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Photoacoustic study of Changes in Optical Properties of Colloids with Silver Nanoparticles Produced by Laser Ablation

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Abstract

Silver nanoparticles were produced by laser ablation of a silver target submerged in 10 ml of bidistilled water. A Nd:YAG laser operating at 1064 nm, 20.4 mJ of pulse energy, 6 ns of pulse duration and 10 Hz of repetition rate was used to realize the synthesis. After the synthesis, changes in optical properties of colloid were studied. To realize the study, the pulsed photoacoustic technique was used. Additionally, UV-Vis spectroscopy, dynamic light scattering and Z potential techniques were used to correlate results. After the synthesis, photoacoustic signals, absorption spectra, and size distributions were obtained at different times. Each absorption spectrum showed a unique peak attributed to spherical silver nanoparticles. It was demonstrated that the root mean square and correlation analysis of photoacoustic signals permit to study changes in optical properties of colloidal nanoparticles.

Keywords: Pulsed photoacoustic technique, correlation analysis, laser ablation in liquids.

INTRODUCTION

The synthesis of metal nanoparticles by chemical and physical methods is nowadays an important subject of study due to their wide range of applications. Metal nanoparticles have unique optical [1-3], electronic [4], antibacterial [5, 6] and magnetic [7] properties.

The pulsed photoacoustic technique consists of irradiating a sample with pulsed laser radiation. If the fluence (energy per area) used is below a threshold value, the regimen is called thermoelastic. The laser-matter interaction produces acoustic waves that can be detected by piezoelectric transducer. The photoacoustic technique has been used to study different laser-matter interactions, such as: ablation of solids immersed in liquids [8, 9], determination of absorption coefficients [10], monitoring molecular orientational order in polymeric films [11], formation and evolution of nanobubbles around nanoparticles [12] and generation of photoacoustic wave from plasmonic nanoparticles [13, 14].

Laser ablation in liquids is a versatile technique to produce colloidal metal nanoparticles. It consists in irradiating with a pulsed laser a metal target submerged in some liquid for several minutes. The interaction of the laser (energy densities greater than 10^8 Wcm^{-2}) with a target generates several mechanisms such as; the extraction of material, formation of plasma, formation of shock waves, evolution of a cavitation bubble [15, 16] and nanoparticles formation. The characteristics and properties of the nanoparticles depend on the parameters of the technique. Several studies report the effect of energy [9, 17] and wavelength [18, 19] on the size distribution of nanoparticles, laser pulse repetition rate [20-22] on efficiency of ablation, focusing conditions [23] on the concentration of nanoparticles and liquid medium on the stability of the colloids [24-29]. One of the advantages of laser ablation in liquids in comparison with chemical methods is that it does not necessarily require chemicals to realize the synthesis so pure nanoparticles can be achieved. The purity of nanoparticles is important for numerous applications. It has been reported that nanoparticles obtained by laser ablation in liquids can grow due to coalescence of atoms and ions dispersed in the solution [23]. The surface of nanoparticles obtained by laser ablation are charged [30, 31] and it is possible to form an oxide layer [32]. The interaction of charged nanoparticles and the liquid molecules can produce an electrical double layer surrounding the surface of nanoparticles [33]. The interaction between nanoparticles depends on all attractive and repulsive forces [28], such as attractive Van der Waals forces that cause growth and/or aggregation, and repulsive electrostatic forces that favor stability of nanoparticles.

In this work, we showed the utility of the pulsed photoacoustic technique to study changes in optical properties of colloidal silver nanoparticles synthesized by pulsed laser ablation in liquids technique. Colloids synthesized by laser ablation in liquids were chosen because it is not necessary to use chemical reagents during the synthesis. This is an advantage when analyzing the photoacoustic signals since the photoacoustic signal would be the result of the contribution of the solvent and the nanoparticles (No contribution of chemical reagents). Using the cross-correlation method, we compared photoacoustic signals stored for different times after the synthesis of a colloid. This analysis showed that decrease of correlation value was due to changes in optical properties of nanoparticles. We correlate our results with well-known methods such as Uv-Vis spectroscopy and dynamic light scattering.

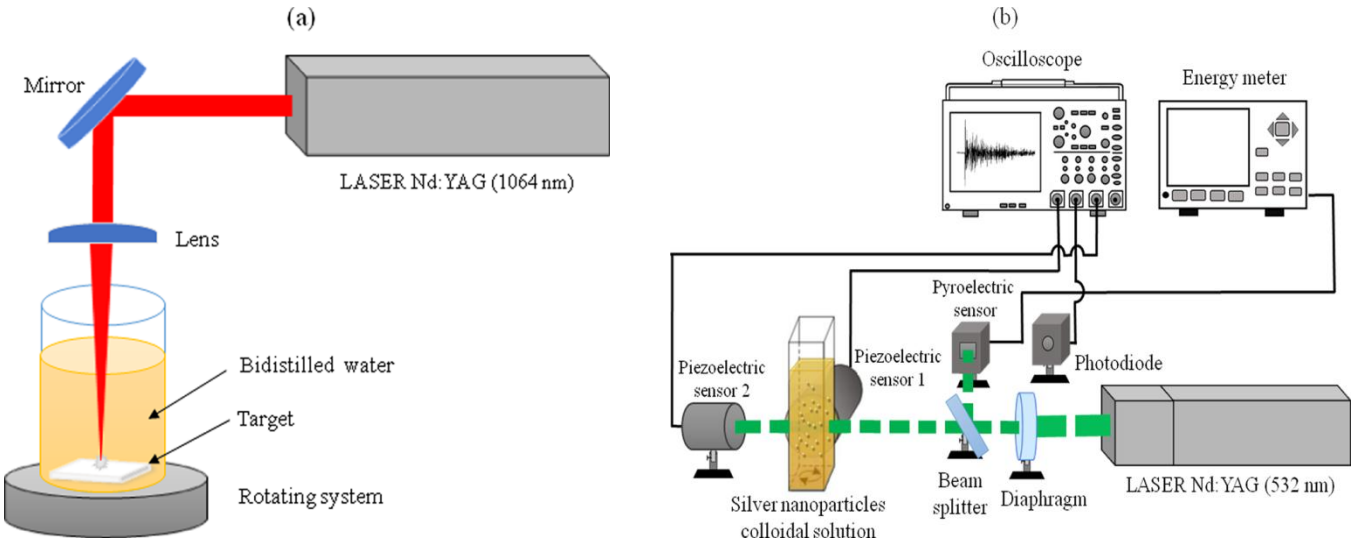

Figure 1. Schematic representation of experimental setups: (a) experimental setup used to realize the synthesis of silver nanoparticles by laser ablation of a silver target submerged in bidistilled water, (b) experimental setup used to study changes in optical properties of silver nanoparticles colloidal solution by pulsed photoacoustic and transmittance techniques.

EXPERIMENTAL

Figure 1a shows experimental setup used to obtain colloidal silver nanoparticles. A clean silver target (99.99 % pure from Sigma Aldrich) with dimensions of 1.3 cm x 1.4 cm x 0.2 cm was immersed in a bidistilled water (10 mL in a rotary glass vessel) and irradiated with the fundamental harmonic of a pulsed laser Nd:YAG "Brilliant" of Quantel (20.4 mJ pulse energy, 6 ns pulse duration, 10 Hz repetition rate and 5 minutes). A lens having a focal length of 10 cm focused the laser radiation on the target surface. The height of the liquid column was 2.5 cm. During the synthesis, the target was moved by the rotating system to avoid ablation of same spot area.

After the synthesis, three samples of 3 mL of colloid were taken. Sample 1 was analyzed using the pulsed photoacoustic technique, sample 2 was analyzed using ultraviolet-visible spectroscopy technique and sample 3 was analyzed by dynamic light scattering technique.

Figure 1b shows the experimental setup used to study changes in optical properties of sample 1 by pulsed photoacoustic technique. To obtain photoacoustic signals of sample 1, pulsed laser radiation of 532 nm, 6 ns of duration and 1 Hz of repetition rate was used. The wavelength of 532 nm was used because of its better absorption by silver nanoparticles compared with 1064 nm. Near to laser, a diaphragm was fixed to reduce the transversal section of the laser to 1 mm of diameter. After the diaphragm, a beam splitter was located. The reflected beam from beam splitter was directed towards the pyroelectric sensor of an energy meter (Newport model 1936-R) and the transmitted beam was directed towards sample 1. Sample 1 was contained in a 1 cm optical path-length quartz cuvette. A piezoelectric sensor of 254 kHz (piezoelectric sensor 1) was glued with epoxy resin to a face of the quartz cuvette, parallel to the direction of propagation of laser beam, to detect photoacoustic signal. After the cuvette, a second piezoelectric sensor of 136 kHz (piezoelectric sensor 2) was located to detect the pulsed transmittance of the colloid for 532 nm. The photoacoustic signal of sample 1 was displayed on a digital oscilloscope (Tektronix DPO 3054 500 MHz, 2.5GS/s). A fast photodiode (Thorlabs PDA10A-EC) was used for detection of laser and trigger the oscilloscope. Each photoacoustic signal in oscilloscope corresponded to a single pulse laser of 0.45 mJ of energy. For this purpose, the HiRes mode of the oscilloscope was used and only photoacoustic signals generated with 0.45 mJ of incident energy on the cuvette were a store. This energy was chosen because the photoacoustic signal of the cuvette with 10 mL of bidistilled water was negligible compared to the photoacoustic signal of sample 1. It was verified that the power used did not modify sample 1.

The absorbance spectra were obtained with a UV-Vis spectrophotometer Analytikjena SPECORD PLUS 250. The measurements by dynamic light scattering and Z potential were obtained with a PSS Niconp nano Z 3000 equipment

RESULTS AND DISCUSSION

Figure 2 shows the photoacoustic signal of a colloid, obtained 16 minutes after the synthesis. The inset shows the first 50 μs of the temporal evolution. This signal is characteristic of the colloid with silver nanoparticles obtained by laser ablation. In the thermoelastic regime, this signal is the result of the acoustic contributions of the nanoparticles and the liquid [34].

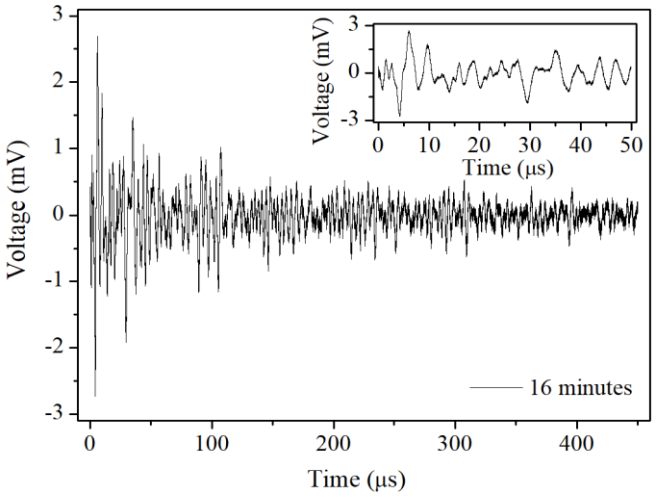

Figure 2. Photoacoustic signal of colloid with silver nanoparticles in bidistilled water. This signal was obtained at 16 minutes after the synthesis. The inset shows the time evolution during the first 50 μs.

Figure 3 shows the comparison between the photoacoustic signals obtained at 16 minutes and 350 minutes after the synthesis. When comparing the photoacoustic signals, correlation coefficient value of 0.55084, suggests changes in both form and amplitude.

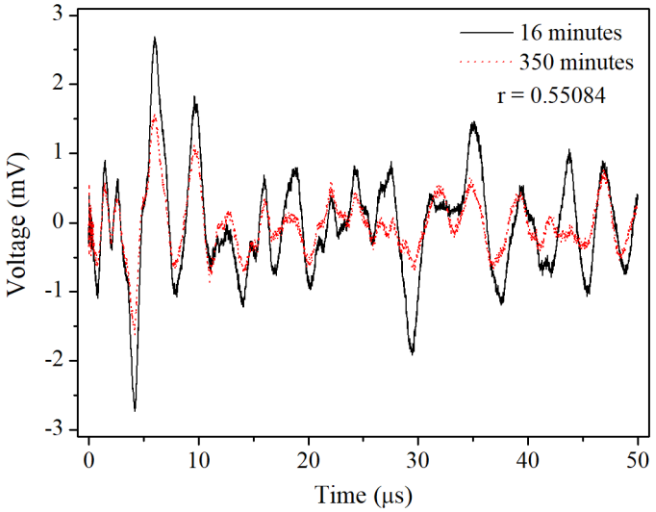

Figure 3. Comparison between the photoacoustic signals obtained at 16 minutes and 350 minutes after the synthesis. When comparing the photoacoustic signals was obtained the correlation coefficient of 0.55084.

Figure 4 shows the correlation coefficient (obtained when comparing the photoacoustic signal recorded at 16 minutes after the synthesis with each of the photoacoustic signals) as a function of the time elapsed after the synthesis. This figure shows a non-linear decrease of the correlation values in the first hundreds of minutes, after which no significant changes occur. This figure suggests that the optical properties of the colloid changed in the first hundreds of minutes and then did not present significant changes.

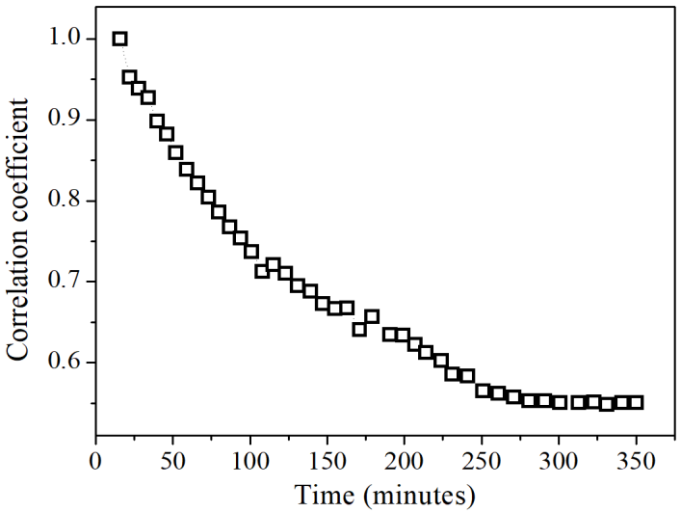

Figure 4. Correlation coefficient as a function of the time elapsed after the synthesis. It was correlated the signal obtained at 16 minutes after the synthesis with all the signals.

Figure 5 shows the root mean square (RMS) of the photoacoustic signal and the pulsed transmittance (detected by the piezoelectric sensor 2) of the colloid as functions of time. The RMS values of the photoacoustic signal decay progressively non-linearly and tend to be similar for the final data. The photoacoustic signal of the colloid is due to the absorption of the laser radiation [13, 35]. The observed behavior for the RMS values of the photoacoustic signal suggests that the absorption of the incident laser radiation of 532 nm decreased progressively in the first hundreds of minutes and subsequently there were no significant changes. The absorption efficiency of a colloid depends on absorption effective cross section, concentration and sizes distribution of the nanoparticles [1, 14]. The transmittance curve is similar to that of the RMS. The transmittance curve shows that the extinction of the incident laser radiation (due to the processes of absorption and scattering) increased in the first hundreds of minutes, after which not present significant changes. The results of Figure 5 suggest that the scattering of the laser radiation increased non-linearly in the first hundreds of minutes after the synthesis and then did not present significant changes. A progressive increase in the size of spherical silver nanoparticles results in an increase in scattering efficiency and a decrease in absorption efficiency [1]. When considering the results of Figure 5, it infers that the nanoparticles were added in the first hundreds of minutes. Some particles were sedimented due to their size and therefore it was presented a progressive decrease of the concentration of nanoparticles in the trajectory of the incident laser pulse of 532 nm. The increase in the size of the nanoparticles and their sedimentation caused the tendency to decrease the RMS values of the photoacoustic signal of the colloid in the first hundreds of minutes after the synthesis. If it is considered that stability of a colloid as non-significant changes in the optical

properties; Figure 5 also suggests that by using the photoacoustic signal can be determined the time from which a colloid it becomes stable.

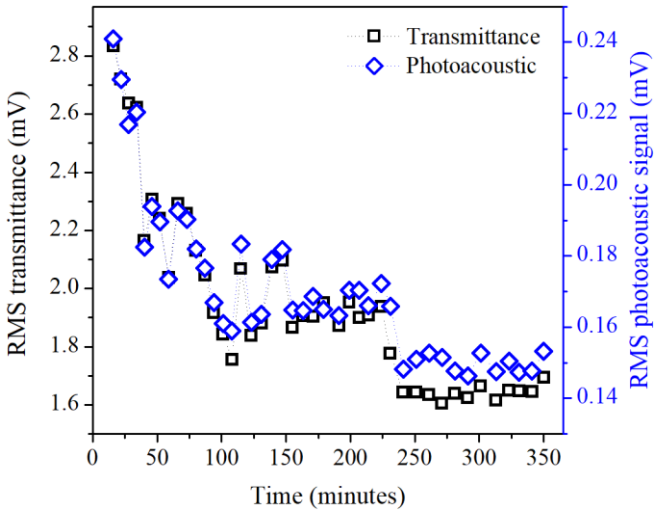

Figure 5. RMS and Transmittance of the photoacoustic signal of the colloid as a function of the time elapsed after the synthesis.

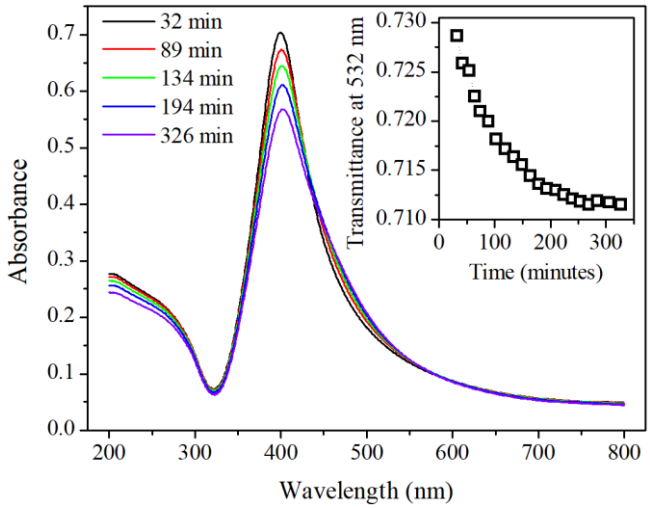

Figure 6. Absorption spectrum of colloid with silver nanoparticles as a function of the time elapsed after the synthesis. The inset shows the transmittance of colloid at 532 nm as a function of time.

Figure 6 shows the absorption spectra of colloid, obtained at different times after the synthesis. It is observed that as time elapsed, the shape and amplitude of the band corresponding to the surface plasmon change. The inset shows the non-linear decrease of transmittance values at 532 nm, which is similar to that obtained by the photoacoustic technique (see Figure 5).

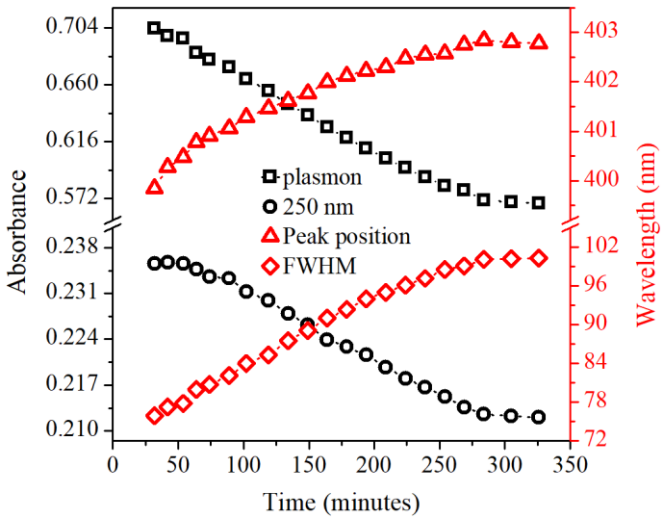

Figure 7. Absorbance of the surface plasmon (□), absorbance at 250 nm (○), position of the surface plasmon (Δ) and width at half height of the band corresponding to surface plasmon (FWHM) (◊) as a function of the time elapsed after synthesis.

Figure 7 shows the non-linear decrease in absorbance at 250 nm and absorbance corresponding to the maximum of absorption due to surface plasmon, in the first hundreds of minutes after the synthesis. The absorbance at 250 nm is proportional to the number of silver atoms present in the optical path of the incident light of the equipment Uv-Vis [36]. The observed decay for the absorbance values at 250 nm and maximum of absorption due to surface plasmon suggests that it was presented sedimentation in the first hundreds of minutes after the synthesis. Figure 7 also shows the non-linear increase in the position and width of the surface plasmon in the first hundreds of minutes after the synthesis. The redshift of the position of the surface plasmon is due to the increase of the average size of the nanoparticles [37]. The widening of the surface plasmon band is due to an increase in width of the nanoparticles sizes distribution [38]. In Figure 7 it is also observed that for the final data there were no presented significant changes in the absorbance values. The trends evidenced in Figure 7 suggest that nanoparticles are added in the first hundreds of minutes after synthesis. The increase in the size of the nanoparticles due to aggregation causes redshift in the absorption spectrum [39]. Some particles precipitate, which results in a decrease in the concentration in the optical path of the light and, therefore, at lower values of absorbance at 250 nm. The non-significant changes of the absorption spectra observed after the first hundreds of minutes in Figure 7 suggest that the optical properties did not change for the final data.

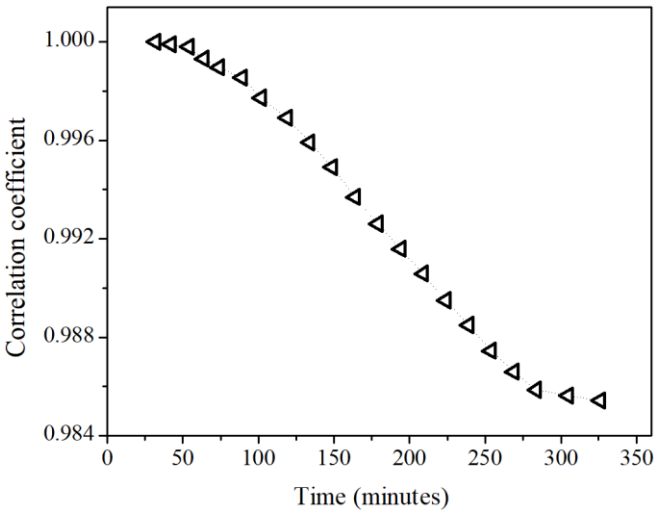

Figure 8. Correlation coefficient as a function of the time elapsed after the synthesis. The first absorption spectrum obtained at 32 minutes after the synthesis was correlated with all spectra.

Figure 8 shows correlation obtained when comparing the absorption spectrum for 32 minutes after the synthesis with all the spectra. A non-linear decay is observed in the first hundreds of minutes after the synthesis. For the final data, not evident significant changes. The results presented in Figure 4 and Figure 8 suggest that the colloid presented changes in its optical properties during the first hundreds of minutes after the synthesis, after which no significant changes were evidenced.

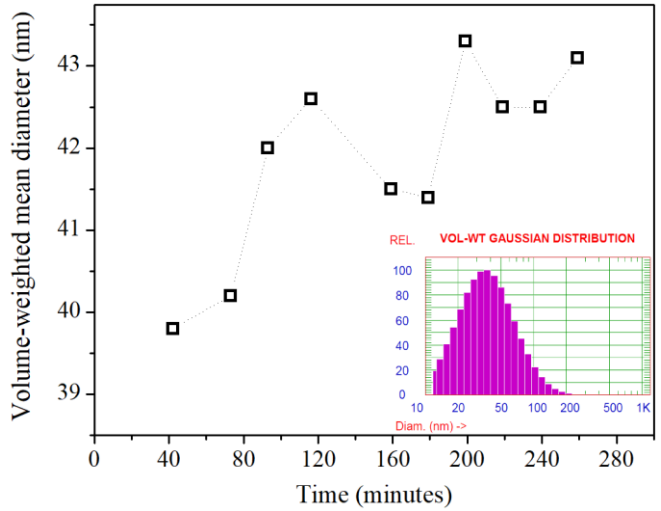

Figure 9. Hydrodynamic size of the silver nanoparticles as a function of the time elapsed after the synthesis. Inset shows volume weighted size distribution of silver nanoparticles for 259 minutes after the synthesis.

Figure 9 shows the hydrodynamic average diameter of spherical silver nanoparticles at different times after synthesis. The average diameter obtained when considering the volume distribution is presented. This figure shows that in the first hundreds of minutes after the synthesis, the hydrodynamic average diameter of the nanoparticles present a tendency to increase. The inset shows volume weighted size distribution of silver nanoparticles for 259 minutes after the synthesis. For this time, the average mean diameter was 43.1 nm and the standard deviation was 26.2 nm.

The Z potential obtained at 326 minutes after the synthesis was -32.21 mV. This value can be attributed to a stable colloid [40]. This result is coherent with no significant changes in correlation values observed in Figure 4 and Figure 8 for last data (hundreds of minutes after the synthesis).

CONCLUSIONS

Finally, our results evidenced that the photoacoustic signal (obtained in the thermoelastic regime) of colloids obtained by laser ablation in liquids can be used to study the changes in the optical properties of them. A good correlation was found between the results obtained by the photoacoustic technique and the techniques UV-VIS, dynamic light scattering and Z potential.

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