

Fabrication of high stable gold nanofluid by pulsed laser ablation in liquids

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ABSTRACT

Laser ablation in liquids by femtosecond radiation has been used to generate gold nanoparticles in a heat transfer fluid to produce a high stable thermal nanofluid as a heat transfer intensification technique. In oil based fluids, no matter the actual fabrication route, nanoparticles tend to agglomerate. Here, we report a new form to control its stability through the addition of a surfactant that does not degrade at high temperatures. It allow us to produce gold nanoparticles of 58 ± 31 nm in the liquid in situ, avoiding in this way the generation of pollution and reducing the maximum point of nanoparticle agglomeration at 370 nm. The developing of this new nanofluid represents a great opportunity for the harvesting of solar energy industry. Copyright © 2015 VBRI Press.

Keywords: Gold nanoparticles; nanofluids; harvesting of energy; pulsed laser ablation.

Introduction

Gold nanoparticles (NPs) have gained a lot of attention in the past decade because of the possibility to exploit the enhancement of their physical and chemical properties [1-4]. In particular their interaction with light leads to a wide range of applications from biomedicine to energy harvesting [5-10]. In this context, the global economy demands the optimization of current green energy technologies, and the search of clean and sustainable working processes. Therefore, a lot of efforts have been performed to produce materials that capture and transport the solar energy efficiently, such as in the transformation of solar radiation into thermal energy used in concentrated solar power (CSP) plants [11, 12].

Nowadays, a heat transfer fluid commonly used in CSP plants is a thermal oil consisting of an eutectic mixture of 26.5 % diphenyl and 73.5 % diphenyl oxide, able to reach working temperatures up to 400 °C. Nevertheless, these kind of fluids present poor thermal properties and therefore it is necessary to increase its heat transfer capability. The use of nanofluids (dilute suspensions with solid NPs) was proposed to face this problem [13], since the addition of solid particles with higher thermal conductivity than the base fluid results in an improvement of the heat transfer performance of the mixture [14, 15].

One of the most important characteristics of nanofluids is to know if they can remain for very prolonged periods of time without significant settling or loss of stability. The stability of the nanofluid depends on a high level of the production process and its base fluid. To produce nanofluids there are two well-known methods. In the first

one the NPs can be produced in the form of colloidal suspension in separated solutions either chemically [16, 17] or physically [18], and subsequently dispersed into the fluid. In this procedure, agglomeration of NPs takes place in either both stages, leading to poor suspension stability and high shear viscosity, causing an increment of the fluid pumping costs. To avoid these problems, NPs can be produced directly in the base fluid. However, the typical physical and chemical methods used for this aim such as chemical reduction, photochemical reduction, glow discharge and sonochemical reduction, are not so effective avoiding the agglomeration [19-22]. Moreover, some of the methods show pollution problems with the subsequent environmental impact.

An alternatively physical laser-assisted method to produce highly stable nanofluids is pulsed laser ablation in liquids (PLAL). It is based on irradiation with ultra-short pulsed electromagnetic waves to promote extraction of high purity, well dispersed NPs inside a liquid [23-26]. After the ablation, to enhance the characteristics of the NPs, the colloidal solution is irradiated again by the laser promoting a photo-fragmentation of the NPs to control their size [27-31].

Apart from the production process, a chemical stabilization is required to enhance the stability against agglomeration of NPs. Stability can be predicted by knowing the total energy of interaction between two particles. In the range of sizes of nanofluids, the ratio particle surface to particle volume is so high that all the interactions are controlled by short-range forces like Van der Waals attraction force. As a consequence of the attractive nature of the forces presented, when primary

particles are suspended in a fluid they have a tendency to agglomerate when come into contact due to their Brownian motion. This agglomeration can be partially avoided by means of repulsive surface forces created among particles. The classical DLVO (Derjaguin-Landau-Verwey-Overbeek) theory only takes into account electrostatic repulsion, however, in non-aqueous media steric or electro steric repulsion is preferred [32]. The latter is achieved by means of the addition of ionic surfactants that generates a charged medium surrounding the polymeric chains adsorbed to the particle surface, thus preventing contact and agglomeration of particles. The amount of surfactant has to be optimized in order to ensure its best performance. If the concentration is low, nanoparticles may not be fully covered and the dispersion can be still improved. However, if the concentration is higher than an optimal value, the formation of micelles occurs and they can act as a grafting agent between nanoparticles [33]. In our case, the surfactant used was tetra-*n*-octylammonium bromide ((C₈H₁₇)₄NBr, TOAB, > 98 %) because this kind of dispersant shows chemical compatibility with the thermal oil and it can be used for practical applications, as TOAB does not degrade at temperatures over 200 °C. The concentration of additive has already been optimized by other authors in previous works [15]. It is necessary to remark that no matter the stabilization methodology used, it never avoids completely the nanoparticle agglomeration for an unlimited period of time. In addition, nanoparticle stabilization presents a special challenge in the case of the complex base fluids as the one used in this work.

In this paper gold NPs highly dispersed in a thermal oil were produced by PLAL method and stabilized with the help of a cationic surfactant previously introduced in the base fluid. Moreover, we experimentally demonstrated that the nanofluid created has a higher stability than those reported in previous publications, but keeping the improvement of thermal properties.

Experimental

Material synthesis

In this communication we present the fabrication of Au NPs in a heat transfer fluid as the base fluid by PLAL using a protocol of two stages. The first stage is based on a laser ablation process where the ejected particles from the target are captured in a liquid (in our case the base fluid is a mixture of the commercial thermal oil Therminol VP-1 and TOAB). This process was carried out with a Ti: Sapphire laser (Femtopower Compact Pro, Femtolasers), which emits pulses of 30fs intensity full width at half maximum (FWHM) with a central wavelength of 800 nm, maximum energy per pulse of 0.5 mJ, and 1 kHz repetition rate. The energy of the pulses was measured with an analogical power-meter (Spectra Physics, Model 407-A) and controlled by using neutral density filters and also an iris of 6 mm of diameter is placed before the focusing optics.

As a target we have used a gold disc of thickness 1 mm and diameter 6.5 mm (99.99 % purity) which was placed at the bottom of a cuvette filled with the base fluid as is shown in Fig. 1 (a). The thickness of the liquid wall above the target was 7 mm. The laser beam was focused with a lens of 750 mm focal length onto the target surface while

the cuvette attached to a 2-D motion controlled stage was moving perpendicularly to the beam propagation axis, at a constant velocity of 4.5 mm/s. The fluence of the laser beam at the sample plane was about 1 J/cm². The dispersion introduced by the liquid and optical elements was compensated in the post-compression stage by changing the relative position of two fused silica Brewster prisms. To control the duration of the ablation process, an electronically controlled shutter was used.

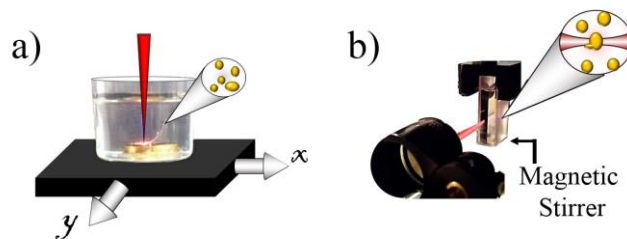


Fig. 1. Experimental setup for the NP fabrication with (a) PLAL and (b) the second photo-fragmentation process.

In the second stage, a 10 ml glass cuvette was filled with the colloidal solution obtained from the first stage and then it was irradiated by the Ti: Sapphire laser beam, which was previously focused to its center with the same lens, as it is depicted in Fig. 1 (b). The suspension was stirred by a magnet to homogenize the photo-ablation process. It consists of a fragmentation of the biggest particles into smaller particles by means of an ablation process of the bigger particles. Under free propagation, the fluence at the focal point of the lens is about 1 J/cm². However, due to the refraction effects this value tends to decrease until 0.6 J/cm² approximately.

Preparation of nanofluids

Three different nanofluids were obtained and characterized in order to study the influence of the production process and the presence of surfactants on the thermo-physical properties of the nanofluids. In the first one (nanofluid 1), NPs were fabricated by PLAL followed by the photo-fragmentation stage in the pure thermal oil. In the second one (nanofluid 2), NPs were fabricated by the same procedure but in a mixture of the thermal oil plus TOAB as base fluid. In the last one (nanofluid 3), commercial NPs were added to the solution of thermal oil plus TOAB. To prepare the mixture in nanofluids 2 and 3, 0.0547g of TOAB was dissolved in 100 ml of the synthetic thermal oil [15].

For preparing the nanofluid 3, to obtain highly dispersed NPs, commercial gold NPs were acquired in aqueous suspension from Sigma Aldrich. NPs are stabilized in 0.1 mM of phosphate buffered saline (PBS) solution and the primary particle diameter is 50 nm. First water content was removed from the aqueous solution by evaporation in a hot plate. Then, the solid product obtained was dispersed in the mixture of the thermal oil plus TOAB with the help of an ultrasound probe. The sonication time varies from 5 to 10 min depending on the sample volume. With this procedure a concentrated nanofluid was obtained. Later, it was diluted to achieve the same optical density and solid content than in nanofluid 1 and 2, where the quantity of the

ablated material is well fixed by the method. Optical density was calculated by measuring the radiation absorption of a laser beam at 635 nm (Model CPS180, Thorlabs) with a photodiode (Model SM05PD1B, Thorlabs) in a rectangular quartz cuvette.

Characterizations

Several NPs characterization techniques as Transmission Electronic Microscopy (TEM), Energy Dispersive X-ray Spectroscopy (EDX), High Resolution Transmission Electronic Microscopy (HRTEM), Selected Area Electronic Diffraction (SAED) and Dynamic Light Scattering (DLS) were used to determine the morphology, purity, NPs size and stability of the fabricated nanofluids. Moreover, the thermal conductivity enhancement was determined by the transient hot wire technique. We observe that the laser procedure can be actually used for fabricating gold NPs in organic oils like Therminol VP-1 mixed with dispersant such as TOAB enhancing the stability of the fluid without concern that it causes any uncontrolled and or unwanted chemical reaction.

The respective micrographs used for the morphology and structural analysis were obtained with a TEM (JEOL 2100 microscope) operating at a voltage of 200 kV for nanofluid 1, 2 and 3, severally. To prepare the samples, just after the fabrication of the fluids a droplet of the colloidal suspension was dispersed onto a carbon-coated copper-based TEM grid. The liquid content was then removed with the help of an absorbent paper so the solid particles can remain in the grid surface statically without the opportunity to agglomerate.

Using an EDX system attached to the TEM (an Oxford Instruments INCA Penta FETX3) EDX spectrums were obtained to determine the pureness of the final material. The mean size and size distribution of the NPs in the fluids were measured by DLS, using a Zetasizer nano ZS (Malvern Instruments Ltd., UK) with a 173° scattering angle. And finally the thermal conductivity of the nanofluids as well as the base fluid (with and without TOAB) was measured at 80 °C by means of the transient hot wire technique, using a KD2 Pro conductivity meter (Decagon Devices Inc.). Six measurements were performed for each sample so the experimental error could be determined at a 95 % of confidence level.

Results and discussion

Morphology and pureness

From the results, it is clear that in contrast with the icosahedral particle shape that corresponds to the commercial NPs, the morphology of the particles synthesized by the laser procedure is mostly spherical as it can be noticed in **Fig. 2 (a-c)**. The difference among the sample shapes can be due to the fact that in the NPs produced by PLAL process, the fragmented material is released from the bulk material and suspended in a liquid ambience which has a certain density. The interaction between the fragment and liquid could lead to the surface tension phenomenon which implies that the atoms located at the particle surface have a higher mean energy than the atoms located inside the particle, therefore the tendency of the system is to diminish the total energy, which is achieved

by decreasing the number of atoms located at the surface, hence the particle acquires the minimum surface shape possible, which is the spherical one.

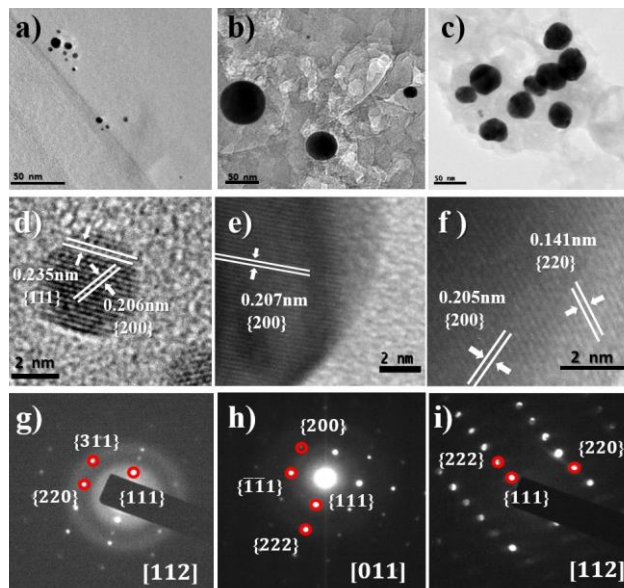


Fig. 2. TEM micrographs of NPs (top), HRTEM micrographs of NPs (medium) and SAED patterns taken of NPs (bottom). From left to right for nanofluid 1, 2 and 3.

Additionally, the laser-assisted production route seems not to affect the structural composition of gold NPs promoting unwanted chemical reactions among gold, the thermal oil and the TOAB; as it is seen in **Fig. 2 (d-f)** where the HRTEM measurements reveal, that the *d* interplanar spacings represented by (*hkl*) Miller index obtained from HRTEM micrographs in **Table 1** match with those included in Joint Committee on Powder Diffraction Standards (JCPDS) files for cubic Au crystals (JCPDS: 4-784).

Table 1. Measurement of *d*-Spacing taken from HRTEM micrographs of the crystalline NPs for a) nanofluid 1, b) nanofluid 2 and c) nanofluid 3.

d-Spacing/Å			(JCPDS:4-784 data)	value (hkl)	Phase assignment
a	b	c			
2.35±0.005			2.35	(111)	Cubic
2.06±0.005	2.07±0.005	2.05±0.005	2.04	(200)	Cubic
		1.41±0.005	1.44	(220)	cubic

In the same direction, diffraction measurements shown in the selected area electron diffraction pattern of the NPs match very well to the cubic structure of Au crystals. As it could be extrapolated from these results, the synthesized particles of Au seems to be free from vacancies or defects in the crystal due to the mixture of gold and another element that comes from the base fluid.

The EDX measurements confirm the purity of gold nanoparticles. It can be also observed that when the area irradiated by the electron beam is bigger, some extra elements like copper (Cu), carbon (C), potassium (K), chromium (Cr), iron (Fe) and bromine (Br) can be detected. The presence of copper and carbon is caused by the fact that the grid substrate is made of copper and it contains a carbon film; in the same direction, iron and chromium can be detected because the microscope objective lenses are

made of it. The presence of potassium and bromide in **Fig. 3 (b, c)** can be attributed to chemicals used to stabilize the nanofluids, indeed, bromide is a fundamental component of TOAB.

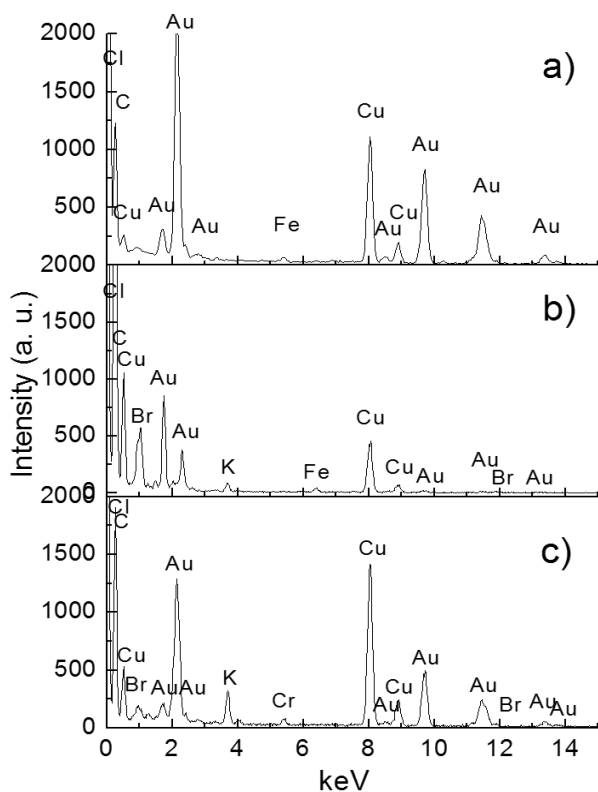


Fig. 3. EDX spectra of NPs (a) nanofluid 1, (b) nanofluid 2 and (c) nanofluid 3.

Size and stability of the nanofluids

Particle size was assessed from Brownian motion by means of the Einstein-Stokes equation as it is depicted in **Fig. 4**. Nevertheless, we have to take into account that the mean size and size distribution value shown for the first size measurement is a rough value estimation of the original size of fabricated NPs because during the time spent between the measurement and the nanofluid fabrication the NPs could start to agglomerate. In fact, the measured NPs size corresponds to the size of agglomerates, but for a particle agglomeration dynamics study this value represents a good figure of merit.

From analysis of the statistical size, the mean size and size dispersion of the formed clusters for nanofluid 1 were 58 ± 31 nm at FWHM. In contrast, in the nanofluid 2 the mean size was of 39 nm, while the size dispersion was the shortest one with a value of 42 nm. The nanofluid 3 shows a mean size of 108 nm and the higher size dispersion (about 124 nm of FWHM). These results are shown in **Fig. 4** (top side) where we observe that the production of gold NPs by PLAL and the subsequent photo-fragmentation allows us decreasing the size dispersion and the mean size of the NP clusters inside the thermal oil, avoiding in a good grade the agglomeration phenomenon in the production process. Moreover, the addition of a surfactant allows obtaining smaller clusters of particles without influencing the production process and the purity of the material.

To determine the stability, the mean size of the formed NP clusters inside the fluid was measured 13 times in 192 hours. The results are shown in **Fig. 4** (down side). The average size of Au particle clusters increases gradually within the first 96 hours due to the clusters growth. After that time, agglomerate size keeps constant and nanofluids can be considered stable for at least 192 hours. From the size study over the time it's noticeable that the nanofluid 2 reaches its stability at the lowest cluster's size of 370 nm. In order to corroborate the stability of nanofluid 2, the cluster's size was measured after one month and it was approximately the same. Therefore, the addition of TOAB using the PLAL technique largely alleviates the unwanted agglomeration phenomena, compared to NPs fabricated in pure thermal oil without any additive, and the commercial nanofluid. In addition, nanofluid 2 cluster size is considerably smaller (factor of 4) than those reported in previous works [15] using the same nanoparticle material, base fluid and chemical additive, but different production method.

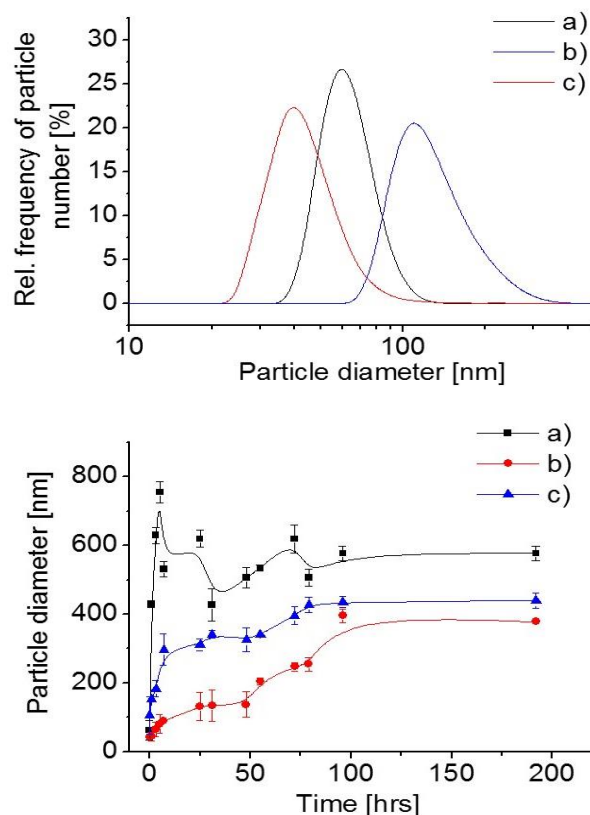


Fig. 4. Study of the size of the NPs of the three nanofluids. Complete size distribution few minutes after the fabrication process (top side) and the evolution of the NPs size over time (down side).

It is also noticeable that for the nanofluid 1, the nanofluid just produced by PLAL, the particle size increases to ~ 750 nm, then decrease to less than 600 nm. The possible reason for this is because, as it is well known, the dynamic light scattering technique is based on the scattering measurement that particles produce when they interact with the path of a laser beam which is located in a specific place of the DLS instrument vessel. When the clusters have settled, they don't interact any more with the laser path, and apparently the growth of the particle clusters

for the nanofluid 1 is so fast, as much as the clusters that reaches sizes above 750 nm tend to settle and fall down to the bottom of the DLS instrument vessel, don't allowing to measure this bigger clusters any more, which doesn't happen with the other two samples. Therefore, it can be concluded that the laser ablation and photo-fragmentation process already containing the surfactant required for each kind of material can be used to produce gold nanofluids highly stable from base fluids.

It can be concluded that the laser ablation and photo-fragmentation process can be used to produce gold nanofluids from base fluids already containing the surfactant required for each kind of material, more stable than those reported in previous publications.

Thermal conductivity vs. temperature

From the results shown in **Table 2**, it can be observed that the thermal conductivity of all nanofluids is higher than that of the base fluid. The addition of solid particles with higher thermal conductivity than the base fluid results in a thermal conductivity enhancement that depends on the solid content and can be predicted by the Maxwell equation [34]. Thermal conductivity of nanofluids produced by laser ablation and photo-fragmentation is higher than for commercial nanofluids due to the different degree of dispersion of the nanoparticles, making this technique more suitable to obtain nanofluids with improved thermal properties. Moreover, the addition of TOAB does not affect the thermal conductivity enhancement compared to the nanofluid produced by the same laser treatment but without any surfactant.

Table 2. Thermal conductivity enhancement at 80 °C.

Sample	k(W/mK)	Δk(%)
thermal oil	0.1280±0.0028	-
thermal oil+TOAB	0.1268±0.0030	-
nanofluid 1	0.1332±0.0014	4.06
nanofluid 2	0.1313±0.0011	3.55
nanofluid 3	0.1288±0.0018	1.58

Conclusion

In summary, we have demonstrated that owing to addition of the stabilizer we have been able to produce a colloid that contains well-dispersed NPs. Over time the NPs tend to form smaller clusters than any other method reported in literature, reaching its stabilization at 370 nm. Thanks to the fact that TOAB does not degrade at high temperatures, it was experimentally demonstrated that our nanofluid has a high stability but keeping the improvement of thermal properties. High stable nanofluid, low environmental impact and improvement of the efficiency are prerequisites for crossing the frontier between research and applied technology. Consequently, these findings open the possibility of a new strategy for the fabrication of nanofluids used in solar energy applications.

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Author contributions

Conceived the plan: EJ, JL, GM; Performed the experiments: RT, RM; Data analysis: RT, RM; Wrote the paper: RT, RM, EJ, OM, JL, GM. Authors have no competing financial interests.

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