



Immobilization of synthetic gold nanoparticles on a three-dimensional porous electrode



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ARTICLE INFO

Keywords:

Electrografting
Nanoparticles
Graphite felt
Gold
Modified electrode

ABSTRACT

Synthetic gold nanoparticles were successfully immobilized on graphite felt fibers previously functionalized by sulfur groups. The nature of the functional groups used to tether them on the surface and the nature of the ligand grafted on nanoparticles play important roles in the immobilization process. The findings offer an effective way to develop three-dimensional porous materials modified by nanoparticles with controlled chemical composition, shape and size.

1. Introduction

Thanks to their unique strong optical absorption combined with their conduction properties, gold nanoparticles (Au NPs) are very attractive candidates for various interesting applications in, for example, catalysis, biosensing and electroanalysis. It has been demonstrated that their size, their composition as well as the plasmonic coupling within the Au NPs network have a significant influence on their electron conduction and optical properties [1–4]. Gold nanoparticles (Au NPs) are particularly interesting for catalysis [5,6]. Their catalytic properties are highly dependent on the size of the nanoparticles [3,4]. In order to take full advantage on Au NPs properties in catalysis, it is interesting to immobilize them on porous substrates, leading to high active surface areas on a support that is fully suitable for flow catalysis. The methods used to immobilize nanoparticles on porous supports are usually based on *in situ* reduction of gold salts by a chemical reducing agent [7–9] or by electrodeposition and galvanostatic displacement reactions [4,10,11]. However, the immobilization of synthetic nanoparticles has been successfully achieved on flat surfaces by electrostatic interactions or by forming covalent bonds such as S–Au, NH–Au and C–Au [12–14]. The synthesis of nanoparticles in solution has the advantage to offer a good control of their size distribution, shape and composition. To our knowledge, the immobilization of previously synthesized gold nanoparticles on porous surfaces has not been reported yet. Indeed, the difficulty lies in the fact that it can lead to nano-aggregation and the ligands used to stabilize the nanoparticles can prevent their binding with the functionalized surface [7].

In this work, we investigated the immobilization of synthetic Au NPs on a graphite felt functionalized with amino and sulfur groups. The role of the interaction strength between the nanoparticles and the three-dimensional porous support will be underlined. The effect of the ligand nature used to functionalize gold nanoparticles in solution will be also studied.

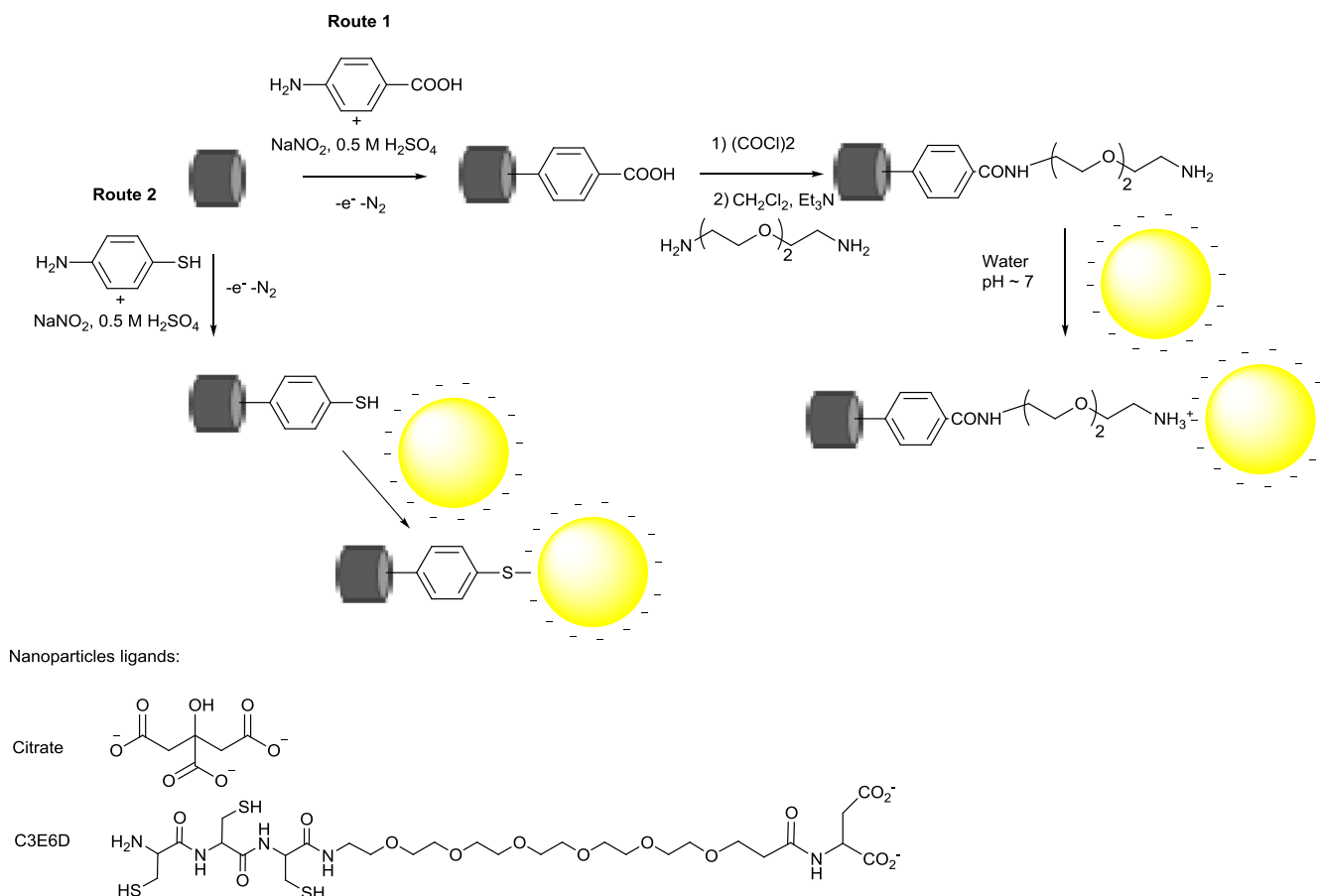
2. Experimental

2.1. Chemicals and materials

4-Aminobenzoic acid (99% purity) was purchased from Fluka and sodium nitrite and 4-aminothiophenol from Acros. Oxalyl chloride was from Alfa Aesar and 2,2'(Ethylenedioxy)bis ethylamine from Sigma-Aldrich. Graphite felt (RVG 4000) was supplied by Mersen (France). Au NPs were prepared according to literature procedures. 1 mL of a sodium citrate aqueous solution (1% by weight) was poured into 50 mL of boiling HAuCl₄ aqueous solution (0,01% by weight) under vigorous stirring [15]. The solution was boiled for 15 min and then cooled at room temperature under continuous stirring to give a mother solution evaluated at 80 nM by UV/vis absorption and an average particle diameter of 16 nm as determined according to literature [16]. The citrate ligand of the obtained Au NPs was then exchanged with an optimized PEGylated peptide bearing three cysteins (noted C3E6D) as shown in Scheme 1 [17,18].

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Scheme 1. Modification of the graphite felt by Au NPs.

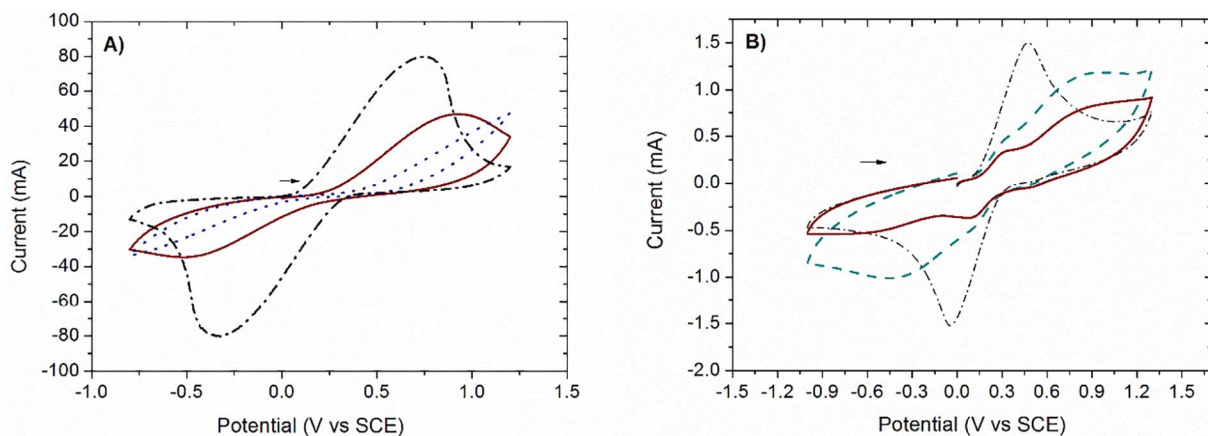


Fig. 1. Cyclic voltammograms of $K_4[Fe(CN)_6]$ ($5 \times 10^{-3} \text{ mol L}^{-1}$) in 0.5 M phosphate buffer pH = 7 A) at graphite felt electrodes (10 mm diameter and 12 mm width) before (---) and after grafting by 4-aminothiophenol (—) and 4-aminobenzoic acid (.....) diazonium salts B) at graphite felt electrodes (2 mm diameter and 3 mm width supported on a graphite rod) before (---) and after grafting by 4-aminothiophenol diazonium salt (—), and then addition of GF-Ph-SH-Au NPs (C3E6D, 8.10^{-8} M) (—). Scan rate 0.1 V s^{-1} .

2.2. Instrumentation

Electrografting of the graphite felt (48 mm diameter, 12 mm width) was performed in a home-made flow cell [19]. Potential control was performed using VersaSTAT3 AMETEK Model (Princeton Applied Research) potentiostat/galvanostat.

Electrochemical analysis was performed using a three-electrode cell with a graphite felt (1 cm^3) as working electrode, a platinum plate as counter electrode and a SCE as reference electrode in a solution of 5 mM

$K_4Fe(CN)_6$ in 0.5 M phosphate buffer solution (pH = 7).

2.3. Preparation and modification of the graphite felt

5 mM 4-aminothiophenol or 50 mM 4-aminobenzoic acid were solubilized in degassed 0.5 M H_2SO_4 . 5 mM or 0.15 M sodium nitrite were then added at 0°C to generate *in situ* the aryl diazonium salts of 4-aminobenzoic acid and 4-aminothiophenol, respectively. The electrografting of the graphite felt was then carried out by

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