only 6 ppm and the size and surface properties of the gold nanoparticles warrant the stability of the dispersion. Both DLS measurement and electron microscopy studies also show that there is a negligible agglomeration phenomenon. Nanoparticles should still be in the independent scattering regime. Another possibility is related to the effect of the fluid thickness on the volumetric absorption of gold nanoparticles. The absorption efficiency of each particle shall be different at different fluid depths. The single temperature measured in the experiments may not represent the overall thermal performance of the fluid. If there was a non-uniform temperature profile, the radiation heat transfer among nanoparticles should be considered carefully. As the l/d ratio is pretty large within the whole experimental range, i.e., the particles cannot feel their neighboring particles by heat conduction only, the radiation among particles would be a potential mechanism for particle-particle interaction. Such interactions may become strengthened as the particle concentration exceeding a certain value, which may result in higher nanoparticle temperature, and increase the heat loss to the environment by particle radiation. Clearly further fundamental study is required to examine the particle-particle interactions and to understand better the mechanisms.

4. DISCUSSION

4.1 Materials influence

The results clearly show that gold nanoparticles, even under very low particle concentrations, can increase significantly the photothermal conversion efficiency. As we only conducted gold nanoparticle in this work, it would be interesting to compare the results with other published studies

to examine the material's effect. However as briefly reviewed in the Introduction, a lot of the studies were based on the optical properties and characterized by the extinction coefficient only [2,3,4,8,9], which makes impossible to compare the photothermal conversion efficiency. Figure 8 shows the comparison of the enhancement ratio in the photothermal conversion efficiency of GNP with spherical graphite particles (~30 nm diameter) and carbon nanotubes (6–20 nm diameter, 1000–5000 nm in length) [2]. It clearly shows that GNP possesses much higher photothermal conversion capability than the other two materials. The required particle concentration is 2~4 order of magnitude lower than those of carbon based materials to reach a similar enhancement ratio. Such a result is plausible as the absorption peak of most carbon materials lay in the infrared regime, overlapping with that of water. As shown in Figure 4, gold nanoparticle interacts strongly with the solar energy in the visible light spectrum. Significant heat can be produced through the surface Plasmon resonance process (SPR), where the absorption from the nanoparticle could be greatly enhanced via the coupling of the incident radiation with the collective motion of electrons in metal. This is complementary to the base fluid, water, which exhibits strong absorption capability in the infrared spectrum. It becomes clear that the energy conversion efficiency will be heavily dependent on the materials used. Plasmonic particles become very effective in converting photo-energy into thermal energy especially in the visible light spectrum. Comparing to other materials, Figure 8 also implies that the cost of expensive gold nanomaterial may be offset by its much lower amount required to achieve a similar efficiency enhancement. As the cost of nanoparticle is difficult to quantify, which is heavily dependent on the scale and production process, a detailed economic analysis is yet to be conducted.

4.2 Photothermal conversion efficiency vs extinction efficiency

The extinction coefficient is a key factor to show the wavelength-selective absorbing feature and absorption ability of nanoparticles on solar energy; however these data may not represent the real photo-thermal energy conversion process. For instance, certain materials (such as many rare earth materials) can absorb certain light spectrum, and may emit to another spectrum but not converting into heat. Some amorphous materials may have deep dark color that renders to a high extinction coefficient, which however may not be efficient photothermal energy convertors. Figure 9 shows a comparison of these two efficiencies. The green curve is the efficiency based on the integration of the absorption spectrum as shown in Figure 4 with the spectrum from the solar simulator.

Significant difference is found at the low particle loadings. It shall therefore be cautious to use the extinction efficiency to represent the photothermal conversion efficiency.

5. CONCLUSION

This work investigates experimentally the photothermal conversion characteristics of gold nanoparticle-based dispersions under a solar simulator. In a short summary,

- Controlled gold nanoparticle dispersions are formulated through the citrate reduction method with the aid of ultrasonification
- Gold nanoparticles are found to have remarkable performance in improving the photothermal conversion efficiency of the base fluid at very low particle loadings, i.e. 20% enhancement at a GNP concentration of 0.15 ppm.
- The photothermal conversion efficiency increases with the particle concentration but in a non-linear fashion. The highest enhancement is found to be 65% at a particle concentration of 6 ppm within the experimental range.
- The comparison with other nanomaterials shows that GNP has the highest specific absorption rate, which is believed to be associated with its surface plasmon resonance characteristics in the visible light spectrum.
- Though all GNP concentrations fall in the independent scattering regime, it is still unclear why the SAR of GNP decreases as the particle concentration increases.
- It shall be cautious to use the extinction efficiency to represent the photothermal conversion efficiency.

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Nomenclature

A: area (m^2)

c: specific heat (J/kgK)

d: particle diameter (m)

I: solar intensity (W/m^2)

L: distance of light travelled (m)

l: particle-particle distance (m)

m: mass (kg)

P: Radiation transmitted through a sample (W/m^2)

P0: Incident radiation (W/m^2)

SAR: specific absorption rate (W/g)

T: Temperature (K)

t: time (s)

Greek

ε : extinction coefficient

Ø : volume fraction (%)

η: photothermal conversion efficiency (%)

Δ: difference

Subscript:

w: water

n: nanoparticle

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ALL FIGURES

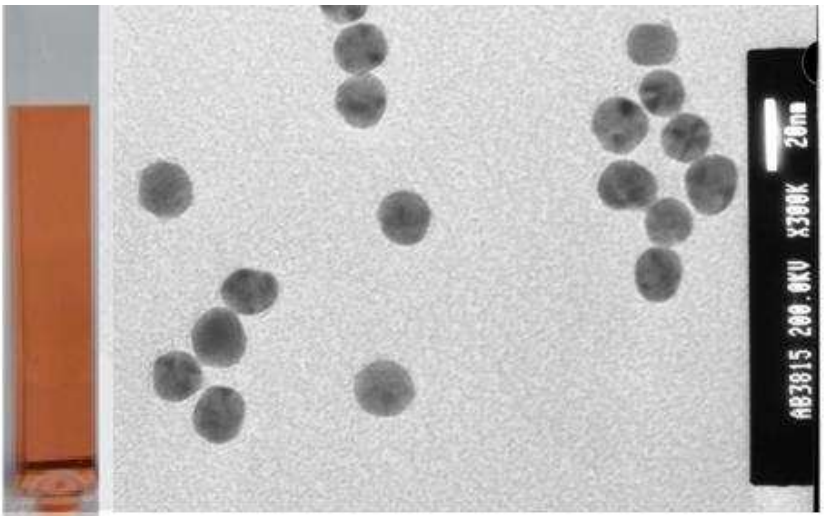


Figure 1 TEM image of GNPs, inset: resulting dispersion of red-wine color

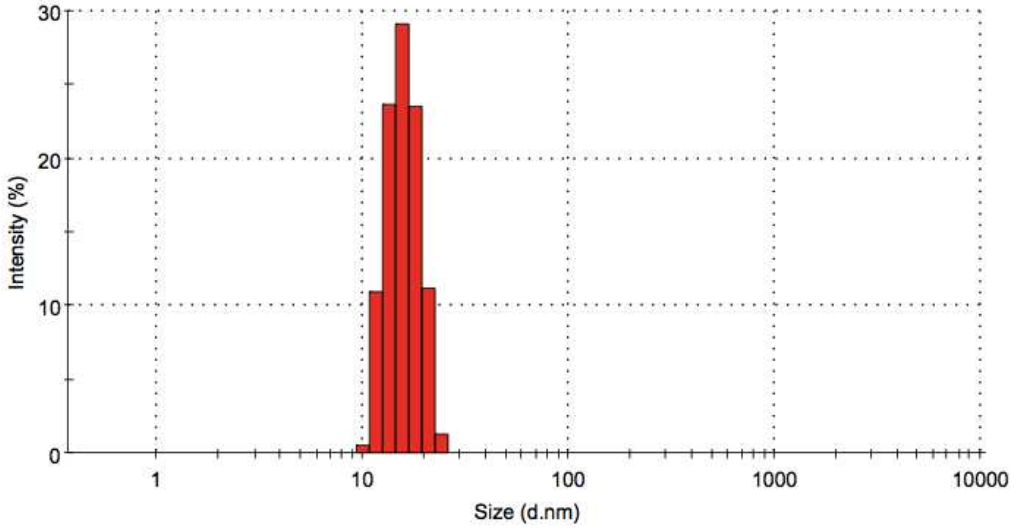


Figure 2 Particle size distributions in an aqueous medium measured by dynamic light scattering (DLS) technique

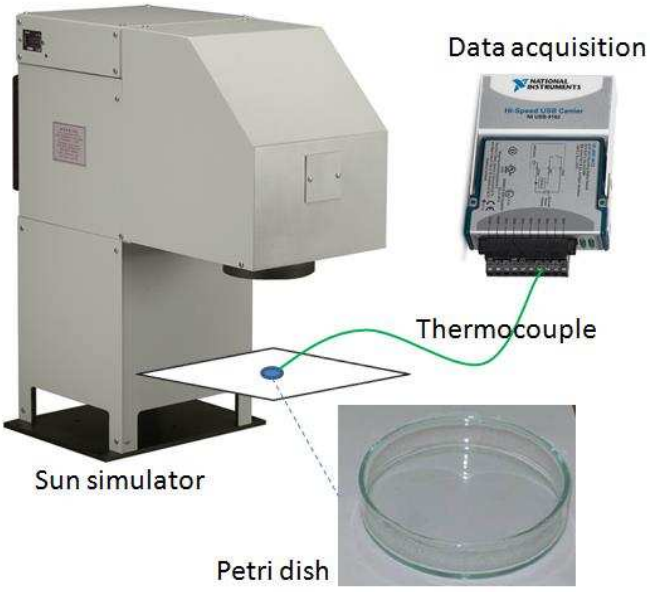


Figure 3 Schematic illustration of the photothermal conversion experimental system

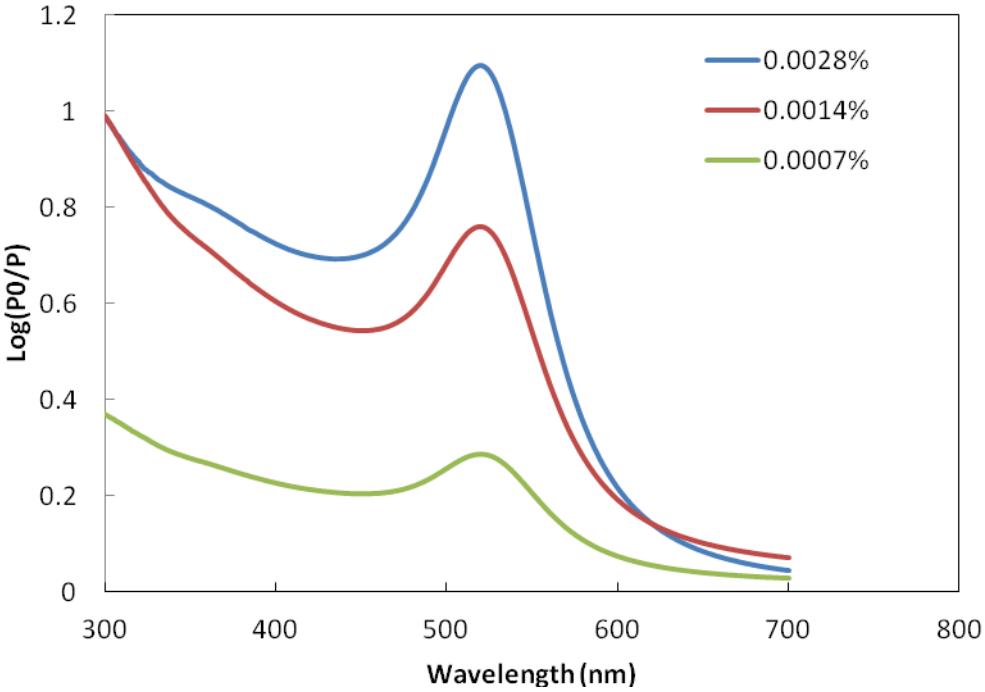


Figure 4 The absorbance spectrum of gold nanoparticle dispersions at different mass concentrations

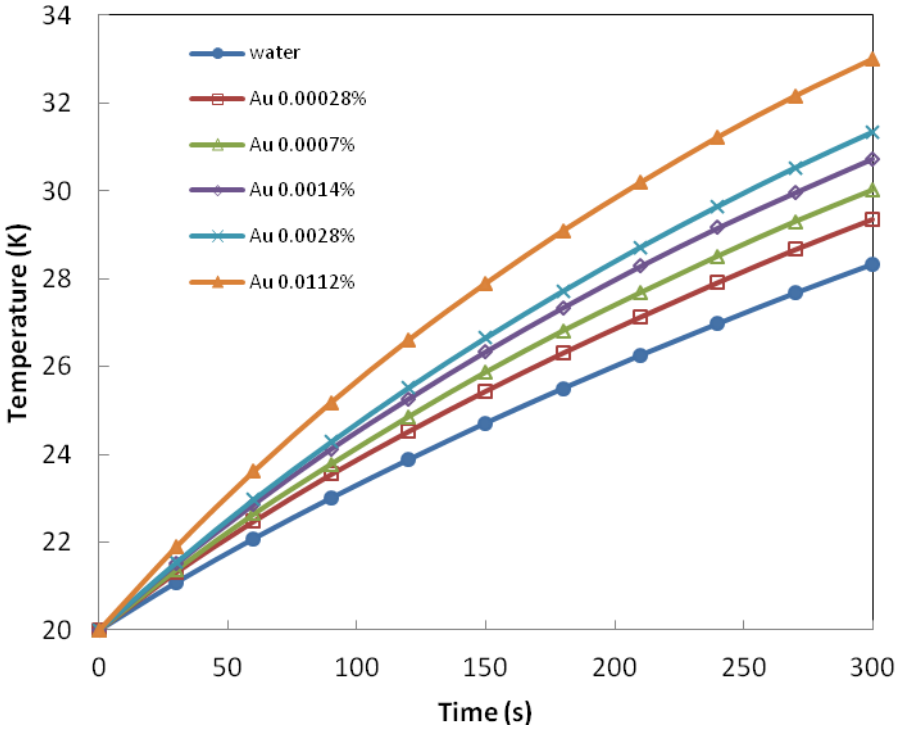
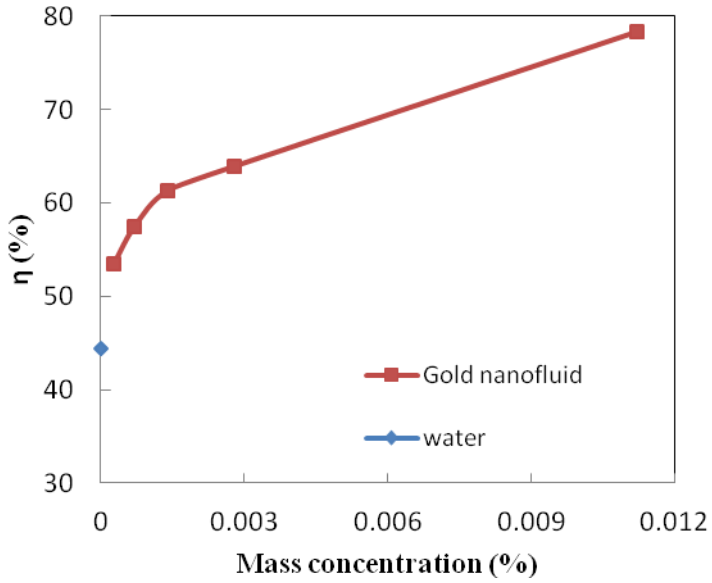
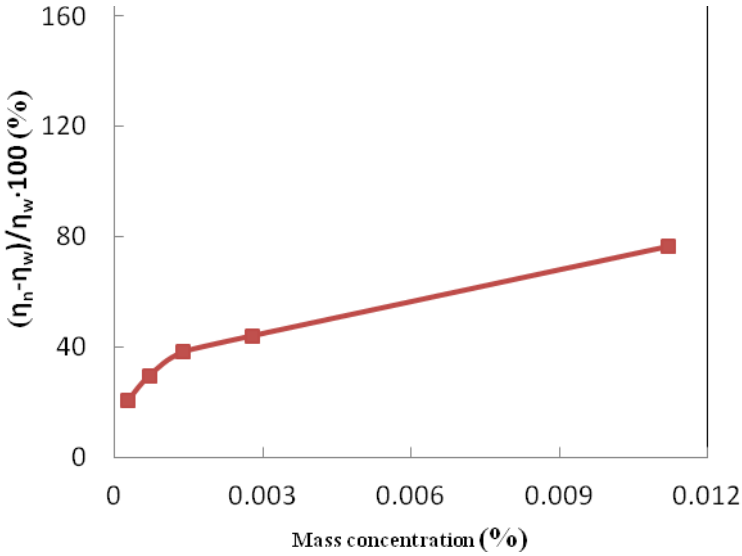


Figure 5 Bulk transient temperature profiles of gold nanoparticle dispersions heated under 1.5 AM



(a) Photothermal conversion efficiency of gold nanoparticle dispersions



(b) Enhancement ratio of the photothermal conversion efficiency comparing to the base fluid

Figure 6 Photothermal conversion efficiency of gold nanoparticle dispersions and the base fluid (based on the first 60 seconds data)

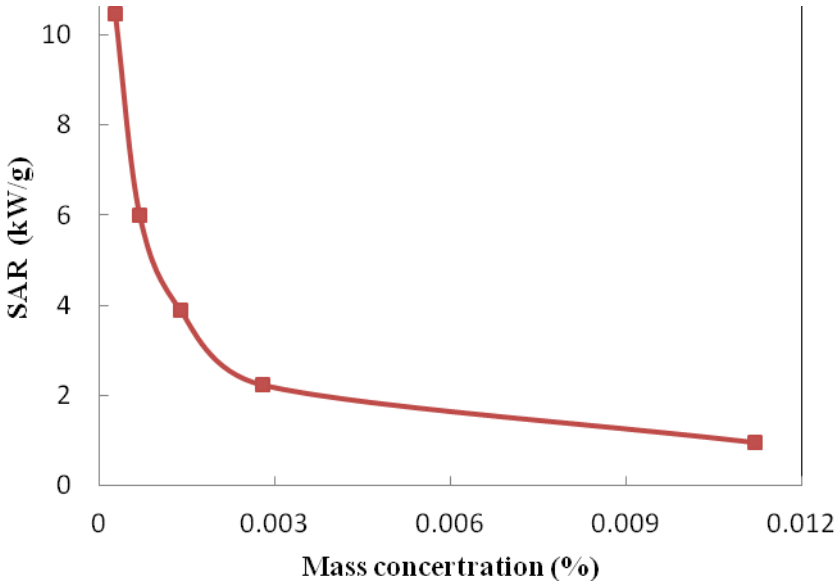


Figure 7 The specific absorption rate of gold nanoparticle at AM=1.5

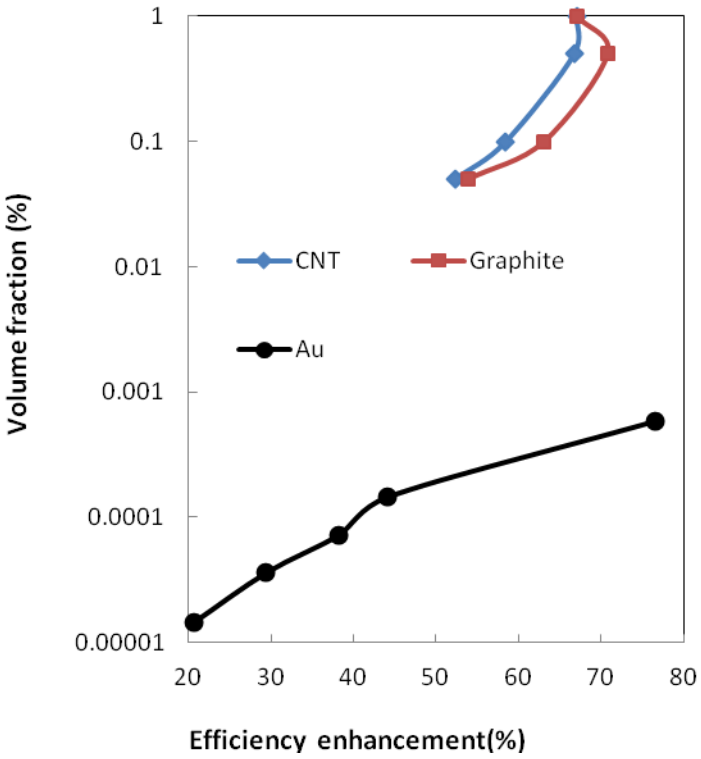


Figure 8 Comparison of the enhancement ratio of the photothermal conversion efficiency for different nanomaterials (CNT and Graphite data from Otanicar et al. [2])

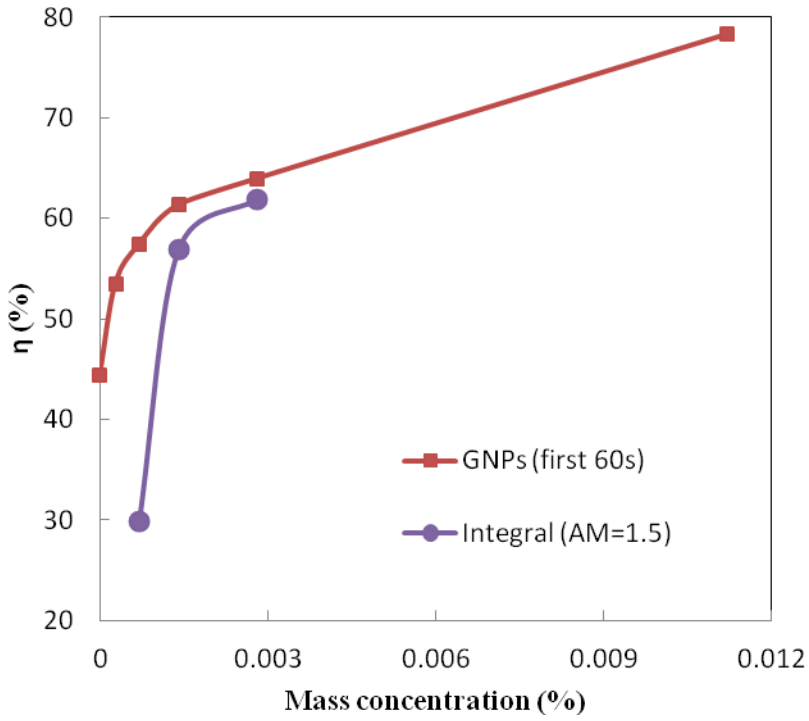


Figure 9 Comparison between the photothermal conversion efficiency and the extinction efficiency