Continuous flow-based laser-assisted plasmonic heating: A new approach for photothermal energy conversion and utilization

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HIGHLIGHTS

- Plasmonic heating effect of Au nanoparticles under continuous flow conditions.
- Optical and photothermal properties of nano fluids investigated.
- Au nano fluid showed higher absorption and photothermal performance.
- Flow rate and nanoparticles concentration affected the photothermal performance.

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ABSTRACT

The aim of this study is to enhance our understanding on photothermal performance of plasmonic Au/TiO2 nanoparticles by conducting a well-controlled experiment under continuous flow conditions. Herein, plasmonic heating experiments of Au nanoparticles decorated on TiO2 nanoparticles were performed using 532 nm laser irradiation. Different parameters, such as Au loading, concentration of nanoparticles, flow rate and laser intensity that could affect the optical and photothermal properties of the nano fluids were studied. The results revealed that the photothermal performance of the nano fluids was remarkably increased in the presence of Au. Particularly, Au nano fluid exhibits a significant higher temperature achieving up to 32 °C compared to that of TiO2 nano fluid (22.5 °C) and water-based fluid (20.5 °C) which is attributed to the localized surface plasmon resonance effect on the surface of Au nanoparticles. The concept of continuous-flow based plasmonic heating of Au/TiO2 nano fluid, with considerable optical and thermal properties, is a promising approach in efficient photothermal applications such as thermal energy supply in industrial chemical processes.

1. Introduction

The ever-growing global energy demand and environmental issues are the major challenges that attracted great attention over the last decades. In this direction, an increasing research interest in the field of light harvesting energy systems, has led to the development of energy and environmental friendly technologies through photovoltaics, photochemical and photothermal processes [1,2]. Among these technologies, photothermal energy conversion is considered as an effective and simple process, in which incident photons are absorbed, and then converted into heat energy. This energy is then transferred through the working fluid. Various potential working fluids have been used to fulfill different energy demands including industrial and residential process heat [3], electricity and heat generation [4], cooling and refrigeration [5] and production of methanol [6] and hydrogen [7].

The selection of the appropriate working fluid with excellent performance in photothermal processes is very important. Nanofluids, which are fluids containing dispersed solid nanoparticles in liquids with unique thermal properties, have shown great potential to improve the photothermal performance due to their superior absorption and scattering [8-10]. The concept of nanofluids was first reported by Tyagi and co-workers [11]. Since then, many studies investigated the feasibility of nanofluids to optimize the photothermal conversion efficiency using metal oxides [12], carbon materials [13] and metal nanostructures such as Al [14], Ag [15] and Au [16]. For example, Said et al. evaluated the performance of a solar collector through the optical...
properties of TiO2 nanofluid [17]. Promising results were observed for NP volume fraction less than 0.1%. Otanicar et al. reported a 5% increase of the photothermal efficiency of solar thermal collectors using carbon materials and silver nanoparticles [18]. Mehrali et al. proved that plasmonic graphene nanofluid increased significantly the volumetric solar thermal conversion [19]. Xuan et al. reported that Ag/TiO2 NPs exhibited good optical and photothermal properties and could be a promising nanofluid for solar thermal receivers [20]. Zhao et al. demonstrated the concept of energy-efficient buildings through the development of Fe₃O₄ nanoparticles for window applications [21].

Utilization of plasmonic metal nanoparticles (NPs) has been reported in many photothermal processes ranging from biomedical applications [22], such as disease diagnosis and therapy, to energy harvesting and conversion [23,24], such as solar vapor generation. Research has showed that metal NPs, such as Au, Ag and Cu nanofluids with very low NPs concentration exhibit considerable photothermal performance due to their unique characteristics such as absorption of photons and scattering [22]. Metal nanoparticles convert the absorbed photons to heat energy, which is then transferred to surrounding medium, increasing the temperature. Nanostructured plasmonic metals can excite the localized surface plasmon resonance (LSPR) effect on their surface and enhance the local electromagnetic field [25]. This effect can be specified as the collective resonant oscillation of free conduction electrons confined within metallic NPs that are stimulated by incident light [26,27]. The LSPR properties of plasmonic nanoparticles can be adjusted by altering different factors, including metallic material, size, shape and the surrounding medium [26]. Additionally, inter-particle distance, which depends on distribution of NPs, plays a significant role in the photothermal behavior of plasmonic NPs due to the presence of two different effects: thermal superposition effect and plasmonic coupling effect. The improved thermal accumulation caused by the heat which is transferred from nearby hot NPs can be described as the thermal superposition effect while the effect of plasmonic coupling can be considered as the thermal accumulation related to the plasmonic coupling of nearby NPs, which can enhance the local electromagnetic field and production of heat. In case of low NPs concentration, where the average inter-particle distance is larger, the two effects can be considered as negligible [28–30].

Many studies reported that due to the LSPR effect on the surface of plasmonic nanoparticles, a significant improvement of optical absorption and photothermal performance of plasmonic nanoparticles can be achieved. For instance, Chen and co-workers performed several experimental studies on the optical properties of metal NPs and the photothermal conversion efficiency of thermal collectors [8,31,32]. The results indicated that nanofluids containing Au and Ag obtained the highest photothermal conversion efficiency. Amjad et al. showed that the solar steam production increased by 95% in the presence of Au nanoparticles [23]. Zeng et al. reported a significant efficiency of the thermal collector up to 97.6% using CNT-TiO2/Ag [33]. Qin and co-workers investigated the heat generation of gold nanoparticles for photothermal therapy under laser irradiation [34]. Zhang et al. showed that Au NPs possessed higher absorption compared to base fluids resulting in an enhancement of the thermal collector efficiency by 80% [35]. Jin and co-workers performed an experimental and theoretical study on the photothermal properties of Au nanofluids for steam generation [36]. The steam production was attributed to highly localized boiling and evaporation which caused by non-uniform temperature distribution.

Research has indicated that Au NPs have higher chemical stability to oxidation compared to Ag and Cu and relatively high absorption and scattering properties, which enable them more attractive metals for photothermal applications [32,34,37]. Although plasmonic metal NPs have shown a good activity in photochemical processes they are not considered as ideal materials for large-scale applications due to aggregation, low stability and high-cost. For this reason, metal oxide materials are used as supports to further enhance the functionality and stability of metal NPs by providing a large surface area to enable a uniform dispersion of metal [37–38]. TiO2 is one of the most promising semiconductors that favours from its chemical and thermal stability and has been employed as an efficient support for particularly low Au loadings under visible light to promote effectively several plasmonic photocatalytic and photoelectrocatalytic processes [39–40].

So far, a plethora of studies as described above has investigated the feasibility of using free-standing metals in photothermal processes. Although there have been many approaches in photothermal heat generation from plasmonic nanoparticles, these studies mainly investigated the optical properties of the nanoparticles with limited photothermal experiments and evaluation of the parameters that affect significantly the efficiency of photothermal conversion.

Additionally, the existing technology of batch-type systems that has been mainly used in photothermal processes has limitations in large-scale applications due to long time-scale of operation hours [8,32]. Development of a continuous-flow system is a promising approach to deal with the required needs of designing a system that comprises advanced light harvesting and reduction of photon loss, characteristics that can ensure a photothermal process with high conversion efficiency [41–43].

The aim of this study is to systematically investigate the parameters that affect the photothermal efficiency of plasmonic nanofluids under well-controlled experimental conditions. Herein, plasmonic heating experiments of Au NPs decorated on TiO2 were performed under continuous flow conditions using visible laser irradiation. The developed flow system benefits from improved heat and mass transfer, enhanced light utilization and allows control of operating conditions over fluids. The effect of Au loading, Au/TiO2 NP concentration and laser intensity versus flow rate on the photothermal properties of nanofluids was explored by measuring the temperature change. Furthermore, the optical and physical properties of Au/TiO2 NPs with various Au loadings were characterized. Finally, the photothermal performance of Au/TiO2 nanofluids under laser irradiation was analysed to evaluate the potential utilization of the nanofluid in thermal supply for industrial chemical processes.

2. Experimental

2.1. Materials

Gold (III) chloride trihydrate (HAuCl₄·3H₂O ≥99.9% trace metals basis) and P25 Titanium (IV) oxide (particle size 21 nm, ≥99.5% trace metals basis) nanopowders were obtained from Sigma-Aldrich. Sodium hydroxide (Pellets, ≥98.5%) and ethanol absolute (≥99.8%) were supplied from Acros Organics and Fischer Scientific respectively. All solutions were produced using milli-Q water with 18.2 MΩcm resistivity.

2.2. Synthesis of Au/TiO2 nanoparticles

The synthesis of Au decorated TiO2 nanoparticles was done by deposition-precipitation (DP) method with NaOH and AuCl₃·3H₂O as precursor (Fig. 1) [40,44]. Commercial Degussa P25 TiO2 was dried in an oven for 24 h before use. Different wt.% of Au NPs (1, 3 and 5 wt.%), were prepared by stirring 25 mL aqueous solution containing different concentrations of AuCl₃·3H₂O. The pH of the different solutions was controlled to 9 by adding certain amount of 0.1 M NaOH. Then, 150 mg P25 TiO₂ were added to the solution while the pH was re-adjusted to 9. The DP was performed at 80 °C under stirring for 2 h keeping the pH stable and then the solution was under stirring overnight. The prepared samples were washed with ethanol and milli-Q water by centrifuge. Then, the samples were air-dried at 80 °C overnight. Subsequently, the obtained powders were annealed at 300 °C for 4 h with a ramping rate of 5 °C/min. The Au/TiO₂ nanopowders were cooled and kept in the dark inside a desiccator over silica gel to prevent photodecomposition.
and moisture. The final Au/TiO₂ samples obtained had purple color.

2.3. Characterization of Au/TiO₂ nanoparticles

Crystal structures of the synthesized nanopowders were identified using a D8 Advance (Bruker AXS) X-Ray diffractometer (XRD) with Cu Ka radiation (\(\lambda = 1.5418 \text{ Å}\)) and a nickel beta filter (20 = 10–80°). Raman spectra were collected with a Renishaw microscope using 785 nm laser excitation. Transmission Electron Microscope (JEOL TEM-1400 Plus) was utilized to characterize the morphology of Au/TiO₂ nanoparticles. Further investigation on the morphology, gold loading and particle size was performed with a high-resolution transmission electron microscope (HR-TEM FEI Titan Themis 200) equipped with an energy dispersive X-Ray (EDX) detector operated at 200 kV. Diffuse reflectance spectra were obtained using a UV–Vis spectrometer (Perkin Elmer Lambda 950) equipped with a 150 mm integrating sphere using barium sulfate as reference for background correction.

2.4. Fabrication of continuous flow PMMA container

A Trotec Speedy 300 laser cutter/engraver was used to fabricate a continuous flow container made of polymethyl methacrylate (PMMA) sheet. The fabricated container consisted of an inlet and outlet to allow the continuous flow of the nanofluids. The different nanofluids were supplied to the PMMA container utilising a syringe pump (Genie Touch, Kent Scientific Corporation) with flow rate ranging from 0.05 to 0.4 mL·min⁻¹. The total volume of the cell was 2 mL (2 cm × 1 cm × 1 cm).

2.5. Experimental set-up

A schematic illustration of the experimental set-up is presented in Fig. 2. The continuous flow container was irradiated from the top using 532 nm laser light while the top was covered with quartz glass. During the experimental procedure, the fabricated container was held in the air using a clamp to prevent heating and was covered by insulated material to reduce heat loss. Two thermocouples were installed to the data acquisition unit plugged in a computer and were placed after the inlet (before illumination) and before the outlet (after laser illumination) of the container to monitor the temperature (Fig. S1). Temperature measurements were recorded with PicoLog thermocouple data logger (TC-08) until a thermal steady state has been reached. The starting temperature of all fluids was 20.3 °C and the error bar was estimated at ± 0.1 °C.

3. Results and discussion

3.1. Characterisation

The structural properties of the plasmonic NPs were identified by X-Ray diffraction as shown in Fig. 3a. The presence of anatase and rutile phase of TiO₂-P25 was confirmed from the XRD pattern. The diffraction peaks detected at 2θ = 25°, 38° and 48° were indexed to lattice planes (1 0 1), (0 0 4) and (2 0 0) of anatase phase while the diffraction peaks at 2θ = 27°, 41°, 54° and 63° were assigned to (1 1 0), (1 1 1), (2 2 0) and (0 0 2) lattice planes of rutile phase of TiO₂ respectively. No characteristic peaks associated to Au were observed in the Au loaded TiO₂ samples. This can probably be attributed to the small particle size of Au NPs in accordance with TEM analysis (NPs, \(\leq 5 \text{ nm}\)) and low Au concentration. Additionally, overlap of expected Au (111, 200) reflection peaks with TiO₂ anatase (0 0 4) and TiO₂ rutile (1 1 1) makes it difficult to identify Au particles.

Further investigation of the crystal structure of Au/TiO₂
nanopowders was performed using Raman spectroscopy. Raman spectra of the different samples are shown in Fig. 3b. The characteristic bands of anatase phase positioned at 142, 395, 515 and 638 cm$^{-1}$, which correspond to $E_g(1)$, $B_1g(1)$, $A_1g + B_1g(2)$ and $E_g(2)$ vibrational modes respectively, are present in all tested samples. After Au loading, no characteristic peak corresponding to Au nanoparticles was detected. However, for Au/TiO$_2$ samples, by increasing Au loading, the intensity of the main anatase peak at 142 cm$^{-1}$ was slightly decreased as well as shifted to 145 and 147 cm$^{-1}$ for 3 and 5 wt.% Au content respectively. This indicates that the Au loading created lattice distortions, known as crystalline defects within TiO$_2$, which increased by increasing Au content. As a result, the characteristic vibrational frequency of TiO$_2$ was affected [45,46]. A further increase of Au content to 5 wt.%, led to a decrease of the intensity of 395, 515 and 638 cm$^{-1}$ peaks of TiO$_2$ which revealed the increased crystalline defects. This observation is in agreement with the large nanoparticles identified in TEM images.

Additionally, an enhanced intensity of LSPR band at 532 nm was observed by increasing Au loading from 1 to 5 wt.%, which was also indicated by the color change of Au/TiO$_2$ powders from light to dark purple. This happens because the surface plasmon resonance wavelength is dependent on the metal content and particle size of noble metal [48]. The plasmon peak of Au NPs is consistent with laser irradiation used in our experiments.

The energy bandgap of the synthesized samples was estimated (Fig. 4b) utilising the following Kubelka-Munk function [49]:

$$F(R) = \frac{(1 - R)^2}{2R}$$

The bandgap for P25-TiO$_2$ was estimated at 3.2 eV, which is in accordance with the literature. In case of Au loaded TiO$_2$ nanoparticles, an increase in the Au content from 1 to 5 wt.% led to a decrease of the band gap. Specifically, the band gap values were found at 3.07, 2.88 and 2.96 eV for 1, 3 and 5 wt.% Au respectively. The decrease of the bandgap stems from the inter-band transition of electrons of Au NPs from the d band to the s band, which shifts the Fermi level closer to the conduction bandgap of TiO$_2$ [50,51].

The morphology of the samples with different Au loading (1, 3 and 5 wt.%) was studied by HR-TEM. It should be mentioned that no
significant alternation was observed in the morphology of TiO2 sample when Au NPs with different concentration were loaded. TEM images (Fig. 5a-c) showed that the surface of TiO2 was uniformly decorated by a significant amount of small Au NPs. The concentration of Au increased by increasing the Au content to 3 and 5 wt.%. However, the average size of Au NPs enlarged with higher distribution upon further increasing the gold content and a few Au NPs tended to agglomerate in case of 5 wt.% (Fig. 5c). Specifically, nanoparticle size was calculated at 3.3, 3.6 and 8.1 nm for 1, 3 and 5 wt.% respectively.

The crystalline structure of Au and TiO2 NPs was confirmed from the ring pattern as it can be seen from the selected area electron diffraction (SAED) pattern (Fig S2). The lattice spacing of 0.325 nm and 0.169 nm correspond to (1 1 0) and (2 1 1) crystal plane of rutile phase of TiO2 while 0.235 nm correspond to (1 1 1) lattice fringe of Au.

The presence of homogeneously distributed Au, Ti and O elements was confirmed using EDX elemental mapping (Fig. 6). Au NPs deposited on the surface of TiO2 were clearly detected in the elemental mapping. EDX analysis showed that the amount of Au attached on TiO2 surface during the synthesis process was lower than the nominal one. Approximately 40% reduction of the actual gold was observed, which is in agreement with the literature [39,40]. More specifically, the nominal gold content was found to be 0.56, 0.84 and 1.89% for 1, 3 and 5 wt.% respectively. This trend is highly related to the fact that the number of TiO2 adsorption sites is reduced due to high pH values of synthesis solution leading to limited Au deposition [40]. It should be also noted, that the deposition efficiency of this method decreases by increasing the amount of the gold in the synthesis solution.

![Fig. 5. TEM images for (a) 1 wt.% Au/TiO2, (b) 3 wt.% Au/TiO2 and (c) 5 wt.% Au/TiO2.](image)

![Fig. 6. TEM-EDX mapping for 3 wt.% Au/TiO2 sample.](image)
3.2. Laser induced plasmonic heating

An experimental exploration of the effect of localized plasmonic heat produced by Au NPs was performed using 532 nm laser irradiation. Fig. 7a shows the recorded temperature profile of the different nanofluids (0.6 mg mL\(^{-1}\)) under 532 nm laser excitation using a flow rate of 0.1 mL min\(^{-1}\). The inlet temperature for the different nanofluids including pure water was 20.4 ± 0.1 °C. All nanofluids presented higher temperature under laser irradiation compared to pure milli-Q water, in which temperature raised just ~0.5 °C. A remarkable increase in temperature of Au nanofluid under laser irradiation compared to pure milli-Q water, in which temperature raised just ~0.5 °C. A remarkable increase in temperature of Au nanofluid under laser irradiation can be seen in Fig. 7a. It should be mentioned that the temperature of all Au/TiO\(_2\) nanofluids was significantly higher compared to pure TiO\(_2\) nanofluid (22.3 °C), indicating a higher optical absorption of the nanoparticles. As expected, the different Au loading affected significantly the temperature profile. More specifically, the temperature reached up to 28.6 °C when 3 wt.% Au/TiO\(_2\) nanofluid was used instead of 1 wt.% Au/TiO\(_2\). However, a further increase of Au loading to 5 wt.% resulted in lower temperature compared to 3 wt.%. Generally, by changing the Au loading, parameters like Au particle size, morphology of the sample as well as the metal dispersion can be altered. The temperature decrease for higher Au loading is related to the increase of the mean size of Au NPs and to the formation of Au agglomerates, as can be seen from TEM images. In case of excess Au loading, particles tend to agglomerate, resulting in limited conversion of the absorbed energy to thermal energy and thus the temperature increase is reduced. This phenomenon is attributed to the plasmonic coupling effect caused by the particle interaction [30]. Additionally, nanofluids with small metal NPs have higher thermal conductivity compared to nanofluids consisting of larger NPs, which results to faster temperature increase of the nanofluid containing smaller NPs [52]. As a result, the temperature of the nanofluid containing smaller NPs increases faster in comparison with that of larger NPs.

The performance of different nanofluids at flow rates ranging from 0.05 to 0.4 mL min\(^{-1}\) was investigated (Fig. 7b). For pure milli-Q water, no significant change of temperature was observed with different flow rates. In the case of nanofluids, the same trend was observed. By increasing the flow rate from 0.05 to 0.1 mL min\(^{-1}\) a higher temperature was achieved while a further increase of the flow rate up to 0.4 mL min\(^{-1}\) led to the lowering of the temperature. At large residence time, the lower temperature observed is attributed to mass transfer limitations of the nanofluid due to low flow rate (< 0.1 mL min\(^{-1}\)). Particularly, blocking effect was observed from the dispersed NPs in the fluid in flow rates lower than 0.1 mL min\(^{-1}\). Under fast flow rates, mass transport is improved but the nanofluid doesn’t remain under illumination for sufficient time to obtain the maximum temperature increase. All the different nanofluids, achieved the maximum temperature when an optimal flow rate was set at 0.1 mL min\(^{-1}\). The highest recorded temperature for 3 wt.% Au loading using 0.1 mL min\(^{-1}\) flow rate was 28.6 °C, while 1.8 °C decrease was noticed when the flow rate was set at 0.4 mL min\(^{-1}\).

The effect of concentration on temperature performance using 3 wt.% Au/TiO\(_2\) was further evaluated (Fig. 8a). Four different concentrations of 3 wt.% Au/TiO\(_2\), varied from 0.2 to 1.4 mg mL\(^{-1}\), were prepared and tested under the same conditions as described above using flow rates between 0.05 and 0.4 mL min\(^{-1}\). Fig. 8a shows a significant temperature rise by increasing Au NPs concentration up to 1 mg mL\(^{-1}\). Among the tested nanofluids, the sample with 1 mg mL\(^{-1}\) concentration exhibited the highest temperature up to 31.5 °C with flow rate 0.1 mL min\(^{-1}\). According to Beer-Lambert law, when the concentration of Au/TiO\(_2\) NPs increased, the amount of suspended nanoparticles in milli-Q water was higher, so more light was confined in the nanofluid, resulting in higher temperature [8]. However, upon further increasing the concentration to 1.4 mg mL\(^{-1}\), the temperature significantly decreased to 26.3 °C. This phenomenon can be ascribed to a possible saturation of the absorption of light caused by a further increase of the concentration [20]. Fig. 8b shows the temperature of the different nanofluids for (a) 0.6 mg mL\(^{-1}\) concentration and flow rates 0.05, 0.1, 0.2, 0.3 and 0.4 mL min\(^{-1}\).
Au/TiO₂ and the flow rate were set to 1 mg mL⁻¹ and 0.1 mL min⁻¹ and respectively. As it can be seen, by increasing the laser power intensity, the Au nanofluid’s temperature presented a remarkable raise. Specifically, a temperature increases of 11.5 °C caused by Au nanoparticles at power density of 500 mW is the highest value obtained. The main reason for this phenomenon is that higher laser power density leads to higher light density and thus the heat generated by Au nanoparticles will subsequently increase. In this way, by increasing the laser intensity, a temperature rise of the heated medium can be achieved [34].

Finally, the stability of the samples was evaluated before and after the photothermal experiments using UV–Vis spectroscopy. UV–Vis absorbance spectra showed excellent stability of Au/TiO₂ nanofluids with a slight decrease of the absorption of 3 wt.% Au/TiO₂ sample as it can be seen in Fig. S4 in supplementary information. Additionally, the TEM images unveiled no obvious change in the morphology and the size of Au NPs on TiO₂.

4. Conclusions

In summary, Au/TiO₂ nanoparticles were synthesized utilising deposition-precipitation method and plasmonic heating effect from the Au nanoparticles under continuous flow conditions using 532 nm laser irradiation was studied extensively. The optical properties of Au/TiO₂ nanoparticles were investigated, indicating that plasmonic nanoparticles exhibited a remarkable absorption in the region 500–550 nm, attributed to the LSPR effect excited on Au NPs’ surface. All the different nanofluids containing Au presented higher temperature (up to 28.5 °C) compared to pure water fluid (20.5 °C) proving the importance of nanofluids. The fabrication of a low cost continuous flow system offered the ability to investigate the effect of residence time of the fluid under laser irradiation. The optimal flow rate was 0.1 mL min⁻¹ for all nanofluids, and the generation of plasmonic heating for 3 wt.% Au loading sample was further investigated by studying the effect of nanoparticle concentration and laser intensity. The results showed that temperature increased to 31.5 °C upon increasing laser intensity to 500 mW with an optimal 1 mg mL⁻¹ concentration after which decreased significantly. The presence of plasmonic nanoparticles and particularly NPs size and concentration affect remarkably the efficiency of photothermal processes. In summary, this study offered an enhanced understanding of the fundamental aspects of plasmon-based photothermal performance under well-controlled continuous flow conditions.

The concept of continuous-flow based plasmonic heating of Au/TiO₂ nanofluid, with considerable optical and thermal properties, could be a promising approach in efficient light harvesting applications such as thermal energy supply in industrial chemical processes. For practical considerations, further improvements from the engineering point of view required regarding the geometry of the continuous flow system, minimizing potential heat loss, while enhancement of absorption coefficient of plasmonic nanofluids is of crucial importance as well.

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Appendix A. Supplementary material

Supplementary data to this article can be found online at https://doi.org/10.1016/j.apenergy.2019.04.069.

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