Electronic Supplementary Material (ESI) for Reaction Chemistry & Engineering.

Supplementary information

In situ synthesis of gold nanoparticles in polymer films under concentrated sunlight: control of nanoparticle size and shape with solar flux

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A. Materials and methods

A.1 Nanocomposite film preparation

All nanocomposites were prepared by spin coating a mixture of polymer and metallic precursors in an appropriate solvent. We used gold chloride ($HAuCl_4$ $3H_2O$, Aldrich) as metallic precursors and potato starch (STARCH, Extra Pure, Acros Organic) and poly(methyl methacrylate) (PMMA, M_w 15000, Aldrich) as polymers. All chemicals were used as received.
The precursor/polymer mixtures were prepared in two steps. We first prepared polymer solutions with mass concentration of 100 mg/ml. STARCH was dissolved in deionized water by heating under reflux while PMMA was dissolved in ethyl acetate (99% pure, Aldrich) under vigorous stirring. The appropriate amounts of metallic salts were then dissolved directly in the polymer solutions so as to obtain blends with a metal/polymer ratio of 20% (w/w). The solutions were then kept in the dark at room temperature until spin coating.
The spin coating was performed by dropping 200 μl of the metal/polymer mixture on a square glass substrate before spinning at 2000 rpm for 60 seconds. The glass substrates were previously cleaned by successive ultrasonic baths in propanol, acetone and deionized water and then dried under nitrogen. The same procedure was used for all samples.
All the samples were irradiated under CSI or annealed rapidly after deposition by using the setup described in the article.

A.2 Film thickness and topography

The thicknesses of the films were measured with a profilometer (DEKTAK) and were found to be around 300 nm and 500 nm, for the STARCH and PMMA composites respectively.

The surface topography of the films was characterized by AFM measurements (Model SMENA, NTMDT).

A.2 Solar spectra before and after concentration

Fig. S1 shows the experimental setup and intensity spectra at the solar simulator output before and after concentration. After passing through the Fresnel lens and the homogenization rod, the light loses intensity in the UV range. Consequently, after concentration, the light intensity decreases dramatically below 380 nm.

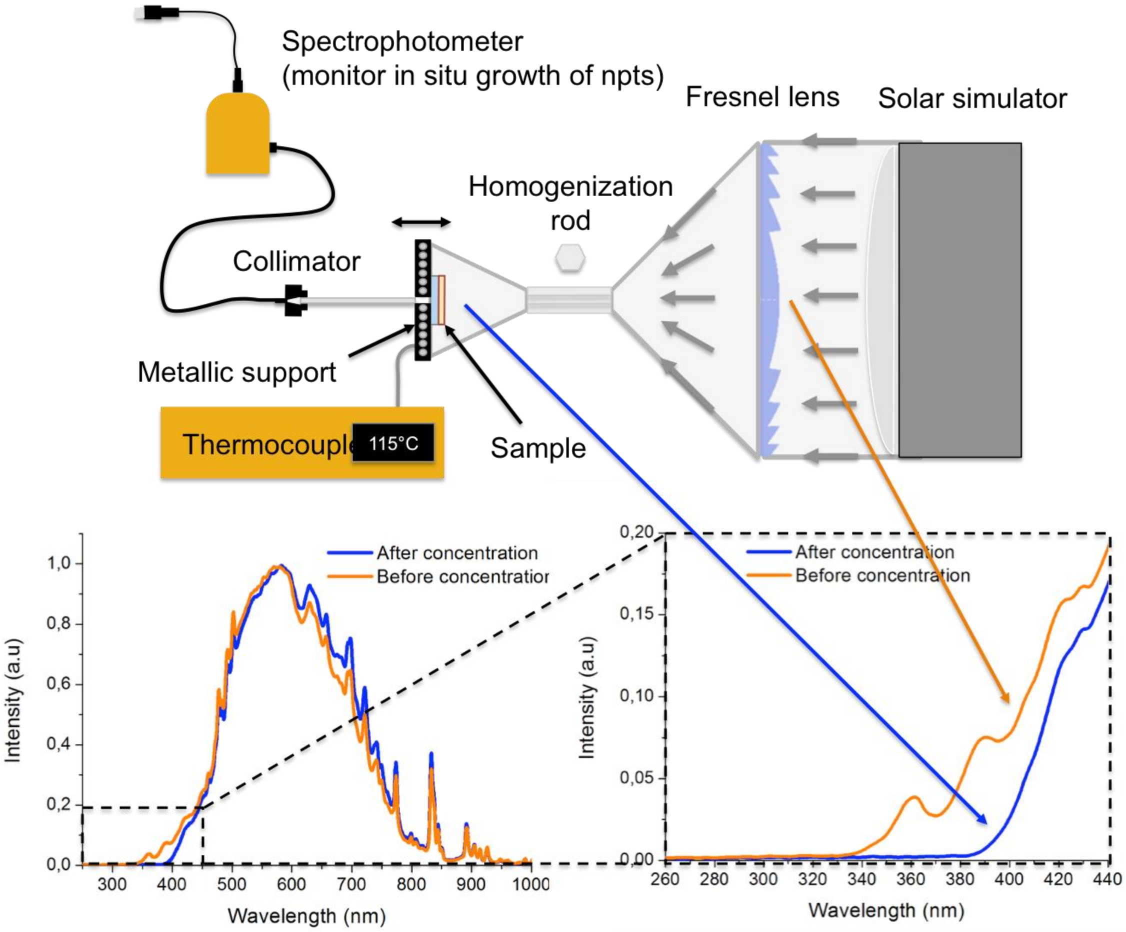

Figure S1: Normalized intensity spectra of the solar irradiation used for the fabrication procedure before and after concentration.

A.3 Control and homogeneity of the sample temperature during the synthesis

The sample holder has been designed so as i) to control at will the temperature of the sample between T_{amb} = 25°C and 150°C and ii) to permit the measurement of absorbance spectra.

The temperature control has been realized by bringing in contact the sample with a metal plate maintained at a controlled temperature by using a heater wire driven by a PID controller and a thermocouple. A detailed description of the holder structure is given in Fig. S2.(a). The thermocouple is glued inside a hole made in the metal which is almost as deep as the metal plate thickness to measure the temperature as close to the metal plate surface as possible. In order to verify the real temperature and its homogeneity at the sample surface, we have used an IR camera. The picture, shown in Fig. S2.(b) shows that the spatial distribution of the temperature is homogeneous across the sample surface and it very close to the temperature measured by the thermocouple (less than 5°C of uncertainty), see Fig. S2.(c).

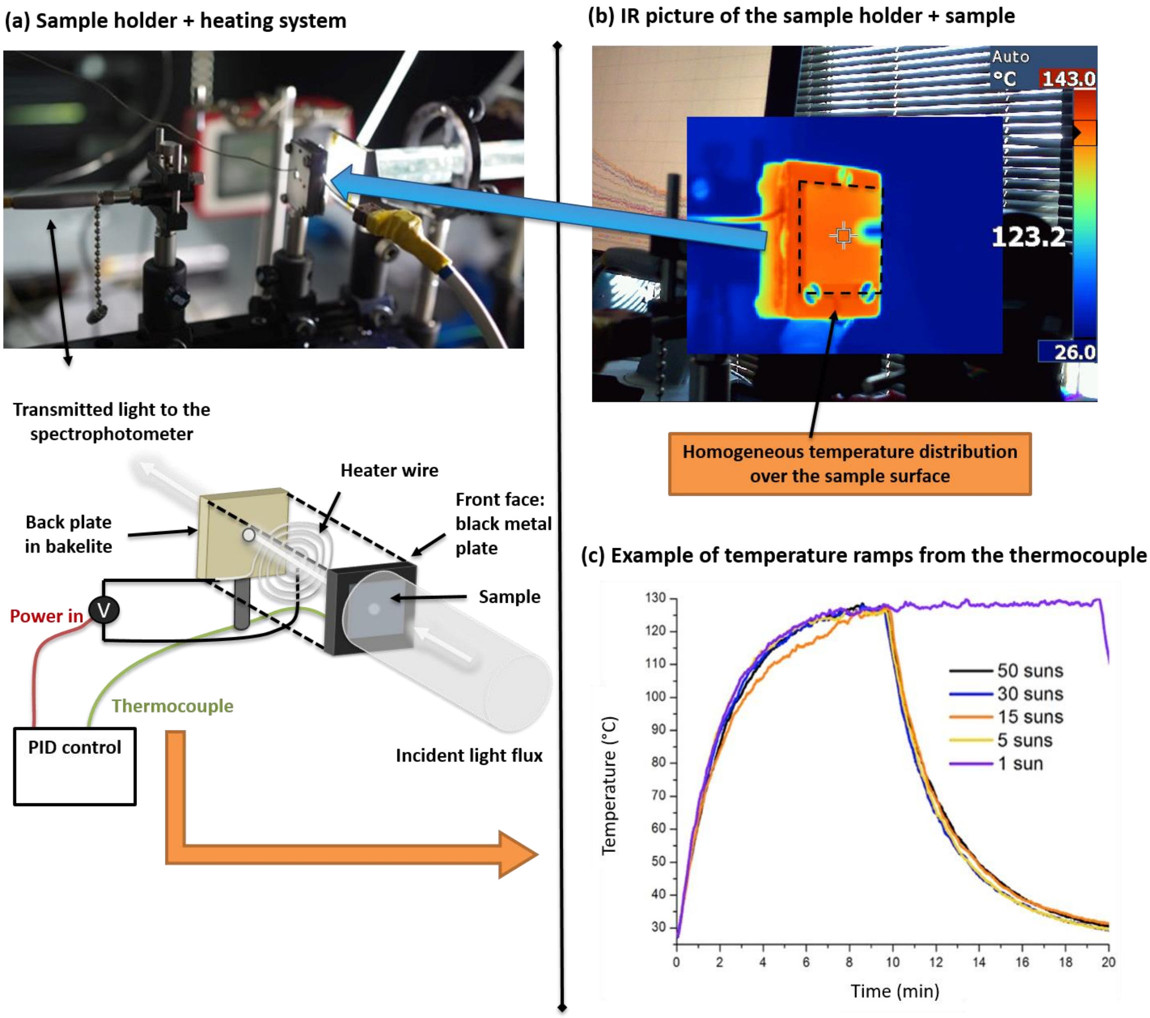

Figure S2: (a) Sample holder designed for temperature control (photo and schemes) (b) IR picture of the sample during synthesis (the blue line is an object between the camera and the sample) (c) Temperature profiles measured during the synthesis.

It should be emphasized that the goal of this setup is not to precisely control or know the absolute temperature of our samples but to permit reproducible conditions of temperature in order to be able to compare our experiments. Regarding this last point, if we consider the temperature ramp applied (approximately 10°C/min) along with the thicknesses and conductivities of the different elements, it is reasonable to presume that the temperature distribution is not limited by conduction and follows the command temperature almost in real time. Consequently, the temperature profiles that we measure are representative of the temperature profile of our samples.

B. Nanoparticle size distribution from TEM images.

The TEM measurements where performed by dissolving the film in an appropriate solvent in order to recover a drop of polymer solution containing the nanoparticles on a TEM grid.
Fig. S4 shows the particle size distributions measured for the Au/STARCH composite prepared under irradiation only.

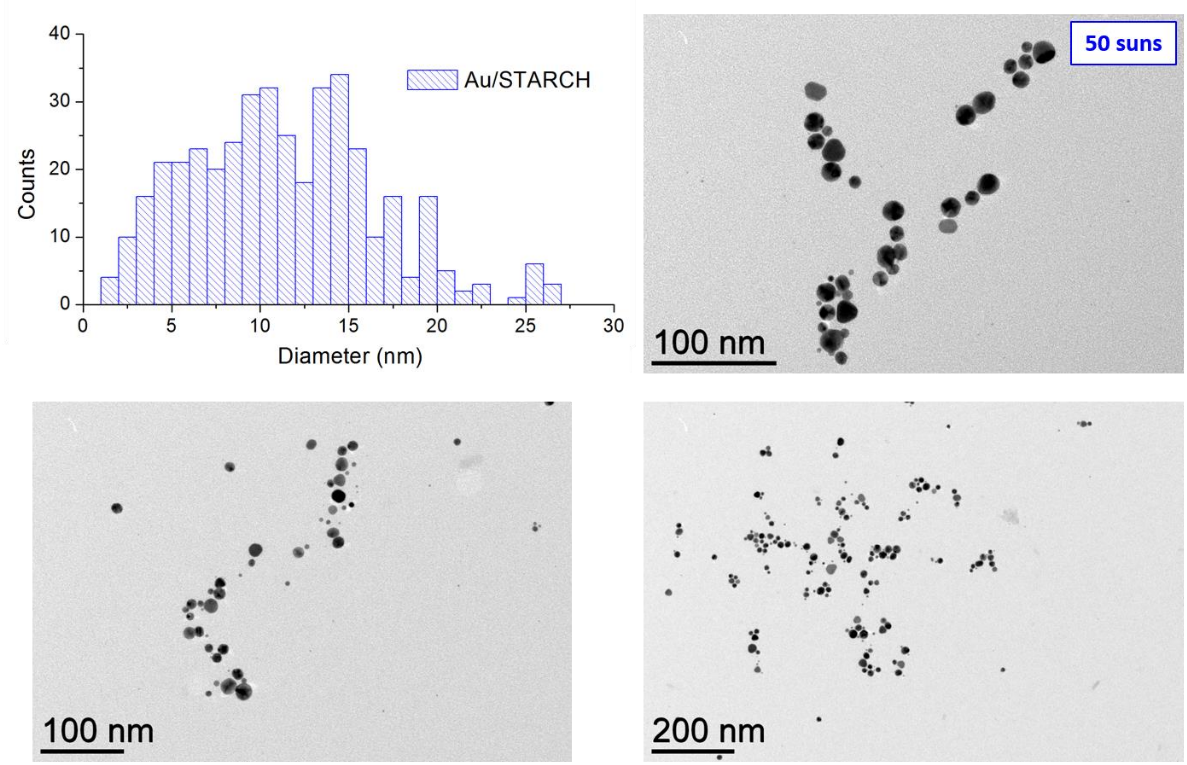

Figure S4: Nanoparticle size distributions and TEM images of the Au/STARCH nanocomposite prepared under irradiation at 50 suns (130°C).