Research Article

Microwave-Assisted Synthesis
Core-Fe$_3$O$_4$ Shell-Au Cubic Nanoparticles

Manuel Ramos,$^{1,2}$ Karina Castillo,$^1$ Domingo A. Ferrer,$^3$ Rurik J. Farias,$^2$
Sergio Flores,$^2$ and Russell R. Chianelli$^1$

$^1$Materials Research and Technology Institute, UT-El Paso, El Paso, TX 79968, USA
$^2$Departamento de Física y Matemáticas, Universidad Autónoma de Ciudad Juárez, Ciudad Juárez, MX 32300, USA
$^3$Microelectronics Research Center, UT-Austin, Austin, TX 78751, USA

Correspondence should be addressed to Manuel Ramos, manuel.ramos@uacj.mx

Received 18 July 2011; Accepted 17 August 2011

Academic Editors: I.-C. Chen and J. Das

Copyright © 2011 Manuel Ramos et al. This is an open access article distributed under the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

Core-Shell (Fe$_3$O$_4$/Au) nanoparticles were synthesized using iron II chloride tetrahydrate (FeCl$_2$$\cdot$H$_2$O) and potassium tetrachloroaurate III (AuCl$_4$$\cdot$K) precursors under microwave-assisted conditions. Products were analyzed using field emission gun electron microscope in transmission and scanning modes; energy dispersive X-ray spectroscopy performed during STEM measurements indicated a signal for gold K and M signals at 9 keV and 13 keV, respectively, confirming Au atoms at nanoparticle’s perimeter and Fe-L signal at 8 keV to be at the center.

1. Introduction

Chemical synthesis, fabrication, and applications of nanoparticles have been an evolving topic in the material science of advanced materials; this is attributed mainly to their specific electronic properties, which in many cases differ from when they are present in bulk, making them strong chemical entities as antibacterial [1], solid-state electronics [2], catalytic reactions [3], optical physics, and petroleum research [4]. In particular, magnetic nanoparticles have attracted a special interest for two main reasons: (1) implementation as contrast agents for magnetic resonance imaging (MRI) [5], (2) magnetic material for data storage in solid-state electronics (SSE) [6]. The achievement of standardized shape and high-quality nanoparticles properties will depend solely on synthesis-fabrication method which is dependable on appropriate precursor solutions ratios and in some occasions a reductant agent [7–11]. Optical properties can be tuned by controlling the coating thickness; previous studies indicate the possibility to tune surface plasmonic properties of Fe$_3$O$_4$/Au/Ag from $\lambda = 560$ nm (red shift) to $\lambda = 501$ nm (blue shift) with the addition of nonmagnetic layers (Au or Ag); however, it will reduce magnetic strength of (Fe$_3$O$_4$) nanoparticles [12]. Other authors achieved spindle-shaped hematite (Fe$_2$O$_3$) using a hydrothermal method of synthesis, and particle shapes depend only on 3-aminopropyl trimethoxysilane (APTMS) which acted as a reduction agent in generating amine moiety-coated surface [13, 14]. This paper presents a microwave-assisted synthesis of cubic core-shell Fe$_3$O$_4$/Au nanoparticles along with atom-resolved scanning transmission electron microscopy and energy dispersive X-ray spectroscopy profiles.

2. Synthesis of AuFe$_3$O$_4$ Cubic Nanoparticles

To avoid any contaminant variations on the results, before any chemical reaction, all glassware was cleaned using aqua regia in a concentration ratio of HCl/HNO$_3$ = 3:1. The synthesis consisted of two main steps. (1) Synthesis of Fe$_3$O$_4$ by using iron II chloride tetrahydrate (FeCl$_2$$\cdot$H$_2$O Alfa Aesar) by dissolving 70 mg in distilled water and slowly titrated for 4 h with 40 mL of 5 M NaOH solution to form iron II hydroxide (Fe(OH)$_2$). The iron II hydroxide solution was oxidized to form Fe$_3$O$_4$ using microwave-assisted synthesis (Multiwave 2000) at a constant temperature of 120°C for 30
3. Results and Discussion

3.1. Scanning Transmission Electron and Energy Disperse X-ray. Morphology of products (AuFe₃O₄) was studied by high-resolution transmission electron microscopy using an FEI Tecnai TF20 equipped with an STEM unit, high-angle annular dark-field (HAADF) detector, and X-Twin lenses. Just one drop of AuFe₃O₄/isopropanol solution was placed into a lacey/carbon (EMS LC225-Cu) grid. The operational voltage was kept constant at 200 kV in both dark field (DF) and bright field (BF) mode images. Scherzer defocus condition was set at \( \Delta f_{\text{sch}} = -1.2(C_{\lambda} \lambda)^{1/2} \). Energy disperse X-ray analysis (EDX) was performed using a solid angle of 0.13 sr in the detector. Cubic structure is observed by HRTEM as presented in Figure 1; in order to locate the gold on nanoparticles surface, a profile was created using EDX while performing STEM as presented in Figures 2 and 3;
Figure 3: Energy disperse X-ray showing K and L signals corresponding to Fe when probe is at center and STEM image.

clearly K and M signals at 9 to 13 keV indicate the presence of gold at perimeter, while Fe-L signal at 8 keV appears when probing nanoparticle center.

4. Conclusions

A successful synthesis of cubic-shaped core-shell Fe₃O₄/Au nanoparticles was achieved using microwave-assisted synthesis. STEM and HRTEM confirm cubic shape. Energy disperse X-ray analysis profiles indicate peak intensities from 9 to 13 keV for gold at the perimeter and 8 keV for iron at the center also confirming a core-shell array.

Acknowledgment

The authors thank The National Nanotechnology Infrastructure Network (NNIN) Research Program of the Microelectronic Research Center of UT-Austin, the Consejo Nacional de Ciencia y Tecnología, México for their economic support, and the Materials Research and Technology Institute of UT-El Paso.

References

[1] L. Liu, K. Xu, H. Wang et al., “Self-assembled cationic peptide nanoparticles as an efficient antimicrobial agent,” Nature Nanotechnology, vol. 4, no. 7, pp. 457–463, 2009.
[2] D. Ghosh and S. Chen, “Solid-state electronic conductivity of ruthenium nanoparticles passivated by metal-carbon covalent bonds,” Chemical Physics Letters, vol. 465, no. 1–3, pp. 115–119, 2008.
[3] Y. Mei, G. Sharma, Y. Lu et al., “High catalytic activity of platinum nanoparticles immobilized on spherical polyelectrolyte brushes,” Langmuir, vol. 21, no. 26, pp. 12229–12234, 2005.
[4] T. Otake, D. J. Wesolowski, L. M. Anowitz, L. F. Allard, and H. Ohmoto, “Experimental evidence for non-redox transformations between magnetite and hematite under H₂-rich hydrothermal conditions,” Earth and Planetary Science Letters, vol. 257, no. 1–2, pp. 60–70, 2007.
[5] M. V. Yigit, L. Zhu, M. A. Ilediba et al., “Noninvasive MRI-SERS imaging in living mice using an innately bimodal nanomaterial,” ACS Nano, vol. 5, no. 2, pp. 1056–1066, 2011.
[6] T. Hyeon, “Chemical synthesis of magnetic nanoparticles,” Chemical Communications, vol. 9, no. 8, pp. 927–934, 2003.
[7] D. Ferrer, A. Torres-Castro, X. Gao, S. Sepúlveda-Guzmán, U. Ortiz-Méndez, and M. José-Yacamán, “Three-layer core/shell structure in Au-Pd bimetallic nanoparticles,” Nano Letters, vol. 7, no. 6, pp. 1701–1705, 2007.
[8] R. Nagarajan et al., Nanoparticles: Synthesis, Stabilization, Passivation, and Functionalization, ACS Symposium Series, Washington, DC, USA, 2008.
[9] M. Spuch-Calvar, J. Pérez-Juste, and L. M. Liz-Marzán, “Hematite spindles with optical functionalities: growth of gold nanoshells and assembly of gold nanorods,” Journal of Colloid and Interface Science, vol. 310, no. 1, pp. 297–301, 2007.
[10] Y. Zhang, E. W. Shi, Z. Z. Chen, and B. Xiao, “Fabrication of ZnO hollow nanospheres and “jingle bell” shaped nanospheres,” Materials Letters, vol. 62, no. 8-9, pp. 1435–1437, 2008.
[11] Y. Sun, B. T. Mayers, and Y. Xia, “Template-engaged replacement reaction: a one-step approach to the large-scale synthesis of metal nanostructures with hollow interiors,” Nano Letters, vol. 2, no. 5, pp. 481–485, 2002.
[12] Z. Xu, Y. Hou, and S. Sun, “Magnetic core/shell Fe₃O₄/Au and Fe₃O₄/Au/Ag nanoparticles with tunable plasmonic properties,” Journal of the ACS, vol. 129, no. 28, pp. 8698–8699, 2007.
[13] J. Lin, W. Zhou, A. Kumbhar et al., “Gold-coated iron (Fe@Au) nanoparticles: synthesis, characterization, and magnetic field-induced self-assembly,” Journal of Solid State Chemistry, vol. 159, no. 1, pp. 26–31, 2001.
[14] Z. Ma, H. Han, S. Tu, and J. Xue, “Fabrication of shape-controlled hematite particles and growth of gold nanoshells,” Colloids and Surfaces A: Physicochemical and Engineering Aspects, vol. 334, no. 1–3, pp. 142–146, 2009.
Submit your manuscripts at http://www.hindawi.com