Green Synthesis of Fe_3O_4 Nanoparticles: Structure and Magnetic Properties

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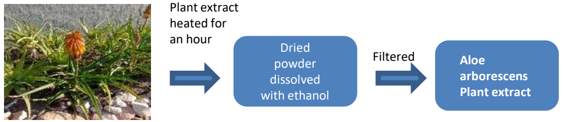
Introduction

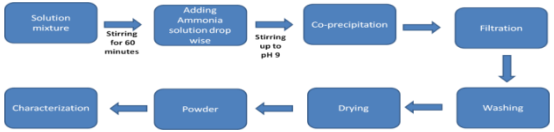
- Magnetite (Fe_3O_4) nanoparticles (NPs) is one of the naturally occurring minerals that has importance in paleomagnetic measurements used to study continental drift and it is also found in the mantle wedge of subduction zones [1].
- Magnetically, Fe_3O_4 orders ferrimagnetically with a Curie temperature of 850 K having an inverse spinel structure, AB_2O_4, with the A site occupied by Fe^{3+} and the B sites populated equally by Fe^{3+} and Fe^{2+} at room temperature [2].
- Interestingly, Fe_3O_4 demonstrates a metal-insulator transition, popularly known as the Verwey transition, at a temperature T_v = 120 K and below which it shows a two-fold increase in the resistivity [2].
- As a consequence, the B sites are randomly occupied by Fe^{2+} and Fe^{3+} even at high pressure. Charge ordering can explain the Verwey transition [3].
- Fe_3O_4 plays an important role as a catalyst in inorganic processes, such as in the synthesis of ammonia and the dehydrogenation of ethyl benzene to styrene [4].
- Fe_3O_4 is a potential candidate for use in various fields – also in the field of magnetic recording media coupled with the imaging of atomic structure and the electronic properties of the surface where there are high demand at present [4].
- With the various importance of Fe_3O_4, the present work aimed to synthesis of Fe_3O_4 NPs using a novel green synthesis approach with *Aloe arborescens* plant extract by the co-precipitation method.
- The effect doping and synthesis methods on Cr^{3+} at Fe^{3+} site is discussed based on the structure, magnetic properties, particle size and morphology in this work.

Methodology

Plant Extract Preparation



Synthesis of NPs by Co-precipitation Method



Results and Discussion

X-Ray Diffraction (XRD) pattern

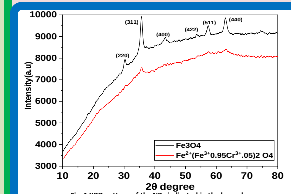


Fig. 1 XRD pattern of the NPs indicated in the legend.

- XRD analyses confirm purity and crystalline nature of the NPs.
- Average particle size of the crystallites obtained from (311) peak at 35.6° by using Scherrer's equation: $D = 0.89\lambda/\beta\cos\theta$. For Fe_3O_4 and $Fe^{2+}(Fe_{0.95}\ Cr_{0.05})_2O_4$, D_{XRD} was found as 22 nm and 17 nm, respectively.

Transmission Electron Microscope (TEM) Micrograph and Size Histogram

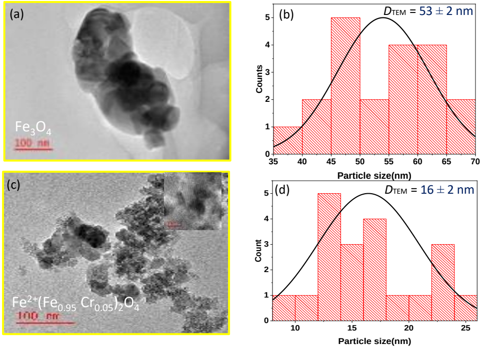


Fig 2 (a), (c) TEM images and (b), (d) particle size distribution of NPs. Inset in (c) shows HRTEM.

- TEM give shape and size of the NPs, with lattice spacing 0.546 nm from HRTEM.

Magnetic Properties of NPs: VSM studies

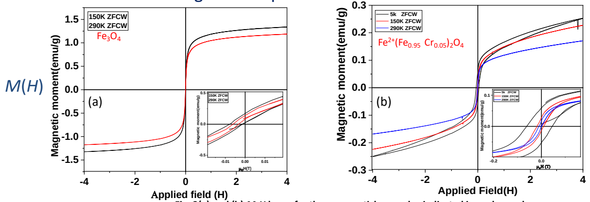


Fig. 3(a) and (b) *M-H* loops for the nanoparticle samples indicated in each panel.

- Magnetic properties of the NPs were studied in a temperature range of 2 < T < 300 K.
- Hysteresis loops indicate that the synthesized NPs have ferrimagnetic behaviour.
- From hysteresis loops the following parameters are obtained: saturation magnetization (M_s), remanent magnetization (M_r) and coercive field (H_c).

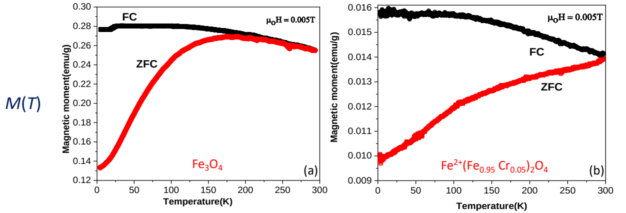


Fig. 4 (a) and (b) Temperature dependent magnetization curves of NPs indicated in each panel.

- Fig. 4 (a) and (b) shows magnetization as a function of temperature, under zero field cooling (ZFC) and field-cooling (FC) modes. Bifurcation to 300 K suggest T_C > 300 K.

Conclusion

- Green synthesize method was successful: The purity and crystalline nature of the synthesized NPs were confirmed with the powder x-ray diffraction studies.
- From the powder XRD, the size of the NPs were calculated for Fe_3O_4 (22 nm) and $Fe^{2+}(Fe_{0.95}\ Cr_{0.05})_2O_4$ (17 nm) by the Scherrer's equation.
- The size of the Fe_3O_4 and $Fe^{2+}(Fe_{0.95}\ Cr_{0.05})_2O_4$ NPs were calculated from the TEM micrograph and the particle sizes is found to be 53 ± 2 nm and 16 ± 2 nm, respectively.
- Temperature and applied field dependent magnetization measurements confirm the retention of ferrimagnetic behavior up to 300 K.

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