Electronic Supplementary Material (ESI) for ChemComm.
This journal is © The Royal Society of Chemistry 2017

Visible light-driven, magnetically steerable gold/ iron oxide nanomotors

Dekai Zhou,[ab]‡ Liqiang Ren,[d]‡ Yuguang C. Li,[b] Pengtao Xu,[b] Yuan Gao,[a] Guangyu Zhang[a], Wei Wang,*[c] Thomas E. Mallouk,*[b] and Longqiu Li*[a]

a. Key Laboratory of Microsystems and Microstructures Manufacturing, Harbin Institute of Technology, Harbin 150001, P.R. China
b. Department of Chemistry, Biochemistry and Molecular Biology and Physics, The Pennsylvania State University, University Park, Pennsylvania 16802, United States
c. School of Materials Science and Engineering, Harbin Institute of Technology, Shenzhen Graduate School, Shenzhen, Guangdong 518055, China.
d. Department of Engineering Science and Mechanics, The Pennsylvania State University, University Park, Pennsylvania 16802, United States
Corresponding authors: weiwangsz@hit.edu.cn, tem5@psu.edu, longqiuli@hit.edu.cn .
‡These authors contributed equally to this work.

SUPPORTING INFORMATION (2 pp.)

Supporting videos

Video S-1: A gold/iron oxide nanomotor moving in different concentrations of H_2O_2 (0.005 v%, 0.01 v%, 0.05 v%, 0.1 v%, 0.5 v%, 1 v%, 2.5 v% and 5 v%), light intensity 33mW/mm^2.

Video S-2: A gold/iron oxide nanomotor moving in 2.5 v% H_2O_2 under illumination with different light intensities (3.0 mW/mm^2, 4.2 mW/mm^2, 5.4 mW/mm^2, 18.1 mW/mm^2, 26.8 mW/mm^2, 33 mW/mm^2)

Video S-3: Light triggered "on/off" control of a gold/iron oxide nanomotor in 2.5 v% H_2O_2, light intensity 33 mW/mm^2.

Video S-4: Magnetic control of a gold/iron oxide nanomotor in 2.5 v% H_2O_2, light intensity 33 mW/mm^2

Supporting Figures

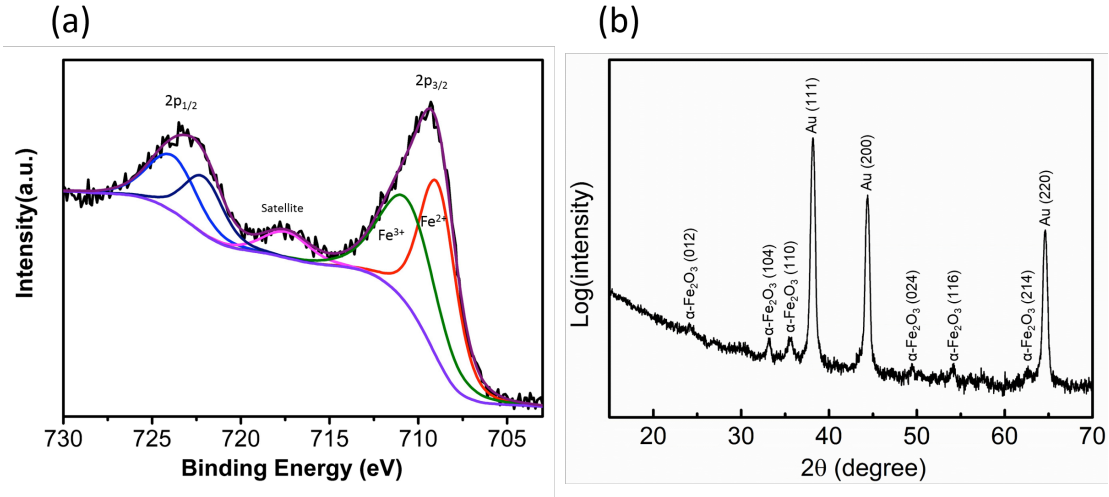

Fig. S-1: (a) XPS showing the presence of ferrous (Fe^{3+}) and ferric (Fe^{2+}) oxidation states. (b) XRD pattern of the nanorods (CuK_α, λ=1.5418 Å), with intensity plotted on a logarithmic scale to show both the Au and hematite reflections. Miller indices are shown in parentheses for the face-centered cubic Au and hexagonal hematite (α-Fe_2O_3, JCPDS number 33-0664) phases. The two most intense reflections of magnetite (Fe_3O_4), the (311) and (440), overlap with the (110) and (214) reflections of hematite, respectively, so we cannot eliminate the possible presence of Fe_3O_4 in the iron oxide segments. From the width of the (104) reflection we estimate that the size of the hematite crystal grains is on the order of ~30 nm.

Materials and Methods

Sample preparation

Gold/iron oxide nanorods were synthesized by previously reported methods. Anodic alumina membranes (AAO, purchased from Whatman Inc., 200 nm nominal pore size) were used as the template for the electrodeposition of metals. A 200 nm thick silver layer was first evaporated on the back side of the AAO membrane by a Kurt Lesker Lab 18 electron-beam evaporator and served as the working electrode. For the deposition of silver and gold, a two-electrode system was used with a Pt coil serving as both the counter electrode and the reference electrode. For the deposition of Fe, a three-electrode system was employed, with an Ag/AgCl electrode as the reference electrode, a Pt coil as the counter electrode and the evaporated silver layer as the working electrode. In a typical procedure, the sacrificial Ag segment was first electrodeposited for 25 min into the AAO membrane at a current of -1.6 mA/cm^2. Then, gold was deposited for 15 min at a current density of 0.96 mA/cm^2. Gold and silver plating solutions were purchased from Technic Inc. Afterwards, Fe was electrodeposited inside the AAO membrane from a plating solution prepared by mixing 6 g of ferrous sulfate, 0.15 g of ascorbic acid, 0.05 g of amidosulfonic acid and 1.5 g of boric acid in 100 mL of DI water. A constant potential of -0.5 V vs. the Ag/AgCl reference electrode was applied for 30 min. After the electrodeposition, the silver layer was dissolved in 8 M HNO_3 solution and the membrane was washed with deionized water. Then the AAO membrane with gold/iron nanorods was thermally annealed in an oxygen atmosphere for 10 h at 500 ℃ to convert iron into iron oxide. Finally, the AAO membrane was removed with 3 M NaOH solution for 1 h and rinsed with deionized water.

Materials characterization

The morphology and elemental composition of the gold/iron oxide nanorods were characterized by using a FEI NanoSEM 630 scanning electron microscope and by energy dispersive X-ray spectroscopy. X-ray powder diffraction patterns of the gold/iron oxide nanorods were recorded on a Philips Empyrean instrument using Cu Kα radiation. The surface chemical composition of iron oxide nanorods was measured on a Si wafer by Kratos Analytical Axis Ultra photoelectron spectrometer. UV-vis diffuse-reflectance spectra of iron oxide nanorods on glass slides were measured by using a Perkin-Elmer Lambda 950 spectrophotometer. The magnetic properties of nanowires were measured by a SOLID magnetometer from Quantum Design, Inc. The zeta potential of the microrods was measured using a Malvern Instruments Zen 3600 Zetasizer.

Video recording and Tracking

The motion of gold/iron oxide nanomotors were observed by an Olympus BX60 M optical microscope and recorded by a video capture device (Dazzle Video Creator Plus). Videos of the gold/iron oxide nanomotors were captured at 30 frames per second. The motion of the gold/iron oxide nanomotors was analyzed by PhysMo software (PhysMo - Video Motion Analysis Package, http://physmo.sf.net). All speeds were calculated by averaging the speeds of at least 10 different micromotors.

Electrochemical measurements

Tafel plots of iron oxide electrodes or gold electrodes were obtained by current-voltage scans in a two-electrode system against an Ag/AgCl reference electrode in the dark and under illumination. The iron oxide electrode was made by the same procedure as the iron oxide nanorods. First, a Fe layer was electrodeposited onto a FTO-coated glass slide for 30 min at -0.5 V vs. the Ag/AgCl reference electrode. Then the Fe electrode was thermally annealed in an oxygen atmosphere for 10 h at 500 ℃ to form the iron oxide electrode. The gold electrode was made by two-electrode deposition at 0.96 mA/cm^2 for 15 min. An Ag/AgCl electrode was used as both the counter and the reference electrode in the measurement since the passed current was very small. 2.5 % hydrogen peroxide solution was used as the electrolyte. In order to measure the J-V curve, a gold/iron oxide solid junction was made. First, iron oxide was fabricated on the FTO glass by the method described above. Then 100 nm of gold was sputtered onto the iron oxide surface by using a 1150R Rotary-Pumped Sputter Coater to form the gold/iron oxide solid junction electrode. The scan rate of the potential was 20 mV/s. A 100 mW/cm^2 Xe lamp and NuVant EZStat potentiostat were used in all electrochemical experiments.