



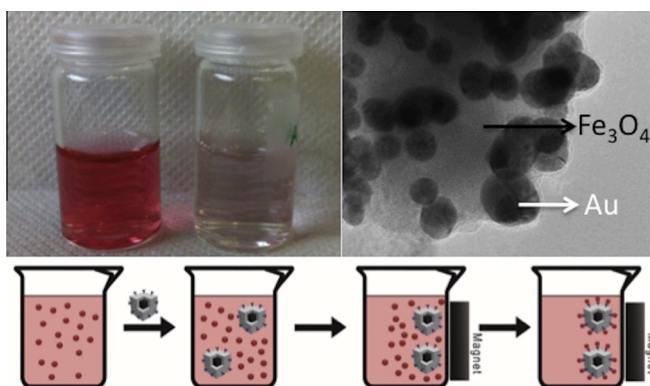
# Functionalized magnetite particles for adsorption of colloidal noble metal nanoparticles



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## GRAPHICAL ABSTRACT



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## ABSTRACT

Magnetite (inverse spinel type) particles have been surface-modified with siliceous shells enriched in dithiocarbamate groups. The deposition of colloidal noble metal nanoparticles (Au, Ag, Pt, Pd) onto the modified magnetites can be performed by treating the respective hydrosols with the magnetic sorbents, thus allowing their uptake from water under a magnetic gradient. In particular, for Au colloids, these magnetic particles are very efficient sorbents that we ascribe to the strong affinity of sulfur-containing groups at the magnetite surfaces for this metal. Considering the extensive use of Au colloids in laboratory and industrial contexts, the approach described here might have an impact on the development of nanotechnologies to recover this precious metal. En route to these findings, we varied several operational parameters in order to investigate this strategy as a new bottom-up assembly method for producing plasmonic-magnetic nanoassemblies.

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## 1. Introduction

In the past years, there has been great interest in exploring innovative solutions to environmental problems based on the application of nanomaterials, such as in water treatment

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technologies [1–3]. On the other hand, there are also concerns about the potential impact on the environment of increasing amounts of engineered metal nanoparticles that have been produced as a consequence of research activities worldwide or from diverse products and technological applications [4–8]. Considering the increasing production of wastes that in certain cases contain noble metal nanoparticles, methods that allow the capture of such nanoparticles from aqueous wastes are of potential interest, not

only due to economic reasons, but also based on environmental impact criteria. In this context, a promising nanotechnology for water decontamination involves its treatment with iron oxide nanoparticles as magnetic sorbents [9–11]. This technology offers the advantages of the surface-mediated sorption behavior of nanomaterials associated with their ability to be collected from water by applying a magnetic gradient, thereby allowing the recovery of noble metals from wastes. Among the parameters that can be exploited in the design of such type of sorbents, the possible size tuning of the magnetic behavior and surface chemical functionalization for specific target contaminants appear as particularly useful. The latter is the focus of this research; indeed, although this approach has been applied to remove several types of water contaminants [12–18], to the best of our knowledge, the uptake of noble metal nanoparticles using functionalized iron oxides has not been reported yet.

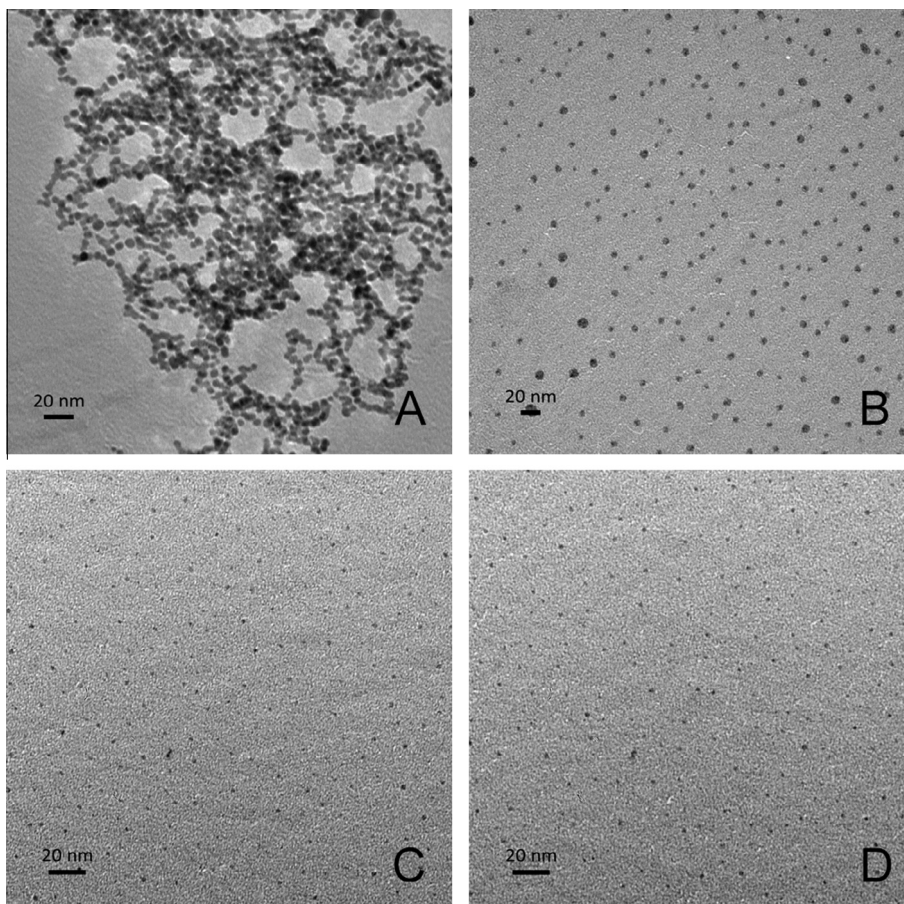
In order to improve the sorption capacity and selectivity of nanoadsorbents for target pollutants, a number of surface modification methods have been explored [19]. For example, silica-coated magnetic nanomaterials have been used because amorphous silica shells confer robustness to the magnetic cores and offer convenient platforms for chemical derivatization, thus making easier surface anchorage of different functional groups. The functionalization of silica surfaces with sulfur donor ligands appears particularly useful to bind to soft metals, for which sulfur shows a strong chemical affinity, such as noble metals [20]. This strategy has been used, for instance, in the functionalization of superparamagnetic nanoadsorbents using dimercaptosuccinic acid aiming the removal of toxic metals, such as Hg, Pb, Cd, and Tl [21]. Also, core/shell-type iron oxide particles functionalized with dithiocarbamate groups have shown high efficiency for the uptake of aqueous mercury via

magnetic separation [22,23]. In this work, we show that the chemical functionalization of silica-coated magnetite particles with dithiocarbamate groups confers strong affinity to noble metal nanoparticles. Despite their distinct behavior in aqueous solution, as compared to dissolved solute species, metal colloids could still be removed from water by using the functionalized magnetite particles. En route to these findings of potential technological interest, the research described here puts into perspective the possibility of using this approach as a bottom-up assembly method for producing a range of coupled colloidal nanoparticles.

## 2. Experimental

### 2.1. Chemicals

All chemicals used in this work were used as supplied without further purification. 3-Aminopropyltriethoxysilane ( $C_9H_{23}NO_3Si$ , APTES, >99%), tetraethyl orthosilicate ( $C_8H_{20}O_4Si$ , TEOS, >99%), trisodium citrate dihydrate ( $Na_3C_6H_5O_7 \cdot 2H_2O$ , 99%), silver nitrate ( $AgNO_3$ , 99%), potassium nitrate ( $KNO_3$ , >99%), gold(III) chloride trihydrate ( $HAuCl_4 \cdot 3H_2O$ , 99.9%), potassium tetrachloropalladate (II) ( $K_2PdCl_4 \cdot H_2O$ , 99.9%) and hydrogen tetrachloroplatinate ( $H_2PtCl_6$ , 99.9%) were purchased from Sigma-Aldrich. Ferrous sulfate heptahydrate ( $FeSO_4 \cdot 7H_2O$ , >99%), citric acid monohydrate ( $C_6H_8O_7 \cdot H_2O$ , 99.5%), carbon disulfide ( $CS_2$ , 99.9%) and ethanol ( $C_2H_6O$ , >99%) were obtained from Panreac. Ascorbic acid ( $C_6H_8O_6$ , 99.7%), ammonia solution (25%  $NH_3$ ) and sodium borohydride ( $NaBH_4$ , 95%) were purchased from Riedel-de-Haën. Sodium hydroxide ( $NaOH$ , >98%) and potassium hydroxide ( $KOH$ , >98%) were obtained from Pronolab.



**Fig. 1.** TEM images of colloidal metal nanoparticles. (A) Au (dav = 4.5 ± 0.1 nm); (B) Ag (dav = 4.7 ± 0.1 nm); (C) Pd (dav = 2.3 ± 0.1 nm); (D) Pt (dav = 2.1 ± 0.1 nm).

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