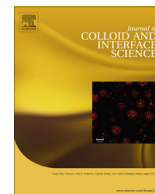




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Synthesis of mercaptopropyl-(phenylene)s-benzoates passivated gold nanoparticles: Implications for plasmonic photovoltaic cells



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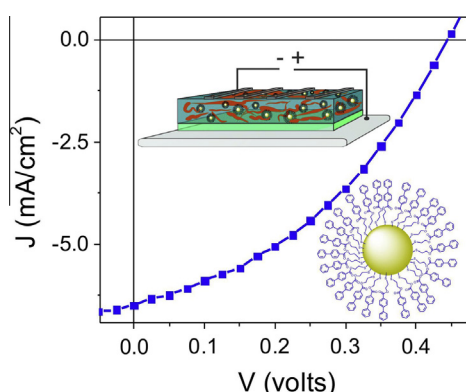
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HIGHLIGHTS

- Synthesis of new mercaptopropyl-(phenylene)s-benzoate ligands.
- Tetraoctylammonium bromide (TOAB) decreases the electrical conductivity of cells.
- Electrical property is the parameter that affects most the cells efficiency.

GRAPHICAL ABSTRACT



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ABSTRACT

The incorporation of gold nanoparticles in heterojunction solar cells is expected to increase the efficiency due to plasmon effects, but the literature studies are sometimes controversial. In this work, gold nanoparticles passivated with $(\text{Ph})_n-(\text{CH}_2)_3\text{SH}$ ($n = 1, 2, 3$) have been synthesized by reduction of tetrachloroauric acid with sodium borohydride in two ways: (1) one-phase where both the thiol and the gold salt are solubilized in a mixture of methanol with acetic acid: Au-s-(Ph)_n or (2), two-phase, using tetraoctylammonium bromide (TOAB) to transfer gold from water to toluene where the thiol is solubilized, Au(TOAB)-s-(Ph)_n. The morphological, experimental and simulated optical properties were studied and analyzed as a function of the thiol and of the synthetic procedure in order to correlate them with the efficiency of plasmonic hybrid solar cells in the following configuration ITO/PEDOT:PSS/P3HT:PCBM-C60: Au-nanoparticles/Field's metal, where PEDOT:PSS is poly(3,4-ethylenedioxythiophene)-poly(styrenesulfonate), P3HT is poly(3-hexylthiophene-2,5-diyl) and PCBM-C60 is [6,6]-Phenyl C61 butyric acid methyl ester. Our findings indicate that the gold nanoparticles incorporation is affecting the electrical properties of the active layer giving a maximum efficiency for Au-s-(Ph)₃. Moreover, TOAB, which is usually used in the synthesis of thiol passivated gold nanoparticles, has negative effects in both plasmonic and electrical properties. This result is important for optoelectronic applications of gold nanoparticles prepared with any procedures that involve TOAB.

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1. Introduction

Conjugated polymer solar cells (CPSC) have emerged as an interesting alternative to classical silicon based devices, due to the cheaper and easier methods of deposition for these materials, combined with the possibility of preparing the cells on flexible substrates. One of the most used CPSC configurations is the bulk heterojunction, where electron – donor and – withdrawing materials are physically mixed in the active layer. Soluble fullerene derivatives such as [6,6]-phenyl-C61-butyric acid methyl ester (PCBM-C60) [1] and [6,6]-phenyl-C71-butyric acid methyl ester (PCBM-C70) [2] are typically applied as electron acceptors, while the most used electron donor conjugated polymer is poly(3-hexylthiophene), P3HT [3]. Despite that efficiency of up to 10% was achieved recently with CPSCs [4], this is still far from the corresponding inorganic counterparts, which is typically 30–40% for silicon wafers devices.

The major drawbacks of organic photovoltaic materials are their limited absorption range and lower charge mobilities with respect to inorganic semiconductors. One strategy to overcome this problem is to prepare hybrid plasmonic cells, combining the interesting optical and electrical properties of noble metal nanoparticles with the processability and photovoltaic properties of conjugated organic materials [5]. The effect of noble metal nanostructures on the solar cell's efficiency, however, is not always predictable. For instance, Kim and Carroll [6], Heeger [7] and Jin [8], among others, reported an increase in all of the photovoltaic parameters for conjugated polymers devices having embedded gold and silver nanostructures, and ascribed the increases to a higher charge transport. On the contrary, Borchert, for instance, obtained a decrease in the efficiency of bulk heterojunction P3HT:PCBM-C60 solar cells through the incorporation of gold nanoparticles prepared with either dodecylamine as ligand or P3HT [9]. The authors explain this result with the quenching of the excited state and possible segregation of gold nanoparticles at the interface with the cathode. However, at least for P3HT passivated particles, the reason could also be related to the use of tetraoctylammonium bromide (TOAB). In fact, the synthesis is performed by adapting the Brust two-phase method [10] where TOAB is employed to transfer the gold salt from water to the organic phase. TOAB is highly soluble in most of the organic solvents, either polar or non-polar. So the precipitation with methanol used in Ref. 9 to purify the P3HT gold particles is not expected to eliminate TOAB. In fact, in the reference cited for the synthesis [11], the authors sketch the formation mechanism of the particles depicting TOAB and not P3HT as the direct ligand.

On the other hand, it is well known that the optical properties of plasmonic particles depend on their size, shape and medium. These morphological parameters not only affect the plasmonic band [12] but also the electrical transport properties [7]. Moreover, the incorporation of metallic nanostructures in a bulk heterojunction active layer impacts the morphological quality of the interface between the electron acceptor and electron donor materials, which in turns depends on the compatibility between the ligand and the organic phase.

In this paper we want to address the effect of the size and ligand of gold nanoparticles on solar cell efficiency by incorporating in P3HT:PCBM-C60 heterojunction devices gold nanoparticles (AuNPs) stabilized with thiols that bear a phenyl biphenyl or terphenyl group. This is aimed at modulating the size and size dispersivity of the particles, which should affect the optical and photovoltaic properties of the hybrid materials. Moreover, the aromatic nature of the ligand is expected to promote a good interaction with the organic conjugated mixture. On the other hand, the particles are prepared by one-phase synthesis, i.e. with the thiol ligands directly attached to the gold surface and by two-phase

synthesis, where TOAB is used as a phase transfer agent. The latter was chosen to improve our understanding of the role of TOAB on the particle size and morphology and finally on the cell behavior.

2. Materials and methods

2.1. Materials

The following chemicals were obtained from Aldrich and used without further purification: 3-chloropropane-1-thiol, isocyanatoethane, p-terphenyl-4-carboxylic acid, biphenyl-4-carboxylic acid, benzoic acid, 1,8-diazabicyclo [5.4.0] undec-7-ene (DBU), tetraoctylammonium bromide (TOAB), sodium boron hydride (NaBH_4), Gold (III) chloride hydrate ($\text{HAuCl}_4 \cdot x\text{H}_2\text{O}$), PCBM-C60 and P3HT were from Rieke Metals, Inc., PEDOT:PSS (Clevios P®) was from Heraeus, while Field's metal was from RotoMetals, Inc. Indium Tin Oxide (ITO, 8–12 Ω/cm^2) substrates were obtained from SPI Supplies®. CH_2Cl_2 , CHCl_3 , hexanes, diethylenamine, NH_4OH , methanol and spectroscopic grade solvents were purchased from Aldrich and Baker.

2.2. Characterization techniques

^1H (300 MHz), ^{13}C NMR (75.4 MHz) spectra were obtained at room temperature with a Jeol Eclipse spectrometer using CDCl_3 as solvent and internal reference. High resolution transmission electron microscopy (HRTEM) studies were carried out using a FEI-TITAN-300 kV field emission gun microscope, which has a symmetrical condenser-objective lens S-TWIN type (with a spherical aberration $\text{Cs} = 1.25 \text{ mm}$). Images were acquired with a CCD camera. The nanoparticles were deposited by casting a dilution of the reaction suspensions on copper grids. Average particle sizes were obtained after the statistical analysis of 300 particles and a lognormal distribution function. UV–Vis spectra were recorded on a Shimadzu 2401PC. For the absorption coefficient, ϵ , we consider the absorption within a 1 cm path length cell of a nanoparticle suspension with known concentration. In order to obtain the concentration, 1 mL of the original suspension (as obtained from the synthesis) was dried in a weighed vial and the solvent was left to evaporate. Another mL of the original suspension was diluted and used for the absorption spectrum. The concentration and absorbance values were introduced into the Lambert–Beer equation.

AFM morphological characterization was performed on a DimensionTM 3100 from Digital Instruments with a Pt-coated Si tip (15 nm nominal radius model:OSCM-PT Bruker, required for EFM studies). In the tapping mode, the images were obtained at a scanning rate (256 lines) of 0.2 Hz. The presence of conductive zones in the samples was investigated using the AFM instrument in electrostatic force microscopy (EFM) mode, for which a voltage of 12 V was applied to the tip and the work distance (separation between tip and sample during scanning) was 100 nm. The scanning rate and lines were the same as in the tapping mode. In the EFM images dark regions are associated to conducting zones and bright regions are insulating regions.

2.3. Solar cells

Solar cells were fabricated with the configuration: ITO/PEDOT:PSS/active layer/Field's metal. The ITO slides were cleaned in a Branson ultrasonic bath with different solvents: (1) methylene chloride (10 min); (2) hexane (10 min) and (3) methanol (two cycles of 20 and 40 min, respectively). PEDOT:PSS was

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