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Nanoparticles of Barium Ferrite Synthesised Using a Water-in-Oil Microemulsion

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Abstract. The application of barium ferrite ($\text{BaFe}_{12}\text{O}_{19}$; BaF) in the magnetic media industry requires materials with strict control of homogeneity, morphology and magnetic properties resulting from their preparation and pretreatment. Here the use of water-in-oil microemulsions for the synthesis of nanoparticles of BaF is considered. Two microemulsions (containing an aqueous phase, cetyltrimethylammonium bromide surfactant, n-butanol co-surfactant and octane) were used. The aqueous phase of the first microemulsion contained ferric/barium salts and the second co-precipitant ammonium carbonate. On mixing, the aqueous cores acted as microreactors for co-precipitation of the BaF precursor which was then separated, dried and annealed (1198K, 24h) to form BaF. It is shown that microemulsions prepared with different surfactant concentrations lead to particles with variable but controlled morphology and magnetic properties. In particular, it is shown that selecting a microemulsion with a specific concentration can allow coercivity and particle size of product to be nano-engineered.

1. Introduction

Interest in barium ferrite ($\text{BaFe}_{12}\text{O}_{19}$; BaF) has intensified, partly because of its large coercivity, which is derived from its magnetocrystalline anisotropy and its small particle size¹. Conventional methods to BaF include co-precipitation (where solutions of metallic salts and alkaline are mixed) followed by precipitate thermal annealing^{2,3}.

Water-in-oil microemulsions have been used for the synthesis of a variety of nano-particles⁴. A microemulsion may be defined as an isotropic dispersion of two immiscible liquids stabilized by an interfacial film of surface active molecules. It consists of domains, typically 5 - 10nm in diameter radius, of one liquid in the other. In a water-in-oil microemulsion, the aqueous droplets continuously collide, coalesce and break apart, resulting in a continuous exchange of solute content. Conceptually if two identical microemulsions are prepared with reactants dissolved in each aqueous phase, upon mixing and collision, the reaction takes place. Precipitation in microemulsions offers a novel technique for the synthesis of BaF, with the ability of precisely controlling the morphology of the product particles⁵. Such an adaptation could be an improvement of conventional co-precipitation methods. This was the focus of the present work.

2. Experimental

The microemulsions consisted of cationic surfactant cetyltrimethylammonium bromide ((C.T.A.B.) Avacado, 98%), co-surfactant n-butanol (BDH, 99.5%), n-octane (Avacado, 99%) and an aqueous phase (containing FeCl_3 and BaCl_2 (BDH, 99%) in a ratio required by the ferrite (12:1) or alkaline, ammonium carbonate (BDH)) in de-ionised water.

Preliminary experiments identified the region where a single phase water-in-oil microemulsion formed and results are shown in the phase diagram in Figure 1, where the single phase area is labelled (α).

Figure 1: Phase Diagram for C.T.A.B./n-butanol/octane/aqueous Microemulsion

Microemulsions of differing compositions were prepared in region α : three sets were prepared, with increasing aqueous phase weight fractions (0.10, 0.15 and 0.20; while the surfactant and hydrocarbon weight fractions were adjusted accordingly) containing Fe^{3+} (0.12M) and Ba^{2+} (0.011M) or ammonium carbonate (0.20M). These were consequently mixed for precipitation and the barium-iron-carbonate precipitate formed was separated in a centrifuge at 3000rpm for 10 min and then washed in ethanol and then de-ionised water and was finally oven dried at 373K and annealed in air at 1198K for 24h.

To avoid relatively low product yields it was desirable to increase Fe^{3+} and Ba^{2+} concentrations in the aqueous phase.

NaOH (another common co-precipitant) was also used in the microemulsions.

The BaF samples so prepared were characterised by thermogravimetric analysis (TGA, Stanton Redcroft STA780 series) in air, by transmission electron microscopy (TEM, JEOL 2000), by X-ray diffraction (XRD Philips PW1700) and by vibrating sample magnetometry (VSM, PAR 155).

3. Results

TGA suggested that the conversion of the amorphous precursor to BaF occurs above 873K, and certainly XRD showed that annealing (1198K for 24h) was successful in converting this to pure BaF. Magnetic characterisation revealed that the composition of the microemulsion affected the pigment. Thus magnetisation values (although a little lower than expected for barium ferrite ($M_s=40\text{emu/g}$)) were unaffected, but coercivity is greatly affected (see Figure 2). In (a) there is a decrease in coercivity, from 5000 to 1500Oe, with TEM showing an accompanying increase in average particle size (from 100 to 170nm). In (b) there is no clear trend. By increasing the aqueous concentration (c), an increase in coercivity (750→1750Oe) was noted and was accompanied by a decrease in particle size (from 170 to 120nm). These results reflect how morphology and magnetic properties are interrelated and can be controlled.

Figure 2: Effect of Surfactant Concentration on Coercivity of BaF Prepared via Microemulsions at Different Aqueous Concentrations; (a) 0.10, (b) 0.15, (c) 0.20

In conventional co-precipitation, increasing the reagent concentration results in an increase in average BaF particle size and a decrease in coercivity (from 5600 to 5300Oe). The use of microemulsions prevents this effect, suggesting that the major influence is the composition of the microemulsion and not concentration. This permits concentration of the aqueous phase, so that relative yields of BaF product can be increased. Interestingly, the quality of BaF produced by a NaOH containing microemulsion was superior.

4. Conclusion

This preliminary work proves that it is possible to nano-engineer barium ferrite with controlled morphology and magnetic properties by employing water-in-oil microemulsions. The concentration of the aqueous component does not effect the morphology or magnetic properties of the pigment and so by increasing this, allows the relative final product yield to be maximised. There is then a choice of co-precipitant (including ammonium carbonate and sodium hydroxide) and the resultant ferrite using sodium hydroxide appears to have superior magnetisation whilst still allowing its coercivity to be manipulated.

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