

## Synthesis and Application of Magnetic Nano- and Nano-composite Particles

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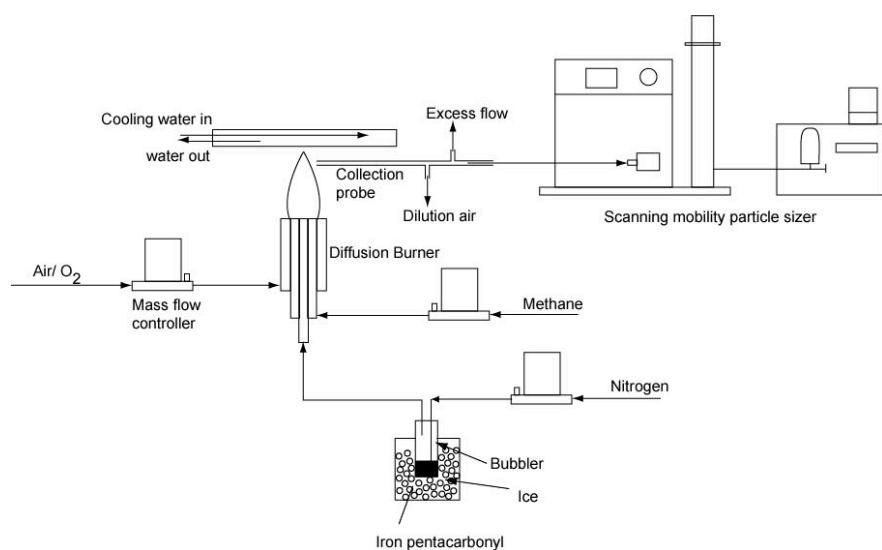
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Magnetic nanoparticles/nano-composites (ferromagnetic, ferrimagnetic and antiferromagnetic materials) have many promising industrial and biomedical applications such as catalysis, magnetocooling, optical and recording devices, purification of enzymes and other biological materials, and water purification devices; however, there are key issues that need to be carefully researched for their wider scale applicability. The first is the ability to synthesize desired magnetic nanostructures for specific applications with tailored sizes, compositions and morphologies; the second is to scale up processes for production of larger volumes of these nanomaterials; and the third is the understanding of the size dependent magnetic properties, the interaction of nanoparticles in high gradient magnetic fields, and incorporation of these dynamic interactions in predicting the transport characteristics in different media.

The overall objectives of this project are: (1) the laboratory scale synthesis of magnetic particles and nano-composites with tailored size, composition and morphologies; (2) the development of systems for on-line measurement of high concentration nanoparticles in high temperature environments; (3) the demonstration of synthesized particles for novel applications, in data storage, recording, and biomedical applications such as in embolics; (4) addressing issues of scale up for processing large volumes of such nanoparticles.

This NIRT team consists of researchers from the Departments of Chemical Engineering, Mechanical Engineering, and Electrical Engineering in Washington University in St. Louis, with expertise in nanoparticle/nano-composite synthesis processes, nanoparticle measurement and characterization, and testing for industrial and biomedical applications.

In its first year the project is focused on the synthesis of magnetic nanoparticles in flame

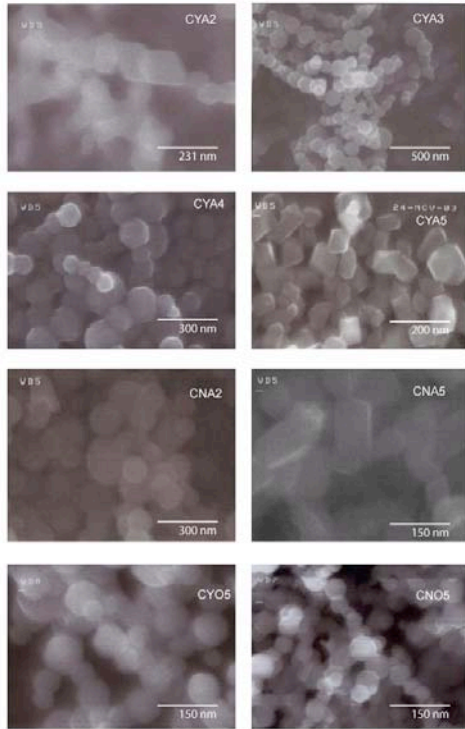


**Fig. 1 is a flame aerosol reactor in action for synthesizing gamma phase iron oxide nanoparticles in a single step process.**

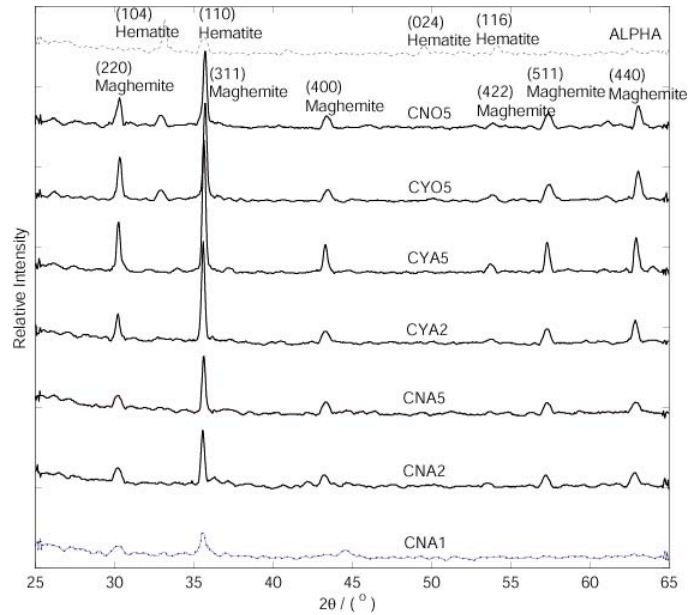
reactors and the characterization of size dependent magnetic property of nanoparticles. Most of the conventional methods for the synthesis of magnetic nanoparticles are rather complex, usually involving several steps; efforts have been made to establish direct preparation routes of these magnetic particles. In this project,  $\gamma$ -phase iron oxide nanoparticles were synthesized using a flame aerosol reactor designed by Dr. Biswas (shown in Fig. 1). The effects of flame temperature profile and sampling conditions have been investigated on the morphology and magnetic properties of synthesized

maghemite nanoparticles. The particle size and magnetic properties of the powders can be controlled by varying the flame temperature and sampling conditions. Some of the key results

are shown in Figs. 2 and 3. The TEM images of flame-synthesized  $\gamma$ -phase iron oxide nanoparticles when sampled at different flame heights are shown in Figure 2. The X-ray diffraction patterns of these samples are shown in Figure 3. The pronounced maghemite peak in these patterns confirms the synthesis of  $\gamma$ -phase iron oxide nanoparticles.



**Figure 2 shows EM images of synthesized  $\gamma$ - phase nanoparticles at different operation conditions of aerosol flame reactor**

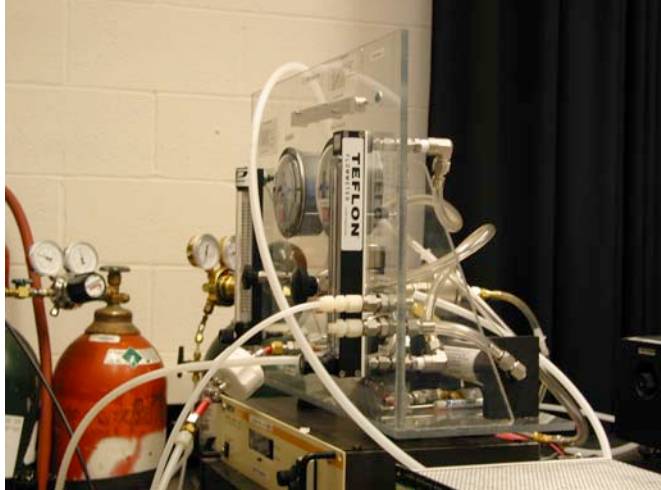


**Figure 3: X-ray diffraction patterns of iron oxide samples extracted from different flame heights and with/without quenching post-process: CNA1(amorphous); CNA2, CNA5, CYA2, CYA5 ( $\gamma$ - $\text{Fe}_2\text{O}_3$ ); CYO5, CNO5 ( $\gamma$ - $\text{Fe}_2\text{O}_3$  +  $\alpha$ - $\text{Fe}_2\text{O}_3$ ),  $\alpha$**

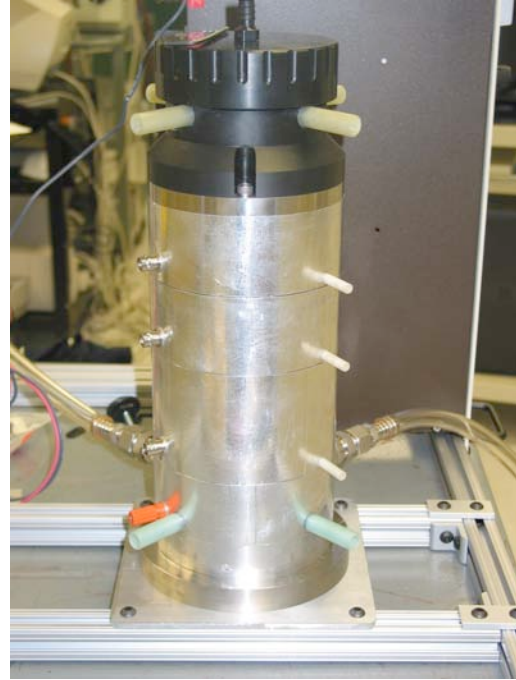
To obtain mono-sized magnetic nanoparticles for different applications, a high-throughput monodisperse particle classification system (shown in Fig. 4), capable of tailoring synthesized nanoparticles into tighter size distributions, has been designed, fabricated by Dr. Chen. The system consists of a high-throughput, high-charging-efficiency nanoparticle charger [1] and a high throughput Nano-DMA (Nanometer Differential Mobility Analyzer; [2]). The performance of this system has been evaluated prior to its integration with the flame synthesis system. Besides the development of the particle classification system, the effort has been also on the development of a multi-stage differential mobility column (in part supported by this project)[3]. This new DMA column (shown in Fig. 5) allows the extraction of mono-sized nanoparticles at different column heights simultaneously. Fig. 6 shows the width at the 50% peak of the transfer function of this multi-stage DMA column operated with the sheath flowrate of 20 lpm. As shown in the figure, the resolution of this DMA column reaches the theoretical value as the particles size increases (when the effect of particle diffusivity is negligible). As the particle size reduces the resolution deteriorates due to the increase of particle diffusivity. The diffusional resolution loss can be improved by operating the column at higher sheath flowrate.

The successful development of this multi-stage DMA facilitates the classification and testing process while reducing the waste of precious nanoparticles synthesized in the flame reactors.

Besides the experimental work, numerical modeling has also been performed to investigate the transport of nanoparticle and particle-particle interaction in the magnetic field.



**Figure 4: high-throughput monodisperse nanoparticle classification system developed by Dr. Chen. The system consists of a high-charging efficiency unipolar charger and nanometer**

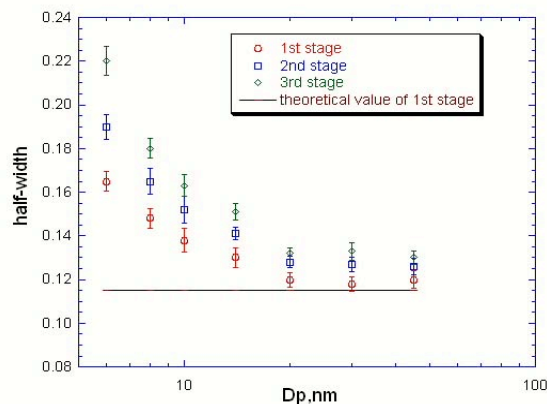


**Figure 5: newly developed multi-stage differential mobility analyzer column capable of classifying different mono-sized nanoparticles simultaneously. Shown in the figure is the 3-stage column.**

Finally, the effort of Dr. Axelbaum has been focused on the synthesis of Fe and Ta nanoparticles using the sodium flame process. Numerical modeling using FLUENT has been also performed to gain the details of the synthesis process and to provide the direction for further process improvement.

#### References:

- [1] D. CHEN and David Y. H. Pui, "A High Efficiency, High Throughput Unipolar Aerosol Charger for Nanoparticles", *J. of Nanoparticle Research*, 1, p115-126, 1999.
- [2] D. CHEN, David Y.H. Pui, D. Hummes, H. Fissan, F.R. Quant and G.J. Sem, "Design and Evaluation of a Nanometer Aerosol Differential Mobility Analyzer (Nano-DMA)," *Journal of Aerosol Science*, for the Special Issue on Nanometer Particles, 29, pp. 497-509, 1998.
- [3] Weiling Li, Da-Ren Chen, and Meng-Dawn Cheng, "Development of a Multiple-stage DMA," 23<sup>rd</sup> annual AAAR conference, Atlanta, Georgia, Oct. 4-8, 2004.
- [4] For further information on the project please email chen@me.wustl.edu.



**Figure 6: The width at 50 % peak of the transfer function of the multi-stage DMA operated at the sheath flowrate of 20 lpm.**