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Synthesis and Characterization of Chromium Substituted Aqueous Ferrofluid

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Abstract: Chromium substituted aqueous magnetite ferrofluids with different concentrations of chromium were synthesized by co-precipitation method. Tetramethylammoniumhydroxide (TMAOH) was used as the surfactant. The synthesized samples were characterized by XRD. The X- ray diffraction analysis showed inverse spinel structure of the samples with crystalline size ranging from 11nm to 22nm. Magnetic measurements were done by employing Vibrating Sample Magnetometer (VSM). Saturation magnetizations of the samples were found decreasing with increase in concentration of chromium which is due to the cation redistribution. Chromium ferrite possess a normal like spinel cubic structure in which chromium ions occupies the octahedral sites which enhances antiferromagnetism and ferrimagnetism gets lowered.
Keywords: Synthesis and Characterization of Chromium Substituted Aqueous Ferrofluid.

Introduction

Iron oxides are one of the most important metal oxides of technological importance one of which is magnetite (Fe_3O_4)[1]. It is crystalline and magnetic in nature and their nanoparticle colloidal suspensions give magnetic fluids known as ferrofluids. They are colloidal suspensions of single domain magnetic particles stabilized in solution with the help of surfactant[2]. Characteristics of these metal oxides include mostly the trivalent state of iron and hence their properties can be easily modified by substituting them with another metal cation.

Trivalent chromium irons can be used as a substitute for iron ions in magnetite which can bring out remarkable changes in the magnetic properties of magnetite. Brajesh pandey has reported the synthesis of chromium ferrite powder by citrate precursor route in different size ranges and the effect of particle size on magnetic properties also has been studied[3]. M. Raghasudha etal have synthesized chromium substituted cobalt nanoferrites by Citrate-Gel Auto Combustion method and variation in properties of the sample with increase in Cr content has also studied.

Ferrofluids possesses a wide variety of application such as in accelerometers, as magnetic ink, in rotating shaft seals and in various medical applications such as hyperthermia treatment cell separation and tumor treatment[4,5]. This paper concerns the structural and magnetic properties of chromium substituted ferrofluid with water as the carrier fluid. The variation of properties of the sample with three different concentration of chromium is also studied.

Experimental

Synthesis

Chromium ferrite was synthesized by co-precipitation method in which 2 Molar $FeCl_3$ (3.244g,10ml) solution is mixed with 1 Molar $FeCl_2$ (1.988g,10ml) solution. Water is used as the solvent for synthesis. Chromium chloride ($CrCl_3$) solution with a molarity of 0.2 molar (0.5329g,10ml) was added to this and precipitation reaction is done by the subsequent addition of NH_4OH solution. The formed black precipitate is $Cr_{(0.2)}Fe_{(0.8)}Fe_2O_4$ which is dried and suspended in water with the help of TMAOH as surfactant to get the required ferrofluid[4]. All the procedures were done in room temperature. The same procedure is repeated with chromium chloride solution of molarities 0.4M (1.0658g,10ml) and 0.6M (1.5987,10ml) to get $Cr_{(0.4)}Fe_{(0.6)}Fe_2O_4$ and $Cr_{(0.6)}Fe_{(0.4)}Fe_2O_4$ samples[6] .

Structural characterization

The prepared ferrofluid is filtered and dried out in open air to get chromium ferrite powder. XRD analysis of these samples were done to find the grain size of the particles. X ray diffractometer (Rigacu Dmax-C) using Cu-Kα radiation of wavelength 1.5406A^o were used

Magnetic Characterization

Magnetic properties of the chromium ferrite powder synthesized were analyzed using Quantum Design-Versa Lab vibrating sample magnetometer. Hysteresis measurements were carried out for the determination of saturation magnetization (M_s).

Result and Discussion

Structural Characterization

Chromium substituted aqueous ferrofluid with three different concentrations of chromium were prepared. X Ray diffraction diagram of each of the sample is as shown in figure.

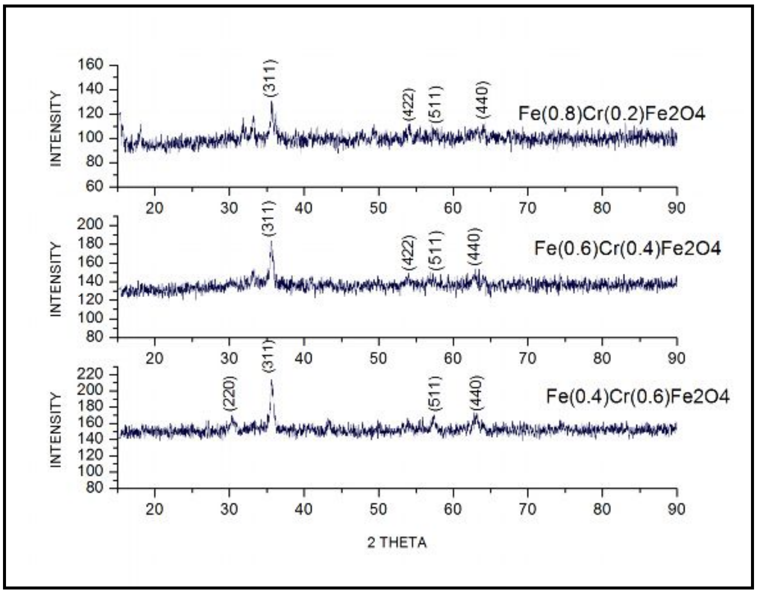


Figure 1. XRD pattern of $Cr_{(0.2)}Fe_{(0.8)}Fe_2O_4$, $Cr_{(0.4)}Fe_{(068)}Fe_2O_4$, $Cr_{(0.6)}Fe_{(0.4)}Fe_2O_4$ nanoparticles

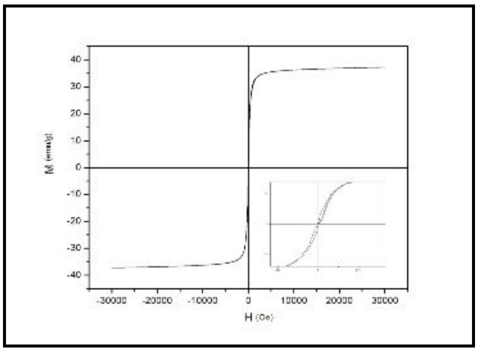
The peak positions in the XRD pattern of all the samples synthesized matched well with the positions of spinel structure. Peaks were broad due to smaller grain sizes of the samples. This is because as particle decreases no. of broken bonds present at the surface increases which offers more scattering centres. Samples showed cubic spinel structure with strongest reflection from (311) which indicate spinel phase. From the X ray diffraction pattern it is clear that the ferrofluids synthesized are polycrystalline in nature[3]. Particle size were calculated using scherrer formula

$$D=\frac{0.9\lambda}{\beta\cos\theta}$$

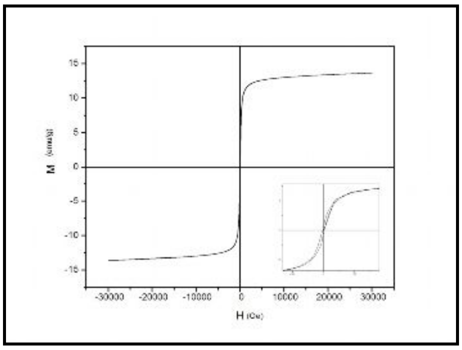
Where λ is the wave length of X-ray, β is the full width at half maximum in radians and θ is the Bragg's angle at peak position. Crystallite sizes of all the samples were found to be in the range of 11nm to 22nm.

Magnetic Characterization:

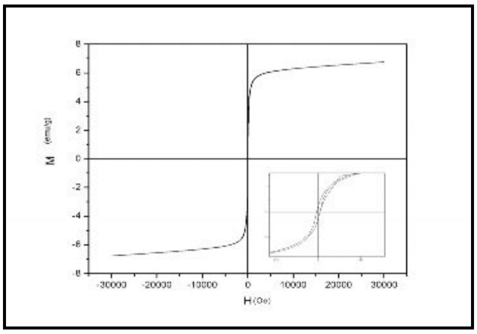
Magnetic properties of the samples were measured using Vibrating Samples Magnetometer. M-H curve for each sample is as shown.



(a)



(b)



(c)

Figure 2. Magnetization curve for (a) $Cr_{(0.2)}Fe_{(0.8)}Fe_2O_4$, (b) $Cr_{(0.4)}Fe_{(0.6)}Fe_2O_4$ and (c) $Cr_{(0.6)}Fe_{(0.4)}Fe_2O_4$ nanoparticles

All these curves show a typical S type magnetization curve with very low coercivity which enables them to be used as soft ferrites. The saturation magnetization of ($Cr_{0.2}$ $Fe_{0.8}$ Fe_2O_4) is 33.305 emu/g. For ($Cr_{0.4}$ $Fe_{0.6}$ Fe_2O_4) magnetization decreased to 10.5844 emu/g. For ($Cr_{0.6}$ $Fe_{0.4}$ Fe_2O_4), magnetization is about 5.6602 emu/g, lower than the other two samples. The following graph describes the variation of saturation Magnetization with the percentage of chromium in the samples.

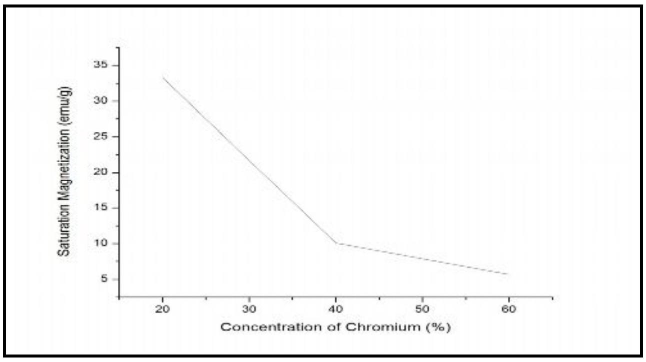


Figure 7: Variation of Saturation Magnetization of the Samples with increase in Concentration of Chromium

Above results shows that saturation magnetization of the sample decreases as the concentration of chromium increases. If Chromium goes to the B site, just as the Fe2+ ions, the magnetization would be slightly lower than magnetite (for ferric ion effective magnetization is $5\mu_B$ where as for trivalent chromium ion it is 3 μ_B). However, a significant reduction in magnetization is observed which is due to the probable cation redistribution. This shows that the chromium ions keep the octahedral preference and hence there is a chance of obtaining a more normal like spinel structure in which Cr3+ ions goes to the octahedral sites by replacing Fe3+ ions with Cr3+ and Fe2+ ions having moments arranged in an anti parallel condition which enhances the antiferromagnetic nature and hence ferrimagnetism gets lowered[7].

Conclusion

Chromium substituted aqueous ferrofluids with different concentrations of chromium were synthesized by co-precipitation method. The synthesized samples were polycrystalline and almost phase pure with negligible impurities. The particle size ranged from 11nm to 22nm. Magnetic hysteresis measured through hysteresis measurements showed that saturation magnetization of the sample decreases with increase in concentration of chromium. This is because it possesses a normal like spinel cubic structure in which chromium ions occupies the octahedral sites which enhances antiferromagnetism and ferrimagnetism gets lowered[8].

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