doi: 10.1017/S1431927609099115
Microsc Microanal 15(Suppl 2), 2009

Sol Gel Processing of Iron(III) Oxides Decorated Carbon Nanotubes

E. L. Camponeschi, J. W. Walker, H. Garmestani, and R. Tannenbaum,*

* Department of Materials Science and Engineering, Georgia Institute of Technology School of Engineering, Atlanta, GA 30332

Introduction The design, development, manufacturing and utilization of nanomaterials have become very broad and vigorously-growing fields of research in recent years.[1-4] Nanomaterials represent a new class of materials that possess distinctive physical and chemical properties that differ substantially from their micron-size and bulk counterparts.[1-4] In order to harvest and translate the fascinating properties of materials in the nanoscale size domain into viable, affordable new technologies, it is imperative that new, facile synthesis and manufacturing methods need to be developed. This will accelerate the creation of fundamental building blocks of the nanomaterials and their subsequent assembly into useful devices.

One such building block is Iron oxide nanoparticles, which offer much potential compatibility for a broad range of applications, such as magnetic materials, catalysts, sensors (chemical, electronic and biological), data storage materials, MRI contrast agents and drug delivery facilitators.[1-4] Hence, the design, synthesis and large scale manufacturing of various types iron oxides with tight control on particle size, size distribution and particle geometry and morphology, has become one the most important research avenues in the area of nanomaterials synthesis.

Experimental Procedure In this research, we have applied a modified sol gel processing method to create iron (III) oxide nanoparticles in a manner that would utilize the advantages of the traditional sol-gel process, but circumvent the pitfalls of network formation. Our strategy was the addition of a common surfactant, sodium dodecyl benzene sulfonate (NaDDBS), to the reaction mixture, at different stages of the reaction, in order to provide an adequate stabilization mechanism for the growing iron oxide nanoparticles, and arrest their growth and the network formation. Probing the effect of the NaDDBS molecules on the iron oxide nanonetwork formation when introduced into the reaction solution at different stages, will provide insight into the nucleation and aggregation of the fundamental particles and will offer opportunity for process optimization without gelation.

Results and Conclusions The primary particle sizes in both systems, $Fe(NO_3)_3 \cdot 9H_2O$ and $FeCl_3 \cdot 6H_2O$, have nanoscale dimensions in the gel phase regardless of the presence of NaDDBS. The addition of NaDDBS to the $FeCl_3 \cdot 6H_2O$ system in the nucleation stage, i.e. prior to gelation, delayed the gelation process and allowed extensive aggregation to occur. The addition of NaDDBS to the $FeCl_3 \cdot 6H_2O$ system both prior and during gelation, delayed the onset of gelation and resulted in small particles. Similarly, the addition of NaDDBS to the $Fe(NO_3)_3 \cdot 9H_2O$ system prevented the formation of a gel to occur, but still lead to the formation of nanosize particles in the same size range of the primary particles obtained upon gel formation.

The addition of an anionic surfactant to the iron oxide sol-gel processing method decreased the surface energy of the fundamental iron oxide particles and allowed the creation of stable nanoscale iron oxide particles without the formation of a 3D network (gel). Despite the fact that a mixture of iron oxide compounds is formed by this modified sol-gel method, it still provides an easy and economical synthesis process for the formation of nanoscale iron (III) oxide particles upon the addition of NaDDBS as a surfactant.

References

[1] J. D. Walker R. Tannenbaum, Chem. Mater. 18 (2006) 4801.

[2] Camponeschi, E. *Dispersion and Alignment of Carbon Nanotubes in Polymer Based Composites*. PhD Thesis. Georgia Institute of Technology. 2007.
[3] Camponeschi, E. *et al.*, J Non-Cryst Solids, 354(34) (2008) 4063.
[4] This work was supported by Institute of Paper Science and Technology at Georgia Tech through the Otto Kress Scholarship.

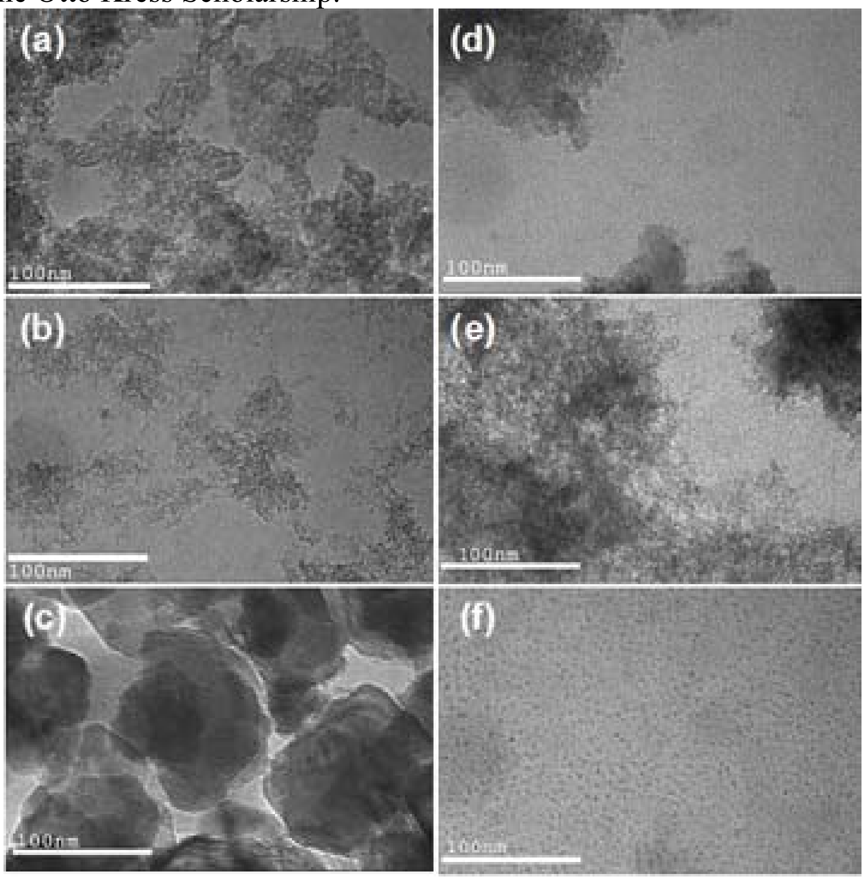

FIG. 1. High resolution transmission electron microscopy images of the iron oxide products: (a)$FeCl_3$ and NaDDBS (b)$Fe(NO_3)_3$ and NaDDBS; (c)$FeCl_3$ and NaDDBS; (d)$Fe(NO_3)_3$ and NaDDBS; (e)$FeCl_3$ without NaDDBS; (f)$Fe(NO_3)_3$ without NaDDBS

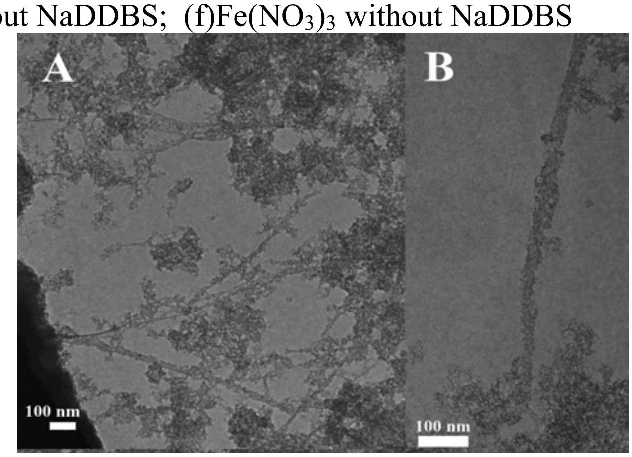

FIG. 2. Transmission electron microscopy micrographs of carbon nanotubes decorated with iron oxide particles with various sizes of CNT bundles decorated with NaDDBS-stabilized iron oxide nanoparticles: (a) 15-40 nm; (b) 45 nm;

https://doi.org/10.1017/S1431927609099115 Published online by Cambridge University Press