Distinct advantages of using sonochemical over laser ablation methods for a rapid-high quality gold nanoparticles production

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Abstract
Gold nanoparticles (AuNPs) have numerous usage in various applications, primarily in chemical and biological sensing. There are also various methods of producing AuNPs. However, in some of those methods, removing excess reagents, including ions or residual surfactants, can be difficult and time-consuming, while the functionalization of AuNPs with other types of molecules can only be achieved at lower efficacy. In this report, the AuNPs were produced using a rapid and solvent-free sonochemical and laser ablation methods. Morphology, structure and the colloidal stability of AuNPs produced by the two methods were analyzed and compared. Results have shown that the purity, homogeneity and crystallinity of AuNPs produced using the sonochemical technique showed better characteristics than those acquired by the laser ablation method. AuNPs also demonstrated smaller particle sizes (22 nm), smooth surfaces with higher colloidal stability (50.7 mV). The findings indicate that ultrasound irradiation power improves the crystalline properties of AuNPs and inhibits their agglomeration. The sonochemical approach can, therefore, be used to produce AuNPs with better physicochemical characteristics compared to laser ablation methods.

1. Introduction

Because of the versatility of their physical and chemical characteristics, AuNPs are of growing interest in materials chemistry. Work relating to the synthesis and applications of AuNPs is becoming an unavoidable area of interdisciplinary study. At the nanometer scale, AuNPs have a distinct color appearance and are reported to have many potential applications, including catalysis, biosciences, optical materials, decorative coatings, drug delivery, nanodevices and nanoelectronics [1–10]. In AuNP synthesis, the control of scale, shape, and surface functionality are very critical issues. Good control of size and shape has been achieved with the AuNP synthesis by chemical reduction of gold ions in solution [11]. Unfortunately, in this case, removing excess reagents, including ions or residual surfactants, or the functionalization of AuNPs with other types of molecules, is difficult. A new approach based on laser ablation of the bulk metal in water has emerged for the synthesis of AuNPs [12]. Mafune et al have demonstrated that proper control of the dimensions of the nanoparticles can also be obtained through the second harmonic of a laser Nd:YAG (532 nm) as well as a surfactant like sodium dodecyl sulfate [13]. With this procedure, other results have been reported by Compagnini et al [14], which showed the possibility of synthesis AuNPs via laser ablation in the alkanes solvents. Unfortunately, in these solvents, many useful molecules for functionalizing AuNPs are not stable or soluble. Nanoparticle synthesized using laser ablation has been aggregated, homogeneous and has a wide size distribution [11, 15–18]. In contrast, the sonochemical approach for the preparation and development of nanostructures is of great interest. It is an effective and fast way to fabricating nanomaterials [19]. This is attributable to the acoustic cavitation mechanism.
that produces mechanical vibration (stirring influence), the hot spot that induces high pressure, high temperature and a high cooling rate of 1000 atm at 5000 K and $10^{10}$ K s$^{-1}$, respectively [20]. The sonochemical method with these unique and exceptional conditions will contribute to the rapid and efficient production and engineering of nanostructures.

Therefore, this study analyzes the physicochemical characteristics of AuNPs that are synthesized through sonochemical and laser ablation. The morphology, structure as well as colloidal stability of prepared AuNPs of both methodologies were analyzed and compared. This study sought to validate the potential of synthesizing AuNPs using a sonochemical method to achieve better physicochemical properties than the laser ablation process.

2. Materials and methods

2.1. Materials

The chemical reagents sodium citrate ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$) and chloroauric acid ($\text{HAuCl}_4\cdot4\text{H}_2\text{O}$), was used as a precursor to synthesize AuNPs. They had been bought from Sigma-Aldrich. All the chemicals were used directly without further purification.

3. Sonochemical method

AuNPs are prepared by employing a single pot and a rapid sonochemical process through the reduction of gold ions by sodium citrate. An ultrasonic processor (SONICS Vibra-cell Ultrasonics Processor (Model: VC 750)) with a solid horn tip size of 1/2 inch, frequency at 20 kHz and maximum output power of 750 watts was used for AuNP synthesis. 20 ml of aqueous (0.03 M) sodium citrate solution and 2 ml of aqueous $\text{HAuCl}_4\cdot4\text{H}_2\text{O}$ (0.03 M) solution were mixed simultaneously in the Erlenmeyer flask. A high-density ultrasonic (probe, horn) was applied to the solution during the mixing phase using an ultrasound power for 5 min. The solution's color changed from light yellow to dark red and finally reddish (figure 1).

4. Laser ablation method

Laser ablation is the removal of material with such short laser pulses is known for its high precision. For the gold nanoparticles formation, the gold plate was placed in the beaker filled with 10 ml distilled water. Target was irradiate by 1.064 $\mu$m Nd:YAG laser in 5 min (figure 2). The fluence was set at 1000 mJ cm$^{-2}$, the number of pulses was 2000, and the spot diameter was 2 mm. The laser ablation was carried out at 25 °C and atmospheric pressure. Based on the surface chemistry of the Au NPs formed in deionized water, we conducted studies on Au NPs synthesis, using the method of sonochemical and laser ablation in the water environment.

5. Results and discussion

5.1. Optical properties

One of the interesting optical properties of AuNPs is surface plasmon resonance (SPR) phenomenon. SPR is the collective oscillation behavior of metal nanoparticles conduction electrons after the incident electromagnetic wave interacts with the electrons at the conduction band. This phenomenon causes the electric field near the

![Figure 1. Schematic diagram of AuNPs synthesis using the sonochemical approach in 5 min.](image-url)
metal nanoparticles’ surface becomes remarkably increased. Consequently, an increased electric field increases the optical extinction significantly. The width and position of the SPR spectrum strongly depend on the size, shape and dielectric properties of metal nanoparticles as well as their chemical properties of surrounding media [21–23]. For metal nanoparticles with spherical geometry, SPR resonance arises in the visible and NIR spectra region [21]. For instance, the AuNPs colloidal with the range of 10–20 nm radius shows a strong light extinction around 520 nm of the UV–vis spectrum because of SPR [21]. SPR was also analyzed under atmospheric conditions within 400–700 nm using deionized water as a control sample. As seen in figure 3, AuNPs UV–vis spectra are formed by sonochemical and laser ablation. The SPR band of the sonochemical approach (AuNPs size: 22 nm) and the laser ablation (AuNPs size: 49 nm) showed strong extreme peaks at 522 nm and 529 nm, respectively. The plasmon peak of AuNPs synthesized using the sonochemical method was much narrower and more substantial than that of laser ablation, indicating the ability of the sonochemical method to generate small and homogeneous AuNPs compared to the laser ablation method. On the other hand, under laser ablation conditions, SPR peak was at around 529 nm with slight broadening due to the larger size of the AuNPs. More specifically, the explanation behind the redshift and broadening of SPR; As the AuNPs size increases, the number of electrons increases in direct proportion to the volume. Because of that, the extinction of AuNPs increases with the volume [24]. While the AuNPs size approaches the wavelength of light, the metal nanoparticles could no longer be homogeneously polarized by incident light. As the AuNPs size increases, there is increased radiative damping, which results in an increase in the scattering contribution and also increased plasmon linewidth [25].

5.2. Structure analysis of AuNPs
Figure 4. shows the XRD results of gold nanoparticles synthesis using sonochemical and the laser ablation method. The XRD pattern of the sample produced by the laser ablation process is not completely in line with the Au Ref. code (01–089–3697), which shows that the crystallinity of products is impure (figure 4(b)). As presented

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Figure 2. Schematic diagram of laser ablation (1.064 μm Nd:YAG laser) for the synthesis of AuNPs in 5 min.

Figure 3. UV–visible spectra of AuNPs synthesis using (a) sonochemical method (b) laser ablation method; the inset (i, ii) shows the colloidal dispersion of AuNPs.
in table 1, it is clear to see the changes in 2θ values as well as d-spacing values of (111) and (200) planes. For AuNPs synthesized using the laser ablation method, the 2θ value of (111) plane shifted from 38.179 to 38.1767, while the d-spacing value changes from 2.35536 to 2.36547 nm. For the (200) plane, 2θ value shifted from 44.375 to 44.4552 nm, while the d-spacing value changes from 2.03980 nm to 2.04884 nm. The peaks for (220), (311) and (222) are absent in the diffraction spectrum of AuNPs. Besides, XRD peaks intensities of the AuNPs produced using the laser ablation procedure are relatively low compared to Au ref. code: 01–089–3697. On the other hand, the XRD peak intensities of the AuNPs produced using the sonochemical approach are higher than those prepared using the laser ablation approach (figure 4(a)). All diffraction peaks precisely match with the face-centred cubic phase (Au ref. code: 01–089–3697). The XRD results were in good agreement with the previous studies [26, 27]. A significant (111) diffraction peak shows a significant increase in the network structure along (111) planes compared to (200). In addition, the other prominent diffraction peak (220) indicates the anisotropic (network) nature of the AuNPs. Besides, a very slight shift is observed in 2θ and d-spacing values (table 1). Therefore, the sonochemical method enhances better crystalline gold nanoparticles as the XRD spectral features (intensities and peak shifts) denote [28].

5.3. Size and morphology
Figure 5 shows that the morphology and size distribution of AuNPs has been studied using FESEM. AuNPs prepared using laser ablation (figure 5(b)) are spherical, semi-spherical in shape, and non-homogenous with a 20–90 nm particle size. The average size is about 49 nm, larger than those produced using the sonochemical
method (figure 5(a)). The sonochemical method developed AuNPs with a scale of 19–26 nm with an average size of about 22 nm. Ultrasound irradiation increases the pressure, temperature through the formation, growth and collapse of the bubbles, i.e., cavitation [29]. Cavitation causes a change in the symmetry of bubble degradation [30]. As a result, different nanoparticle structures are formed under different conditions through ultrasonic irradiation due to cavitation. Based on the SEM data, the particle size of the AuNPs under ultrasonic radiation decreased significantly due to micro-bubble formation and collapse arising from acoustic cavitation [31].

In laser ablation, the most important parameters that effect on size, shape and optical absorption are laser fluence, liquid medium, pulse repetition rate and laser wavelength. An increase in the size of the nanoparticles produced with target fitted with physical boundaries (types of liquid) may be explained based on the rate at which the thermalization of the plasma plume takes place. The previous studies documented that the formation of metallic nanoparticles by laser ablation is due to the plume’s thermalization. Thus, the rate at which the plasma plume cools down, plays an important role in this entire process [32]. A very high cooling rate of the plasma plume is expected to result in a smaller size of the nanoparticles produced. The formation mechanism of nanoparticles after the completion of the ablation process depends on two co-occurring processes, namely, nucleation and particle growth. The nucleation process starts as soon as the ablated species come together, followed by the coalescence of these nuclei that leads to the formation of bigger particles. If the thermalization is fast enough, the nucleation process dominates, leading to the formation of smaller nanoparticles.

By using the laser pulse method, the size and shape of AuNPs can be changed by change the spot size diameter and adjusting the focal length of the lens to achieve focal length. The size of AuNPs NPs using laser ablation depends on the density of metal atoms during the nucleation and growth processes and the temperature. Moreover, the expansion of the plasma plume in the liquid buffer produces a concentration gradient of the solvent’s atomic and molecular species, which is complementary to the concentration gradient of metal atoms. During the plasma plume expansion, several species can participate in chemical reactions. The solvent’s choice not only controls the shape, size and size distribution but also the composition of the nanoparticles.

**Figure 5.** FESEM image and size distribution of AuNPs produced using (a) and (c) sonochemical method and (b) and (d) laser ablation.
5.4. Dynamic light scattering (DLS)

Zeta potential, polydispersity index (PDI) and hydrodynamic size were measured using the Dynamic Light Scatter (DLS) analysis. The PDI and zeta potential have been used to assess the stability of the as-synthesized AuNPs. The zeta potential values of a nanoparticle contribute to the electrostatic potential produced by the aggregation of electrons on the surface of the nanoparticles. Because of their high specific surface area, AuNPs are resistant to agglomeration. Zeta potential assessment gives information about the particle surface charge to evaluate the suspensions’ physical stability. Highly positive or negative zeta potential results represent that the particles lead to the repulsion of each other without a tendency to aggregate. Nevertheless, there is no power to stop the clustering of particles with low zeta-potential values suggesting a high flocculation tendency. The overall criteria for separating stable and unstable suspensions are usually about $+30$ or $-30$ mV. The particles suspension with zeta potential greater than $\pm 30$ mV are considered stable [33–37]. The zeta potential values of AuNPs processed by the sonochemical and laser ablation methods were $-50.7$ mV and $-30.7$ mV, respectively (figure 6). The measured zeta potential value of AuNPs produced using the sonochemical approach is so much higher than that obtained by the laser ablation method. As shown in table 2, the PDI values for AuNPs prepared by sonochemical and laser ablation are 0.276 and 0.409, with hydrodynamic sizes of 76.71 nm and 116.6 nm, respectively (figure 7). The difference in size between the SEM and the hydrodynamic size is attributed to the hydrolysis molecules of water that generate a free radical of the OH that finally binds the particles together, confirmed by the negative charge of the zeta potential.

It can be concluded from experimental investigations that ultrasonic irradiation destroyed the large AuNPs clusters into smaller masses, allowing more free radicals to be attached to the particles. The sonochemical method stabilizes the colloidal solutions of AuNPs by functionalizing the structure and surface of the NPs, preventing the aggregation of the particles [38, 39]. Using sonic energy enables a variety of dispersion behaviors to be exhibited by various nanofluids. Thus, the basic sonochemical conditions for different nanofluids must be calculated. From previous research, it can also be concluded that the dispersion of AuNPs into a base fluid by direct sonication (horn/probe) is comparatively more effective than traditional methods because of the distinct

![Figure 6. Zeta potential analysis of AuNPs prepared through sonochemical method (red solid line) and conventional method (green solid line).](image)

| Method               | Zeta potential (mV) | PDI     | Hydrodynamic size (nm) | SEM average size (nm) | SPR (nm) |
|----------------------|---------------------|---------|------------------------|-----------------------|----------|
| Sonochemical         | $-50.7$             | 0.276   | 76.71                  | 22                    | 522      |
| Laser ablation       | $-30.7$             | 0.409   | 116.6                  | 49                    | 529      |

Table 2. Comparison of AuNPs synthesis using sonochemical and laser ablation methods through DLS, UV-Vis and FESEM.
interface involving energy and matter [40]. The sonochemical method allows for the possibility of a wide variety of chemical reactions and an array of diverse nanostructures to be synthesized [41]. Additionally, the acoustic cavitation dynamics depend on the local (uniform liquid) environment, in which the phenomenon acoustic cavitation produces shock waves and collapses the bubbles, which then creates higher pressures that exceed 10 kbar [42]. Sonication has caused phenomena that improve the attachment of the free radical to the particles, which is of a larger hydrodynamic size compared to the laser ablation method. This enhanced size describes the increased stability of AuNPs that are synthesized using a sonochemical method. The properties of nanoparticles are strongly dependent on the size and uniformity of size. Comparison between the two methods, based on our results (zeta potential value, PDI, hydrodynamic size, SEM average size, and SPR) indicates that the sonochemical method was better than the laser ablation method.

There are various mechanisms have a role in nanoparticle synthesis namely, plasma confinement by the liquid medium and cavitation bubble dynamics. Here, the atoms and small clusters ejected from the surface undergo many more collisions than they would for laser ablation in air or vacuum due to the much smaller mean free path for the liquid. The liquid medium also induces both inertial and plasma confinement and strong cavitation near the surface. These processes strongly influence the observed particle size and distribution that is obtained. The experimental studies suggest that cavitation bubble serves as a reaction chamber for the nanoparticle nucleation, growth, coalescence, and solidification. Researchers have also shown that parameters like laser wavelength, laser fluence and height of the liquid column also affect the size and size-distribution of the resultant nanoparticles [43, 44]. It has been shown that the size of nanoparticles formed depends on the grain size of the material forming the film and also on the thickness of the film [45].

6. Conclusion

In this work, the morphology, structure and colloidal stability of AuNPs produced by sonochemical and laser ablation have been analyzed and compared. It has been shown that the purity, homogeneity and crystallinity of AuNPs produced using the sonochemical technique are better than those acquired by the laser ablation method. Laser ablation is also disadvantaged by high input energy and a small laser-irradiating area for the evaporation of target materials. To achieve large-scale production of nanoparticles by laser ablation compared to the sonochemical method, it is necessary to use femtosecond pulses laser to achieve higher ablation rates. Furthermore, to generate shape-controlled nanoparticles, the laser system and the target environment parameters have to be precisely controlled.
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Conflicts of interest

Authors declare no conflict of interest.

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