PREPARATION OF IRON OXIDE NANOPARTICLE FROM $FeCl_3$ SOLID POWDER USING MICROEMULSIONS

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Nanoparticles are small atomic clusters, less than 100 nm, with size dependent properties. Due to their small size and large surface area, nanoparticles exhibit novel properties which differ from those of bulk materials. Nanoparticles find application in: modern material (1), microelectronics (2), catalysis (3), and chemical and biochemical sensors (4). There are several techniques for nanoparticle preparation, including: sol-gel processing; forced hydrolysis; hydrothermal synthesis, electrochemical methods; and microemulsion techniques.

The use of w/o microemulsions for nanoparticle preparation has attracted a lot of attention for its: simplicity, the control it provides over the particle size, and the highly homogenous product it gives (5,6). This work presents a novel approach for the preparation of iron oxide nanoparticles in w/o microemulsions. This approach involves subjecting $FeCl_3$ bulk solid powder to the action of microemulsions formed with sodium bis(2-ethylhexyl) sulfosuccinate (AOT), an anionic surfactant. Iron chloride first solubilizes into the water pools of the microemulsions, then reacts with sodium hydroxide added to the water pools of the microemulsion to eventually form the iron oxide nanoparticles. This technique serves as an in-situ preparation of nanoparticle catalysts which find applications in the in-situ heavy oil upgrading.

The effect of the following factors on the nanoparticle formation is investigated: (i) operating variables; including mixing speed, mixing time, and temperature; (ii) microemulsion variables; including the surfactant concentration, the amount of NaOH, and the amount of $FeCl_3$ powder. The nanoparticle morphology and size distribution is determined using the Small Angel X-Ray Scattering, Transmission Electron Microscopy (TEM), and UV-spectroscopy.

Results and discussion

Effect of the amount of $FeCl_3$ powder: Increasing the amount of $FeCl_3$ powder resulted in the collapse of the microemulsion into 2 layers. The size of the new layer was proportional to the amount of $FeCl_3$ powder. It is expected the collapse of the microemulsion resulted from the increase in the ionic strength inside the water pools in addition to the migration of the surfactant to the excess $FeCl_3$ phase. Figure 1 shows that the UV-absorbance of the colloidal iron oxide nanoparticles increases with decreasing the amount of $FeCl_3$ powder. This increase in the size of the absorption spectra is associated with an increase in the nanoparticle size as well as the nanoparticle concentration (8,9).

Effect of NaOH: The UV results depicted in Figure 2 suggest that as the amount of NaOH increases the concentration of iron oxide nanoparticles increases. Figure 3 shows TEM image for iron oxide nanoparticles collected at NaOH concentration of 0.5 N in the water pool.

References

1. Loscertales, I. G.; Barrero, A.; Guerrero, I; Cortijo, R.; Marquez, M.; Ganan-Calvo, A. M. *Science* 295, 1695 (2002)

2. M. Pileni, in: J. H. Fendler (Ed.), *Nanopartilces and Nanostructured Films*, Wiley-VCH, New York, 1998, p 53 and 71
3. Ohde, H.; Wai, C. M.; Kim, H.; Kim, J.; Ohde, M. *J. Am. Chem. Soc*. 124, 4540 (2002)
4. A. R. Varlan, J. Sules, P Jacobs, W. Sansen, *Biosens. Bioelectron.* 10, 15-19 (1995)
5. V. Pillai, P. Kumar, M. Hou, P. Ayyub, D. Shah, *Adv. Colloid Interface Sci.* 55, 241 (1995)
6. M. Husein, E. Rodil, J. H., Vera, J. *Colloid Interface Sci*. (2005) *in press*
7. Yaacob I. I.; Nunest A. C.; Bose A.; and Shah D. O., J. *Colloid Interface Sci*. 168, 289-301 (1994)
8. M. Husein, E. Rodil, J. H., Vera, *Langmuir* 19, 8467 (2003)
9. M. Husein, E. Rodil, J. H., Vera, J. *Colloid Interface Sci*. 273, 426 (2004)

Figures

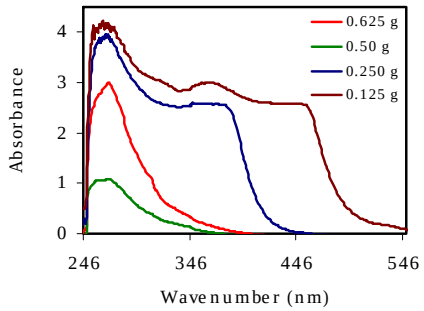

Figure 1: Adsorption spectrum of iron oxide nanopartilces prepared by mixing a certain amount of $FeCl_3$ with AOT microemulsion. 0.2 M AOT, R = 2.12, sample volume was 10 ml.

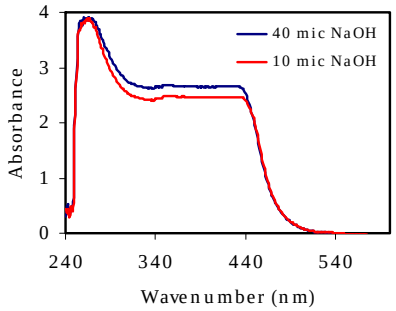

Figure 2: Adsorption spectrum of iron oxide nanopartilces prepared by mixing 0.1 g of $FeCl_3$ with AOT microemulsion. 0.2 M AOT, R = 2.12, sample volume was 20 ml.

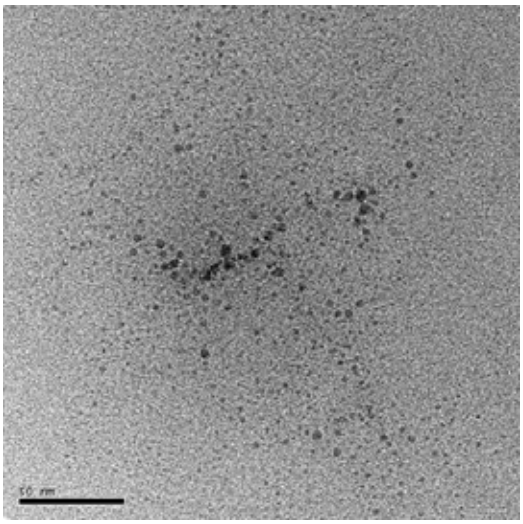

Figure 3: TEM micrographs of iron oxide nanopartilces prepared by mixing 0.1 g of $FeCl_3$ with AOT microemulsion: 0.2 M AOT, R = 2.12, volume= 20 ml. T= 25^{o}C