

Supporting Information:

Preparation of magnetic mesoporous core-shell nanocomposite for cinnamic acid hydrogenation

Xiaofang Liu, Lingjuan Shi, Wenhui Feng, Libo Niu, Chen Liu, Guoyi Bai*

Key Laboratory of Medicinal Chemistry and Molecular Diagnosis of Ministry of Education, College of Chemistry and Environmental Science, Hebei University, Baoding 071002, P.R. China

Experimental:

Preparation of Fe₃O₄@nSiO₂@mSiO₂ Nanocomposite

Fe₃O₄@nSiO₂@mSiO₂ was prepared by a modified Stöber sol-gel process with tetraethyl orthosilicate (TEOS) as the silica source,¹ as described below.

0.7 g of Fe₃O₄ nanoparticles (NPs) was first treated with 0.2 M HCl solution (100 ml) under ultrasound for 10 min to enhance the surface charge density,² followed by separation with magnet and washing with absolute ethanol for several times.

A dense and thin silica layer was then deposited onto the surface of Fe₃O₄ NPs in order to protect the iron oxide core from leaching and prevent its aggregation.³ In brief, the pretreated Fe₃O₄ NPs were first dispersed in 35 ml deionized water under ultrasound for 10 min. Then, an absolute ethanol solution containing 2.5 mL 25 wt% aqueous ammonia was added to the above magnetite suspension under ultrasound within 20 min. After that, 1 ml TEOS was slowly introduced into this mixture. After being stirred at 303 K for 5 h, the obtained Fe₃O₄@nSiO₂ NPs were separated and washed with absolute ethanol for several times.

Then, the obtained Fe₃O₄@nSiO₂ NPs was dispersed in a mixed solution containing 0.75 g of cetyltrimethylammonium bromide (CTAB), 200 mL of deionized water, 2.5 ml of 25 wt% aqueous ammonia, and 150 mL of absolute ethanol and kept under ultrasound for 30 min. Subsequently, 1

ml of TEOS was added dropwise to the solution with vigorous stirring. After being stirred for another 5 h, the obtained $\text{Fe}_3\text{O}_4@\text{nSiO}_2@\text{CTAB}/\text{SiO}_2$ was separated with magnet, washed with absolute ethanol for several times and allowed to age in absolute ethanol (100 ml) at room temperature overnight.⁴ After that, the as-synthesized $\text{Fe}_3\text{O}_4@\text{nSiO}_2@\text{CTAB}/\text{SiO}_2$ was transferred to 200 mL absolute ethanol containing 1.2 g of NH_4NO_3 and mechanically agitated vigorously at 333 K for 2.5 h. The extraction step was repeated twice to completely remove CTAB.⁵ Finally, the $\text{Fe}_3\text{O}_4@\text{nSiO}_2@m\text{SiO}_2$ thus obtained was washed with deionized water and absolute ethanol several times, collected by magnet, dried at 313 K, and kept for further use.

References

- 1 Y. H. Deng, D. W. Qi, C. H. Deng, X. M. Zhang and D. Y. Zhao, *J. Am. Chem. Soc.*, 2008, **130**, 28-29.
- 2 M. H. Chen, Y. N. Kim, C. C. Li and S. O. Cho, *J. Phys. Chem. C*, 2008, **112**, 6710-6716.
- 3 W. R. Zhao, J. L. Gu, L. X. Zhang, H. R. Chen and J. L. Shi, *J. Am. Chem. Soc.*, 2005, **127**, 8916-8917.
- 4 Y. G. Zhan, B. X. Cai, B. Wang, X. N. Huang, P. Q. Zhang, L. L. Li, Z. J. Wu, Z. L. Yin and Q. Y. Chen, *J. Mater. Chem.*, 2008, **18**, 5967-5973.
- 5 Y. S. Lin and C. L. Haynes, *Chem. Mater.*, 2009, **21**, 3979-3986.