

## Effect of Doping in Magnetic Character in $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> Nano Particle

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### ABSTRACT

Fe<sub>2</sub>O<sub>3</sub>Ti and Fe<sub>2</sub>O<sub>3</sub>Mg nano particles were prepared by self-propagation method using PEG as capping agent. Nano particle was characterized by XRD (X-ray Diffraction), SEM (Scanning Electron Microscope), and VSM (Vibrating Sample Magneto Meter). Conform the structural, textural and magnetic property. Surface morphology changed cubic to needles shape and size where in between 25-70 nm confirmed by SEM analysis, XRD analysis shows that doping reveals the cubic with hexagonal edge structure. VSM shows the magnetic behavior of the sample.

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### Introduction

Magnetism is a phenomenon by which materials exert an attractive or repulsive force on other materials. The magnetic field is seen whenever electrically charged particles are in motion. This can arise either from the movement of electrons in an electric field, resulting in “electromagnetism”, or from the constant subatomic movement of electrons, resulting in what is known as “permanent magnetism”. The origin of magnetism lies in the orbital and spin motions of electrons. Electron interactions have also strong effect of magnetism<sup>(4)</sup>.

### Classifications of Magnetic Materials

The very first distinction is based on atoms carries permanent magnetic dipoles or not the materials which lack permanent dipoles are called **diamagnetic**<sup>(9)</sup>. If the atoms of the material carry permanent dipoles, such a material may be paramagnetic, ferromagnetic, antiferromagnetic or ferromagnetic, depending on the interaction between the individual dipoles. If the permanent dipoles do not interact among themselves, the material called **paramagnetic**. If the interaction among permanent dipoles is strong such that all dipoles line up in antiparallel direction, the material is **ferromagnetic**. If the permanent dipoles line up in antiparallel direction, the material is **antiferromagnetic** or **ferrimagnetic**. We discuss a little about ferromagnetism.

### Domain Theory of Ferromagnetism

A region in Ferro or Ferri-magnetic material where all the magnetic moments are aligned in the same direction is called a **domain**<sup>(10)</sup>. A specimen when magnetized suddenly gives a slight change in its length which is due to rearrangement of domains inside. This is called **magnetostriction**.

### The Motion Domain Walls

Increasing in the volume of domains that are favorably oriented with respect to the magnetizing field at the cost of those that are unfavorable oriented shows in the fig-1(b); fig-1(a) shows that arrangements of domains for a zero resultant magnetic moment in a single crystal or virgin specimen when there is no applied magnetic field.

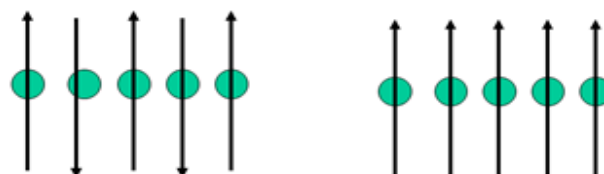


Fig .1a

Fig .1b

### Rotation of Domains

The rotation of the direction of magnetization of domain along the direction of field shown in the figure-1(c). In the weak magnetizing field the magnetization of the specimen is due to the motion of domain walls and in stronger that is due rotation of domain.



Fig .1c

### Magnetic Nano Particle

Nanometer-scale materials with novel properties have attracted extensive interest in a variety of fields<sup>(1)</sup>. Magnetic nanoparticles exhibit size effects and show interesting phenomena such as super para magnetism and quantum tunneling of magnetization. Because of the unique properties, magnetic nanoparticles have great potential for applications in diverse areas such as cell separation<sup>(8)</sup> and magnetically guided drug delivery, magnetic resonance<sup>(4)</sup> (MRI) contrast enhancement and Ferro fluid & industrial waste water treatment.

### Experimental

#### Materials and methods

Ferrous ammonium sulphate and oxalic acid used in this study are AR grade chemicals.

Polyethylene glycol of molecular weight 6,000, Magnesium sulphate ( $MgSO_4$ ) & Titanium dioxide ( $TiO_2$ ) is AR grade chemicals. Double distilled water was used to make all solutions. Nanoparticles were prepared by self-propagation method.

### Preparation of Iron oxalate

In this paper iron oxalate is used as a precursor for preparing  $\gamma$ - $Fe_2O_3$ . Precursor is prepared by dissolving equimolar amount of ferrous ammonium sulphate and oxalic acid in a minimum amount of water. The mixture is stirred well in one liter beaker using a magnetic stirrer at room temp. After 30 min dark yellow precipitate of iron oxalate dihydrate was obtained. The precursor was cleaned with distilled water three times and washed with acetone for removing trace of carbon impurity. Finally it was dried under high vacuum.

### Synthesis of Gamma Ferric Oxide ( $\gamma$ - $Fe_2O_3$ )

In the present study, we use ferrous oxalate as precursor. Thermal decomposition of Iron Oxalate precursor was done by employing Polyethylene glycol (PEG) as a surface modifying agent. In the present process, a stainless steel bowl shaped container was used. The ferrous oxalate was taken and mixed with PEG in the ratio of 1:5. The container was given heat treatment, the reaction mixture burned initially with an oxidizing flame which then converts to reducing flame and leads to formation of final residue. The flame once got ignited leads to the formation of high surface  $\gamma$ - $Fe_2O_3$  as residue. The whole reaction is completed within 10 minutes. On cooling to room temperature, no traces of carbon impurities were observed in the final residue of  $\gamma$ - $Fe_2O_3$ . The highest temperature of the reaction was found to be around  $500^\circ C$ .

### Synthesis of Ti and Mg doped with Gamma Ferric Oxide ( $\gamma$ - $Fe_2O_3$ )

To prepare doping material, the following solutions were used. Fixed concentration of  $TiO_2$  &  $MgSO_4$  (0.001Mol/L) and  $Fe_2O_3$  (0.1Mol/L) was used<sup>(1, 2)</sup>. The solution of  $Fe_2O_3$  was taken in a beaker then 30 ml of  $TiO_2$  &  $MgSO_4$  solutions are added separately to  $Fe_2O_3$  solution at  $100^\circ C$  for 2 hours in the ratio of 0.1ml/min, after two hours the color change from dark brown to blood red color for titanium doped material & dark brown color for Mg doped material, it indicates the formation of product. The product is washed with water and 0.01N  $HNO_3$ . Then it was dried in  $120^\circ C$  for half an hour and then calcinated at  $650^\circ C$  for 1-2hrs to get a fine powder.

## Results and Discussion

### XRD (X-ray diffraction)

XRD pattern of sample (1) and (2) are shown in the fig (2, 3) the sharp peak of XRD shows the samples have highly crystal nature. Cell parameter of normal  $\gamma$ - $Fe_2O_3$  is  $a = 3.860$ <sup>(7)</sup> but doped material it's found as  $a = 5.090$  &  $c = 14.090$ . (JCPDS Card No 89-2811) for Ti doped, and  $a = 8.366$  (JCPDS Card No 89-4924) for Mg doped. From the report Ti doped  $\gamma$ - $Fe_2O_3$  shows the change in morphology hexagonal structure. Mg doping does not give change in morphology sample remain cubic structure. But cell parameter slightly changed. This is due to the atomic radius of Mg is greater than Ferrate.

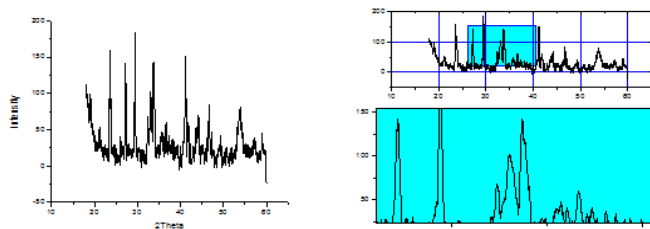


Fig. 2a

Fig. 2b

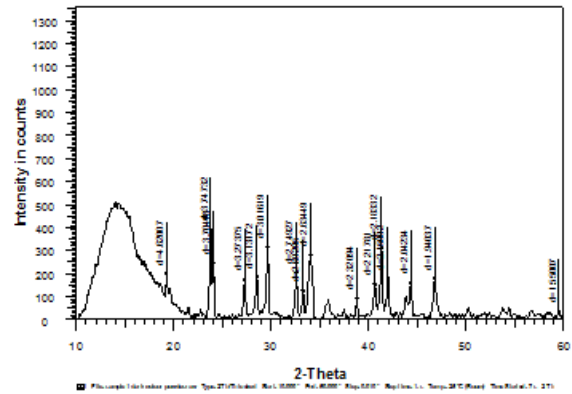


Fig. 3.

### SEM (Scanning Electron Microscope)

Scanning electron microscopy was used for further analysis of the samples morphology and size. The SEM image  $\gamma$ - $Fe_2O_3$  doped with Ti and Mg (Fig.3, 4) indicates the accurate morphology of the nano particles. From the image and calculation the average particle size is 60nm-85nm for Ti doped and 30-60nm for Mg doped. Fig 3 shows Ti doped Ferrate and Fig 4 shows Mg doped Ferrate. Fig-4(a) and Fig-4(b) shows histogram graph of particular SEM images respectively. From SEM image we get clear view about morphology change. Ti doped particle shows needle like structure and Mg doped particles spherical shape particle.

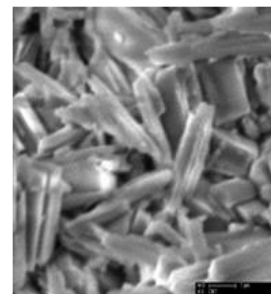


Fig. 4

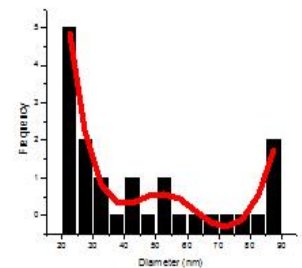


Fig. 4a

Fig-4(a).Number Frequency histograms showing particle size distribution of synthesized  $\gamma$ - $Fe_2O_3$  doped with Ti nanoparticle in linear scale. The particle size based on image analysis of less than 100 nm (60-70)

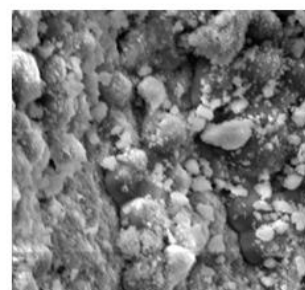


Fig. 5

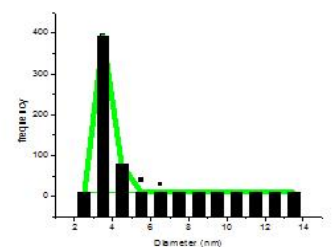


Fig.5a

Fig-5(a).Number Frequency histograms showing particle size distribution of synthesized  $\gamma$ - $Fe_2O_3$  doped with Mg nanoparticle in linear scale. The particle size based on image analysis of less than 100 nm (30-70).

### VSM (Vibrating Sample Magneto Meter)

In here normal  $\gamma$ - $Fe_2O_3$  shows the magnetization  $M_s$  as  $3.73 \times 10^5 Am^{-1}$  and the coercivity shows 60 Oe<sup>(11)</sup>. But Ti doped particle shows 344.84 Oe and  $M_s$  is found as  $2.73 \times 10^5 Am^{-1}$ , shows in Fig-6 (b).

And Mg doped particle shows  $H_c$  as 325.06 Oe and  $M_s$  as 15.00(emu/g). And the graph shows the material moves from soft magnetic material to hard magnetic material. The VSM curve shows in Fig-6 (a).

And the graph shows the material moves to soft magnetic material to hard magnetic material. Due to Structural defects such as particles of a nonmagnetic phase or voids in the magnetic material tend to restrict the motion of domain walls, and thus increase the coercivity<sup>(9, 11)</sup>. The reason for Ti doped  $\gamma\text{-Fe}_2\text{O}_3$  has higher coercivity than Mg doped  $\gamma\text{-Fe}_2\text{O}_3$  is it has lower atomic radius than Fe so it doped perfect manner<sup>(6)</sup>. But Mg has almost equal atomic radius so the magnetization and coercivity is less than Ti doped particle<sup>(3,11)</sup>.

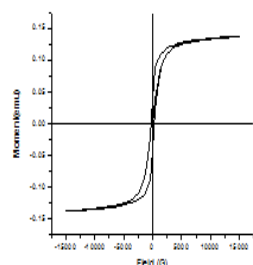


Fig .6 a

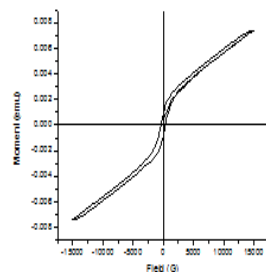


Fig.6b

### Conclusion

The  $\gamma\text{-Fe}_2\text{O}_3$  nano particle was successfully prepared by self-propagation method and doped with Ti and Mg. characterized by various instrument. From the reports we get clear view about doping materials magnetic property and effect of doping in it. Ti doped material have more magnetic moment and coercivity because Ti particle give +4 oxidation states to  $\gamma\text{-Fe}_2\text{O}_3$  and the overall oxidation state is +6 for Ti doped material. But Mg gives +2 oxidation states in doping and overall oxidation state become +4 oxidation states play a main role in coercivity. High oxidation state particles give high coercivity (Oe) and magnetization ( $M_s$ ). But doping makes both particle moves to ferromagnetic nature to paramagnetic nature<sup>(5)</sup>. From the reports we can prove the oxidation state of the  $\gamma\text{-Fe}_2\text{O}_3$  is increase. Increasing in oxidation state gives best result in heavy meal and organic wastes removing. So the prepared material is strongly recommended to use in waste water treatment.

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