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Iron oxide nanoparticles prepared by laser ablation: Synthesis, structural properties and antimicrobial activity

Enza Fazio[a,*], Marco Santoro[a], Germana Lentini[b], Domenico Franco[b], Salvatore Pietro Paolo Guglielmino[b], Fortunato Neri[a]

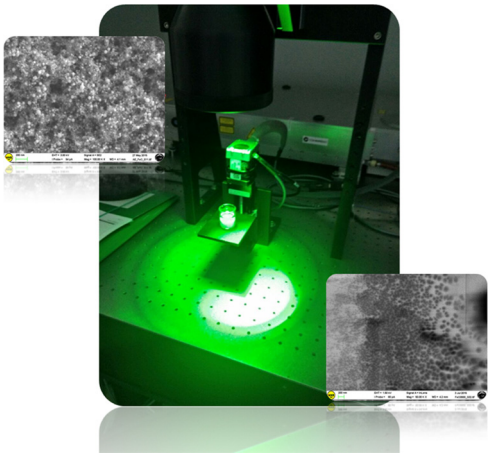
[a] Dipartimento di Scienze Matematiche e Informatiche, Scienze Fisiche e Scienze della Terra, Università di Messina, Viale Ferdinando Stagno d'Alcontres 31, 98166 Messina, Italy

[b] Dipartimento di Scienze Chimiche, Biologiche, Farmaceutiche ed Ambientali, Università di Messina, Viale Ferdinando Stagno d'Alcontres 31, 98166 Messina, Italy

HIGHLIGHTS

- Iron oxide nanoparticles embedded in a polymeric matrix.
- Iron oxide nanoparticles synthesized by a high-power picosecond laser.
- Effects of PVA on Fe_2O_3 nanoparticles distribution and dimensionality.
- Fe_2O_3 nanoparticles toxicity on *Staphylococcus aureus*.
- Fe_2O_3 nanoparticles as vectors for drug targeting platforms.
- Antimicrobial activity by MTT assay.

GRAPHICAL ABSTRACT



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ABSTRACT

Pulsed laser ablation of iron rod target in water-based solutions were carried out, varying the ablation parameters. The structural, morphological and compositional characteristics of the nanoparticles were studied by micro-Raman, dynamic light scattering (DLS), X-ray photoelectron (XPS) spectroscopies and electron scanning microscopy (SEM/STEM). Slight changes in the ablation parameters result in significant variations in the nanoparticles morphology. As observed by STEM imaging, particles size and distribution was tuned from agglomerated to nearly spherical structures, mainly changing the medium (water or polyvinyl alcohol PVA water solution). On the other hand, the polymeric phase increases the iron oxide nanoparticles stability, biocompatibility and interactive functions on the surface. Antimicrobial activity of iron oxide nanoparticles on *Staphylococcus aureus* was studied by means of MTT assay. The results indicate that the iron oxide nanoparticles are interesting for potential applications as vector for drug delivery and as constituent of specific platforms for drug targeting.

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1. Introduction

In the rapidly emerging field of nanopiotechnology, metal nanoparticles (MNPs) are extensively used in drug delivery [1],

* Corresponding author. Fax: +39 090 676 5458.
E-mail address: enfazio@unime.it (E. Fazio).

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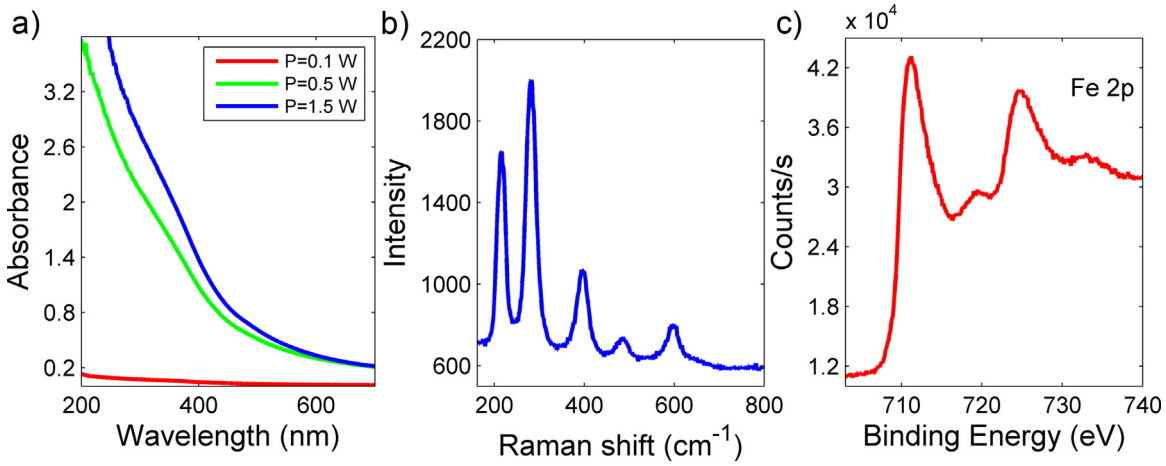

Fig. 1. (a) Optical absorption spectra of the iron oxide nanocolloids prepared in water, varying the laser power; (b) Raman spectrum and (c) Ag 3d XPS photoemission spectra of the sample prepared in water, at the laser power of 0.5 W, for an ablation time of 2 min.

biosensors [2], bio-imaging [3], antimicrobial activities [4] and food reservation [5], by exploiting their unique physical chemical and biological properties. Their nanoscale size, three-dimensional structure, large surface area and negligible side effects make them highly effective for biomedical applications such as molecular imaging [3] and cancer therapy [6]. Nontoxic superparamagnetic magnetic nanoparticles with functionalized surface coatings can conjugate chemotherapeutic drugs or active molecules which can be released on a specific target manipulating the MPs by an external magnetic field. The effect of size and surface coating of magnetic nanoparticles are very important for their role as diagnostic and therapeutic agents [7]. Iron oxide has been widely used in biomedical research because of its biocompatibility and magnetic properties. Iron oxide nanoparticles, with sizes less than 100 nm, have been developed as contrast agents for magnetic resonance imaging [8] as hyperthermia agents [9] and as carriers for targeted drug delivery to treat several types of cancer [10], by using an external magnetic field to direct FeO nanoparticles to desirable sites (such as implant infection) for immediate treatment. Several studies report that iron nanoparticles exhibit antibacterial activity in dependence of their size, concentration and oxidation state [11]. Antimicrobial activity of the nanoparticles is known to be a function of the surface area in contact with the microorganisms. The ions released by the nanoparticles may attach to the negatively charged bacterial cell wall and rupture it, thereby leading to protein denaturation and cell death [12]. Xiu et al. [13] found that the anaerobic dechlorinating bacteria *Dehalococcoides* sp. was sensitive to nanoscale zero valent iron exposure when they studied the bioremediation of trichloroethylene, using a mixture of bacterial species. Increasing the Fe nanoparticles concentration substantially inhibited the growth of *Escherichia coli* and *Staphilococcus aureus* [14]. Recent attention has been turned to the development of synthetic procedures that are environmentally friendly in order to minimize chemical waste as well as potential safety issues associated. On the other hand, the attention is focused to fully understand the interaction mechanisms between iron oxide nanoparticles and living systems.

The present study is focused on the synthesis of iron oxide nanocolloids by means of a picoseconds pulsed laser ablation technique. This latter is a fast and clean technique which allowed the formation of chemically and morphologically stable Fe_2O_3 nanocolloids with a narrow size distribution. Our technological challenge was been to optimize the ablation process parameters that make the Fe_2O_3 nanostructures stable, both at rest and under high light/magnetic flux, with a narrow size distribution and to assess their antibacterial activity.

2. Material and methods

Pulsed laser ablation of high purity (99.9%) iron rod target in pure water (H_2O) and in PVA water solution has been carried out using the second harmonic (532 nm) of a laser operating at 100 kHz repetition rate with a pulse width of 6–8 ps. The target was irradiated for typical laser power of 0.1 0.5 and 1.5 W and an irradiation time of 16 min in water. The ablation processes in the PVA water solution were carried out also with steps of 1 min for a total time of ablation of 16 min, by spending 10 min between an ablative process and the other one. The PVA water solution has so been obtained: 7.5 gr of PVA (PM = 86,000) are dissolved in 30 mL of distilled water. The dispersion was heated up to the temperature of 90 °C and, subsequently, left to reflux under magnetic agitation for 2 h; then, cooled down to room temperature. The dispersion was further diluted in water to obtain a 7.5% P/V final solution. The iron oxide content was estimated to be about 0.2% P/V.

The micro-Raman responses of the materials were investigated after the deposition of some drop of the water colloids on a CaF_2 substrates. Raman spectra were excited by the 638 nm radiation of a 30 mW diode laser, for an integration time of 80 s. The backscattered radiation, collected by an Olympus BX 40 microscope optics using a 50X objective lens, was analyzed by an XploRA 1800 cm^{-1} monochromator equipped with a Peltier CCD sensor. The optical transmission of the iron oxide colloids was analyzed by means of a PerkinElmer Lambda 750 UV–vis spectrometer in the 190–1100 nm range, using quartz cells. The size of the nanoparticles was determined by dynamic light scattering (DLS) measurements using a Horiba NanoParticle Analyzer SZ-100 (range: 0.3 nm–8 μm). Using the same Horiba NanoParticle Analyzer SZ-100, the Zeta potential was quantified with a laser Doppler method, based on the principle of electrophoretic mobility under an electric field. A fraction of the same colloids was deposited on carbon substrates to carry out Scanning Electron Microscopy (SEM) characterization. SEM images were taken by a scanning electron microscope (Merlin; model ZEISS-Gemini 2) operating at an accelerating voltage of 30 kV and at a working distance of 4 mm when the measure was carried out in transmission mode (STEM) and at an accelerating voltage of 3 kV for the collection of the SEM images. X-ray photoelectron spectroscopy (XPS) spectra were acquired using a K-Alpha system of Thermo Scientific, equipped with a monochromatic Al-Kα source (1486.6 eV) and operating in constant analyzer energy (CAE) mode

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