

Synthesis and Characterization of Nanomagnetic Materials

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Introduction

The synthesis and characterization of nanomagnetic materials is essential to the future applications of data storage and biotechnology/medicine. For biomedical applications most research is conducted on iron oxide; however, another material being investigated for potential use is cobalt.¹ My project explores the synthesis of cobalt magnetic nanoparticles as well as various characterization methods to determine the properties of these particles.

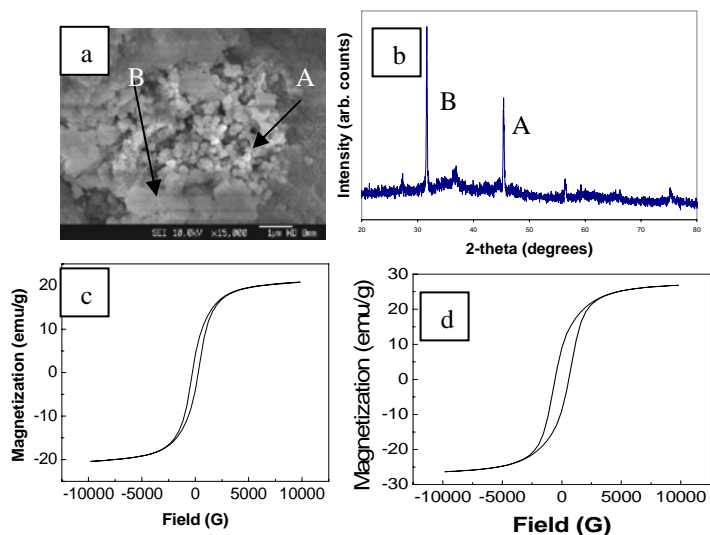
Procedure

Cobalt nanoparticles were synthesized by two methods. The first method simply involves the borohydride reduction of cobalt salts by mixture of an aqueous cobalt chloride solution and an aqueous sodium borohydride solution. The second method involves the same reduction but inside reverse micelles. Two micellar solutions were prepared, one with cobalt chloride and the other with sodium borohydride. Both solutions also contained AOT (sodium bis(2-ethylhexyl) sulfosuccinate) as the surfactant and isoctane as the oil phase. Different molar ratios of water to the surfactant ($w=[\text{H}_2\text{O}]/[\text{AOT}]$) were used for each sample to observe the effects on size of the particles. When two reverse micelles come into contact with one another, they reform into two distinct micelles through the exchange of their water content. The water cores of these reverse micelles are a few nanometers in diameter and serve as nanoreactors for aqueous reactions.^{2,3} Mixture of the two solutions causes the reaction with NaBH_4 reducing $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ to form cobalt particles. The surfactant was removed by centrifuge, washing with acetone and D.I. water, and drying in vacuum overnight. For both synthesis methods, the cobalt particles were heated to 350°C for 2 hours to introduce crystallinity in the material. Particles were also coated with platinum with an auto fine coater to ensure that the surfaces of the particles are conductive.

Results and Discussion

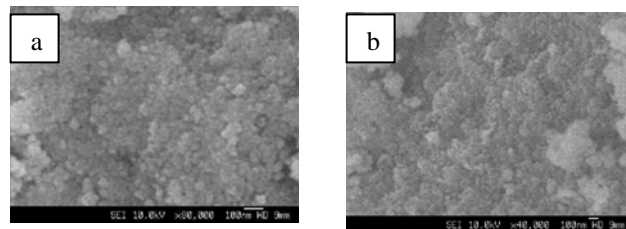
The morphology of the particles was observed through FESEM. The structural and magnetic properties were investigated by XRD and VSM. Figure 1a depicts the cobalt particles formed by borohydride reduction of cobalt salts with sizes in the range of 50 nm (A) and 100 nm (B), which indicates that the size distribution is widely spread. In figure 1b XRD shows the formation of hcp cobalt (peak A) in the sample. In addition to hcp cobalt, formation of B_2O_3 (peak B) is also observed. Boron content is from the reducing agent (NaBH_4). The magnetic properties were measured as well with saturation magnetization of 20.5 emu/g (fig 1c). The sample was then washed with DI water and the measured saturation magnetization is 26.5 emu/g (fig 1d). This shows that B_2O_3 content was considerably removed from the sample.

Figure 1 Cobalt particles formed by borohydride reduction (a) SEM (b) XRD (c) VSM curve for as synthesized sample (d) VSM curve for sample after washing with DI water.



The reverse micelle technique provides small particle sizes as well as low size distribution as seen in figure 2a and 2b. The size of the particles are controlled by the w value ($[\text{H}_2\text{O}]/[\text{AOT}]$). An increase in the w value results in an increase in particle size, evident in figure 2a and figure 2b. The particle size observed is approximately 20 nm for $w=11$ and 35 nm for $w=22$.

Figure 2 SEM images of cobalt particles formed by reverse micelles (a) $w=11$ (b) $w=22$.



Conclusions

Ferromagnetic cobalt nanoparticles can be synthesized by borohydride reduction of cobalt salts; however, the size distribution of the particles is not uniform. By applying the reverse micelle technique uniform size distribution as well as smaller cobalt particle sizes can be achieved. The size of the cobalt particles is determined by variations in the water to surfactant ratio (w value).

References

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