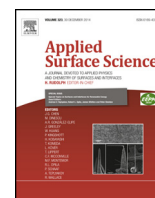




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## Formation of plasmonic colloidal silver for flexible and printed electronics using laser ablation

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### ABSTRACT

Laser ablation (LA) in liquids has been used for the development of various nanoparticles (NPs); among them, Ag NPs in aqueous solutions (usually produced by nanosecond (ns) LA) have attracted exceptional interest due to its strong plasmonic response. In this work, we present a comprehensive study of the LA of Ag in water, chloroform and toluene, with and without PVP, using a picosecond (ps) Nd:YAG laser and we consider a wide range of LA parameters such as the laser wavelength (1064, 532, 355 nm), the pulse energy (0.3–17 mJ) and the number of pulses. In addition, we consider the use of a secondary nanosecond laser beam for the refinement of the NPs size distribution. The optical properties of the NPs were evaluated by in situ optical transmittance measurements in the UV–vis spectral ranges. The morphology of the NPs and the formation of aggregates were investigated by Scanning Electron Microscopy and High-Resolution Transmission Electron Microscopy.

The ps LA process resulted in the development of bigger Ag NPs, compared to the ns LA, compatible with the size requirements of the printed organic electronics technology. The optimum conditions for the ps LA of Ag in organic solvents include the use of the 355 nm beam at low pulse energy (<1 mJ); these conditions rendered isolated Ag nanoparticles manifesting strong and well defined surface plasmon resonance peak. The use of the secondary ns laser beam was proven to be able to refine the nanoparticles to intermediate size between those produced by the single ns or ps LA.

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### 1. Introduction

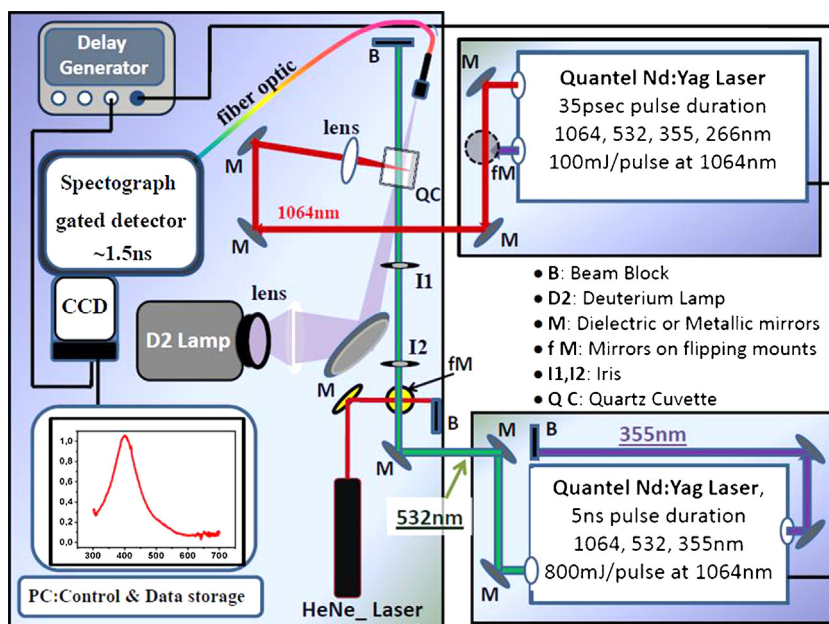
Organic and printed electronics promise to revolutionize the industries of renewable energy and solid state lighting by the production of large-scale and high-efficiency organic photovoltaics (OPV) and organic light emitting diodes (OLED). Since the original reports about solar cells with increased efficiency due to the incorporation of plasmonic nanoparticles [1–3], great efforts have been dedicated in developing the technologies of plasmon-enhanced OPV [4–9] and OLED [10–12]. Ag is the element that exhibits the stronger plasmonic effects, when in nanoparticle form. Therefore, it is of paramount importance to develop colloidal Ag solutions that would fulfill the requirements of organic and printed electronics, in particular:

- (1) The liquid solvent should be compatible with solvents routinely used for printed electronics (e.g. chloroform and toluene are used to print the standard P3HT:PCBM active layer of OPV).
- (2) The Ag particle sizes should range from a few nm to some tens of nm to maximize light absorption and scattering, respectively, and preventing the formation of Ag nanoparticles larger than 120–150 nm or micron-scale Ag particles that would short-circuit the OPV or OLED devices, since the thickness of the device individual layers is from 100 to 200 nm.
- (3) The Ag particles should have minimal or no capping, in order to maximize the scattering and the near field effects around them.

Laser ablation (LA) of solid Ag in liquid can meet all the aforementioned requirements. LA in various configurations was used previously for the production of colloidal Ag [13–28]. In particular, most of the relevant, reported works are dealing with colloidal Ag in aqueous solutions [13–23], while studies dealing with organic solvents, such as THF [24], PGMEA [25], various alcohols [13,26,27] and toluene and chloroform [27,28], DMF and THF [29], are less frequent

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**Fig. 1.** The experimental set up used for laser ablation of the solid target (primary ps laser), laser refinement of particles in the liquid phase (secondary ns laser) and, in situ optical transmittance spectroscopy.

and are focused to ns laser beams [25–29]; when a ns laser beam is used to ablate Ag in organic environment, the produced nanoparticles are exceptionally fine (about 5 nm) [29], therefore, their ability to scatter light is limited. There is still a lack of understanding the effects of pulse duration in the ps–ns range and the wavelengths spanning from infrared (1064 nm) to visible (532 nm) and to UV (355 nm) spectral ranges to the morphology of the produced Ag nanoparticles, as well as the stability of the solvent during the ablation process. The behavior of the solvent under laser irradiation is of particular interest, as it can be the source of various physical effects (e.g. beam confinement and particle cooling [30,31], and it can trigger photochemical reactions, such as photo-isomerization [32] and solvent pyrolysis (in the case of toluene) [33].

In this work, we aim to develop silver NPs plasmonic solutions for application in flexible printed organic solar cells. Therefore we aim to develop Ag NPs bigger than those produced by ns laser beams [29] and with size not exceeding 100 nm, immersed in solutions compatible with the printed solar cells technology, such as water (used as solvent to PEDOT:PSS), and volatile solvents like chloroform and toluene (used as solvents for the active layers, such as P3HT:PCBM). A ps laser beam was used to achieve cold ablation of the silver target and to avoid the stronger thermal effects that a ns beam induces. We study the effects of laser wavelength, fluence, and of the solvent (chloroform or toluene). We also consider the implementation of polyvinylpyrrolidone (PVP) additive, which was proven to be incompatible with the organic solvents and the ps laser beams, because it agglomerates upon laser irradiation.

Finally, we show that the ps laser beams produce bigger Ag nanoparticles, compared to ns beams [29], and the produced nanoparticles can be further refined to a desired size with the use of a secondary ns beam. By this combined, two step process, particles with intermediate sizes, between those produced by the exclusive use of ps or ns laser beams, can be produced.

## 2. Experimental

A home-made optical set up (Fig. 1) was used for the laser ablation process of the solid silver target (primary ps laser), the particle refinement in the liquid phase by laser radiation (secondary

ns laser) and in situ and *real-time* optical transmittance measurements.

The laser ablation of the Ag target (polished foil, purity 99.99% by Kurt J. Lesker company) was carried out using the 1064 nm, 532 nm and 355 nm beams of a 35 ps Nd:YAG laser by Quantel (primary laser). The silver target was placed inside a quartz cuvette that was filled with the solvent liquid (chloroform, toluene, or water). The primary laser beam was hard focused on the Ag surface using a fused quartz lens. The silver target was ablated by delivering laser pulses with 10 Hz repetition rate and varying the pulse energy in the range 0.8–17 mJ. The NPs size refinement was made by a secondary nanosecond laser pulse (5 ps), which irradiate and fragment the already developed Ag NPs in out-of-focus mode.

Time resolved transmission spectra are recorded after every single laser shot of the ps laser pulse used for the ablation of the Ag target or the ns pulse used for shaping the resulting nanoparticles in liquid phase. The spectrograph consists of an Andor iStar-CCD camera and an imaging monochromator (Andor-Shamrock 303i). A 150 l/mm grating was used offering a 1 nm spectral resolution and the ability to record in a single shot a region of 400 nm UV–vis spectrum. A D2 lamp is employed as a white light source for the transmission measurements and a couple of lenses are used for collimation and focusing through the liquid sample. Time “slices” of the transmitted D2 lamp spectrum, using gated amplification, in the order of 1 ms have been recorded at a fixed time delay (50 ms) with respect to the ps or the ns laser pulses. The delay time was controlled with sub-nanosecond accuracy via a time-delay-generator (Stanford Research Instruments), “clocked” to the Q-switch process of the ps and ns laser systems.

The Scanning Electron Microscopy (SEM) characterization were carried out in a JEOL JSM-6510LV SEM equipped with LaB<sub>6</sub> filament after drop casting and drying of the solution through evaporation of the solvent on aluminum tape; the SEM is equipped with an energy dispersive X-ray detector (EDX) for chemical analysis. Transmission Electron Microscopy (TEM) characterization for selected samples were performed in a JEOL JEM-2100 high resolution transmission electron microscope (HR-TEM) equipped with LaB<sub>6</sub> filament after drop casting and drying of the solution through evaporation of the solvent on standard hexagonal 600 mesh copper grids.

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