Magnetic behavior of Fe₃O₄ nanoparticles on post-HCL treatment in synthesis

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Abstract

During the time of synthesis after precipitation of Fe₃O₄, the post treatment has been conducted with varying volume concentration of diluted HCL. The effect on magnetic properties was studied to analyze at ambient temperature 300K. For inverse spinal approximately 17nm Fe₃O₄ nanoparticles, the single domain is found diminishing and superparamagnetism enhancing with a approximation to linear rate for higher concentration.

Keywords: Synthesis of Magnetic Nanoparticles, Iron Oxide Nanoparticles, hysteresis, post treatment of HCL

1 INTRODUCTION

Magnetic Fe₃O₄ is found very versatile material in the running year due to its coverage of the scopes (medical, industrial area) and applicability (in drug delivery, MIR etc and as Ferrofluid) [1-9]. As per the requirement, various methods of synthesis have been employed. Coprecipitation is one which has considered for narrower distribution in size with greater quantity production. [10] During synthesis sometimes HCl has been utilized. The reaction of magnetite nanoparticles formation is depended on initial pH and temperature of Iron salt solution [11]. Synthesis with final pH between 9.7-10.6 produced nearly pure magnetite with little or no other associated Iron Oxide and also the saturation magnetization measurements have
evidenced a remarkable difference of the magnetic behavior of samples, and it was depending on the final pH of the solution after reaction. [12]. Does any change in magnetic behavior occur on the post treatment of HCL or not? HCl-modified Fe₃O₄ nanoparticles solution was found stable, clear, transparent cationic colloid. The results showed that HCl had a great influence on the dispersivity of Fe₃O₄ nanoparticles [13]. But in that paper, the study has reported almost no influence on the magnetism of the material. Therefore in a present work aimed to investigate the effect of post HCl treatment on the magnetic properties of Fe₃O₄.

2 MATERIAL AND METHODS

The Fe₃O₄ is synthesized using chemical route coprecipitation method. GR grade FeCl₃.6H₂O and FeSO₄.7H₂O and extra pure NH₃ Chemicals from Merck and double distilled water were used without post-purification. For the synthesis the same procedure has been conducted as mention in reference paper [14]. After two hours on the completion of reaction and treatment of controlled nucleation and growth, the solution was divided in four small reactors and put in same water bath and resumed the stirring. Diluted HCL with same concentration with different volume amount were added drop wise in a three reactors. This has been then stirred for one hour. Then washed all sample by hot water few times. The powder was collected by performing dispersion in ethanol, centrifugation and decantation steps. Dried yield of all samples were kept in oven at 300K for two hour. The powder is named Fe₃O₄ for untreated, and O, A and B as treated with increasing amount of volume of HCL 1ml, 2ml and 3ml respectively during the synthesis. The stalk solutions have been prepared by 2ml of quantities of HCL mixed with 10ml distilled water.

The effect on changing magnetic properties has been analyzed using M-H loop tracer which was pre-calibrated using standard Ni material sample. And formation of Fe₃O₄ was characterized by using XRD. K-alpha of Cu X ray source of 0.154nm wavelength with step size 0.02 and range 20 to 80 degree has been used. After removal of the instrumental and broadening errors, the data has been used for analysis. Both characterization experiments were conducted at 300K temperature.

3 RESULT AND DISCUSSION

XRD has confirmed the formation of Fe₃O₄ nanoparticles (Figure1). Indexing of peak revealed (311) is highest peak and all other matched closely to the JCPDS card number 19-692. Inverse spinel structure with space group Fd3m has been revealed. Lattice parameter of cubic edge is calculated as 0.837nm. Average size is estimated as 17.70nm with standard deviation 1.76nm by Debye Scherer’s formula without considering residual strain.
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Figure 1. X-ray diffraction pattern of Fe₃O₄ which is not treated with HCL during synthesis

Figure 2: M-H loop of the samples (Inset: The return part of hysteresis which is close to Hc i.e where the y-axis intersects).

The M-H plots are showing the behaviors of all samples are very close to an Anhysterisis. From the Figure2 it is observed that there is a significant change in saturation magnetization due to post HCL treatment. The formation of below single domain phase (i.e. close to superparamagnetic) has been confirmed. The M-H plots and its essential details have been illustrated in Figure3 and Figure4.

Table 1 summarized the numbers, how Hc increases for the first concentration and then decreases for further samples. And the Figure3 shows the slope at Hc of an O sample decreases with higher rate than later. But it is also showing higher deviation than the rest. Let us consider the size dispersion would be larger due to size of some particles has reduced in O. Then in samples A and B the size dispersion again minimized as in Fe₃O₄. Because almost all particles must have reduced their size, this must reduced the Ms which has been confirmed from Figure4. The rate of decrease in Ms from Fe₃O₄ to O sample is larger than O- to A- and A- to O- samples. The rates of change in Ms are -7.35, -3.43 and -3.05 respectively. Remanence are also observed decreasing with post-treatment of HCL. Remanence ratios are indicated that it is good for treatment of higher quantity of HCL. This is the indication of the occurrence of monodispersion in size which had been previously considered. Ms, Mr, Hc and Remanence ratio are found linear in nature.
Figure 3: nature of change in the slope at Hc and corresponding standard deviation obtained in regression of data in linear fitting.

Figure 4: The effect increasing amount of HCL in post-treatment on Mr and Ms (upper plot) and effect on Remanence ratio (lower plot).

Table 1: Magnetic properties variation

<table>
<thead>
<tr>
<th>Sample</th>
<th>Hc (G)</th>
<th>Slope at Hc</th>
<th>S.D. Slope at Hc</th>
<th>Mr (emu/g)</th>
<th>Ms (emu/g)</th>
<th>Rem. Ratio Mr/Ms</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe₃O₄</td>
<td>105.4</td>
<td>94.133</td>
<td>0.12447</td>
<td>9.422</td>
<td>63.09</td>
<td>0.1493</td>
</tr>
<tr>
<td>O</td>
<td>113.0</td>
<td>80.545</td>
<td>0.82487</td>
<td>9.337</td>
<td>55.74</td>
<td>0.1675</td>
</tr>
<tr>
<td>A</td>
<td>91.2</td>
<td>81.093</td>
<td>0.18219</td>
<td>7.413</td>
<td>52.31</td>
<td>0.1417</td>
</tr>
<tr>
<td>B</td>
<td>81.1</td>
<td>77.042</td>
<td>0.17148</td>
<td>6.02</td>
<td>49.26</td>
<td>0.1222</td>
</tr>
</tbody>
</table>

4 CONCLUSIONS

Therefore only the post HCL treatment is not enough for the sake of goodness of monodispersibility in size of the nanoparticles to shape their better magnetic behavior but also its volume concentration is required. But for that the saturation magnetization gets reduced, which has to be higher for many applications with fast switching (below single domain). Nearly linear variations in magnetic parameters have been obtained for post-treatment and shows reduction of magnetic properties. Therefore there must be the limiting value of volume concentration of HCL in post-treatment is revealed.
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REFERENCES


