

Synthesis and Characterization of Silica Coated Iron-Oxide Composites of Different Ratios

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Abstract Silica coated Iron-oxide composites of different ratios (iron oxide / SiO₂ = 3:7, 1:1 and 7:3) have been synthesized. Iron oxyhydroxide, as the precursor of iron oxide, has been prepared by electrochemical method. The Iron oxide and tetraethylorthosilicate (TEOS), as precursor of silica, have been used at different ratios to synthesize silica coated Iron-oxide composite of different ratio. Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), Differential thermal analysis (DTA), Thermogravimetric analysis (TG), Scanning electron microscopy (SEM), and Energy dispersed X-ray spectroscopy (EDS) technique have been employed to characterize the precursors as well as the prepared composite materials. The results reveal that well dispersed silica coated iron-oxide composites of different ratios were formed. The prepared silica coated iron-oxide composite material can be used for treatment of wastewater to remove heavy metal.

Keywords Silica coated composite material, Electrochemical and mesoporous

1. Introduction

In the last decades, composites are playing a vital role in synthesizing new materials. Composites provides high quality mechanical and physical properties because it combines the most desirable properties of its constituents while suppressing their least desirable properties [1]. The attractive properties shown by the composites are high thermal or electrical conductivity [2a], a low coefficient of thermal expansion [2b], adsorbent [2c, d, e], surfactant [2f], catalytic [2g], photo catalytic [2h] and elastic properties [2h]. The properties shown by the composites could not be achieved by either of the constituent materials alone.

In recent years, Silica coated Iron oxide composites have attracted a great deal of attention in the field of chemistry and applied sciences. As a result extensive study have been carried in the last few years globally on it [3, 4]. In the Silica coated Iron oxide composite, usually SiO₂ matrix structure plays the role of host for Iron oxide fine particles, which nucleates in the Silica pores, conveniently. High performance technological and scientific and operational applications such as magnetic technology [5-7], catalysis [8], sensors [9-11], optics [12], adsorbent [13] properties has widen the research on the Silica coated Iron oxide composite. Hence Silica-coated composite nanoparticles (Fe₃O₄@SiO₂/core-shell) have been synthesized and studied by many

groups [14-17]. Recently, Silica coated magnetite composite have been successfully applied to extract some heavy metals, like Cd²⁺, Cu²⁺, Hg²⁺, and Pb²⁺ from water in a wide pH range [18].

Different physical methods, chemical methods and electro-chemical methods have been using to synthesize the Silica-Coated composite materials [19]. In the present investigation, we focused our attention on the preparation of Silica coated Iron-oxide composites by Sol-Gel method because it offers wide range of advantages over other techniques [20-21]. Despite of all these, scientists all over the world are still trying to develop a technologically simple, cost effective and the most versatile sol-gel synthetic route for preparation of specific type of composite materials with desired chemical composition.

In this work, the Silica coated Iron-oxide composite particles of different ratios were synthesized through two steps. At first, the hematite particles were prepared by electrochemical method [22] and then the composite material were synthesized through the modified sol-gel method using the prepared hematite and tetraethylorthosilicate (TEOS). TEOS was used as the precursor of Silicon (IV) oxide. The precursors and composite materials were characterized with Fourier-transform infrared spectroscope, Simultaneous Thermal Analyzer (TGA/DTA) and X-Ray Diffractometer (XRD). The compatibility and morphology of the materials were investigated by using the scanning electron microscope. Composite materials prepared by different method can be used for the removal of different heavy metals from wastewater. Thus we can use composite materials for environmental applications.

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2. Experimental

2.1. Materials Used

All the chemical reagents used were of analytical grades. Double Distilled Water (DDW), pure ethanol (Merck, Germany) and analytical grade acetone (Merck, Germany) were used as solvent. Other reagents were Tetraethylorthosilicate (TEOS) (Aldrich, Germany), Hydrochloric acid, HCl (Merck, Germany), Sulphuric acid, H₂SO₄ (Merck, Germany), Phosphoric acid, H₃PO₄ (Merck, Germany), Ammonium Hydroxide (Merck, India), Sodium Chloride (Merck, India) and Sodium Hydroxide (Merck, India).

2.2. Electrochemical Preparation of Iron Oxide

The electrochemical experiment was carried out in a two-electrode electrochemical cell containing iron as sacrificial electrodes. The experimental set up included the electrode assembly, a DC power supply unit, a voltage stabilizer, a resistance box to regulate the current and a multimeter to read the current values. The electrode assembly consisted of pairs of iron plates (7.0 cm × 7.0 cm × 0.15 cm) placed in parallel arrangement. A photograph of the experimental set up is shown in **Figure 1** which is based on the diagram reported by Mollah *et al* [22]:

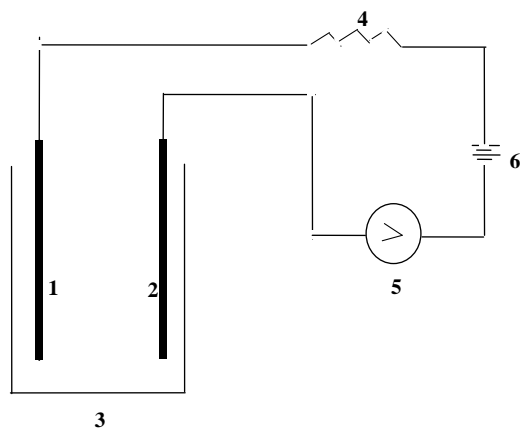


Figure 1(a). Schematic diagram of the cell. 1. Anode, 2. Cathode, 3. Pyrex beaker, 4. Variable resistance, 5. Ammeter, 6. D.C. source

During the process, the electrodes were immersed in 0.025M aqueous NaCl (99.5%) solution. Electrolysis was then carried out using 1A current for about one hour and suspension of oxyhydroxides was generated into the

electrolytic solution. At the end of each run, the suspension was kept in open air for 24 hours to settle completely and then filtered through a 2.5 μm pore size membrane filter and the recovered solid was washed with double distilled water until free from chloride. The solid mass was dried in an oven at a temperature of 120°C for 24 hours.

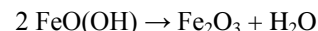
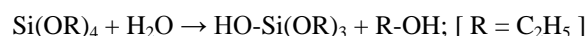


Figure 1(b). Electro-coagulator used in the process

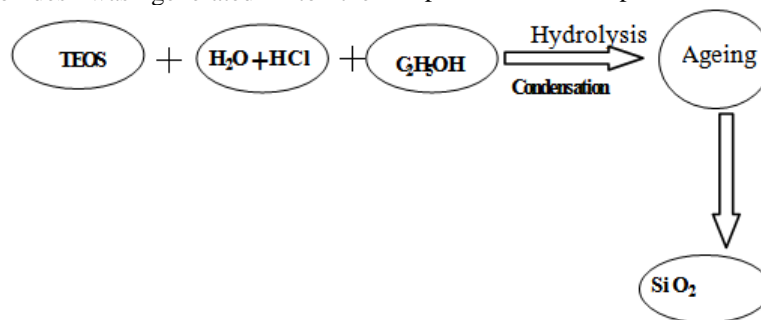
2.3. Preparation of Silicon (IV) Oxide

Silicon (IV) oxide was prepared by hydrolysis of tetraethylorthosilicate (TEOS) in ethanol. The ionic equation for the reaction between tetraethylorthosilicate (TEOS) and water in ethanol is:



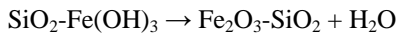
A simple one step protocol was used for the preparation of silica, which involves with the hydrolysis and condensation of tetraethylorthosilicate (TEOS) in ethanol water mixture under acidic condition at room temperature. TEOS, water and ethanol were mixed at 1.00: 10.20: 3.85 molar ratio followed by 9.00 mL water containing 0.20 mL molar HCl [23]. It was then stirred constantly using a thermostated magnetic stirrer at ambient temperature for about one hour. The stirred mixture was then allowed to stand for two days for ageing so that gel formation was completed.

The gel of silicon (IV) oxide was collected as a residue by filtration. Distilled water was poured on the silicon (IV) oxide gel several times to ensure removal of chloride or any other undesirable ions from silicon (IV) oxide because the presence of impurity will cover up the surface and hence the actual surface area will decrease. The gel was dried in an oven at 110°C for 4 hours. Silicon (IV) oxide thus prepared was preserved in a desiccator for characterization. Schematic representation of the process is shown below:



2.4. Preparation of Silica Coated Iron-oxide Composite by Sol-Gel Method

In a beaker, small amount of water was taken and reddish brown precipitate of iron oxide (prepared according to the process described in **Section 2.2**) was instantly added into it. Then the mixture was sonicated for about 30 minutes. After that the ethanol solution containing TEOS was added into that iron hydroxide. Dilute HCl solution was added to maintain the pH value of 5.0 and the mixture was sonicated for about 30 minutes. After sonication, the mixture was left for 24 hours at ambient temperature to form $\text{SiO}_2\text{-Fe(OH)}_3$ composite. The composite settled at the bottom of the beaker. Double distilled water was added to the composite and stirred for half an hour and then again left for 2 hours to settle the composite. This process was done to ensure complete removal of chloride and other undesirable ions from the composite. Water from the composite was evaporated by gently heating with a low flame and then the composite was heated in the woven at 300°C for 6 hours to convert it into composite.



The silica coated iron-oxide composite thus prepared was preserved for characterization and subsequent uses.

All three samples of the composite of different ratios were prepared in the same process (only the molar ratio were maintained as 3:7, 1:1 and 7:3).

2.5. Characterization

Silicon (IV) oxide, Iron oxide and the composites prepared in this research were characterized by FT-IR, XRD, TGA-DTA and SEM –EDS techniques.

The molecular characterization were carried out by FT-IR spectrophotometer (Model: FT IR -8400/8900) using potassium bromide pellet. The phase composition of prepared silicon (IV) oxide, iron oxide and composites were analysed by an X-ray Diffractometer. The XRD analysis of the samples were carried out by an X-ray Diffractometer (XRD) (Model: Philips X'pert) with CuK_α radiation ($\lambda = 1.54178 \text{ \AA}$) source with a nickel filter. Thermo gravimetric

Analysis (TGA) and Differential Thermal Analysis (DTA) of the samples were carried out using simultaneous Thermal Analyzer (DTA/TGA) (Model-SII EXSTAR6000 TG-DTA6300 Seiko Instrument Inc., Japan). Liquid nitrogen gas was used as a stripping gas. The samples of silicon (IV) oxide, iron oxide and silica coated composites were analysed by Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Spectroscopy (EDS). Analyses were carried out on a JOEL JSM6490LA, (Japan) equipped with germanium detector and diamond window.

3. Results and Discussion

3.1. FT-IR Spectroscopic Analysis

Close examination of the **Figure 3 (a)** shows that major absorption bands of the Silicon (IV) oxide are in good agreement with the reported values of its authentic sample. Therefore, the FT-IR analysis supports that the sample prepared was Silicon (IV) oxide.

Table 3.1. FT-IR absorption bands of Silicon (IV) oxide with band assignments and reported bands of its pristine sample

Probable band assignment	Absorption bands (cm^{-1}) [25]	Experimentally obtained absorption band (cm^{-1})
Si-O-Si bond vibration Symmetric stretching	450-460	466
vibrations of Si-O-Si belonging to ring structures	800-820	797
Asymmetric stretching vibration of Si-O-Si	1000-1260	1081
Combination of vibrations of the SiO_2 network	1640,1870,1960	1643
Due to stretching and bending vibrations of Si-O-H and O-H bonds in surface water molecules	3000-4000	3450

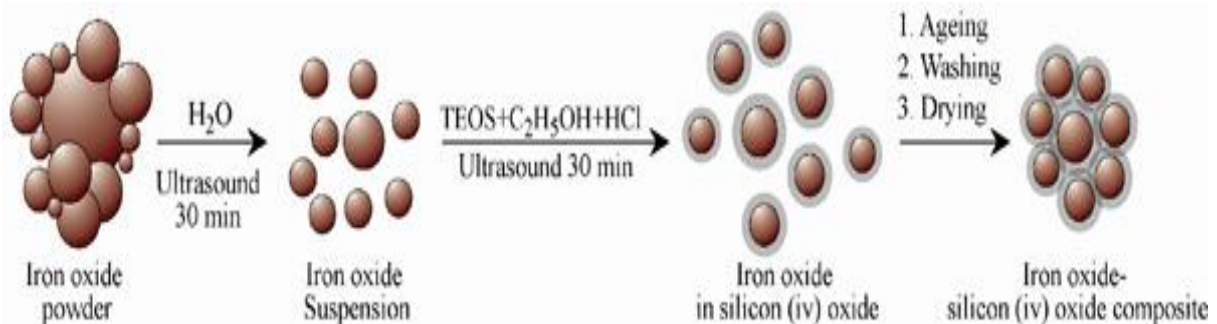


Figure 2. Illustration of the silica coated iron-oxide composite preparation based on the diagram reported by Meera [24]

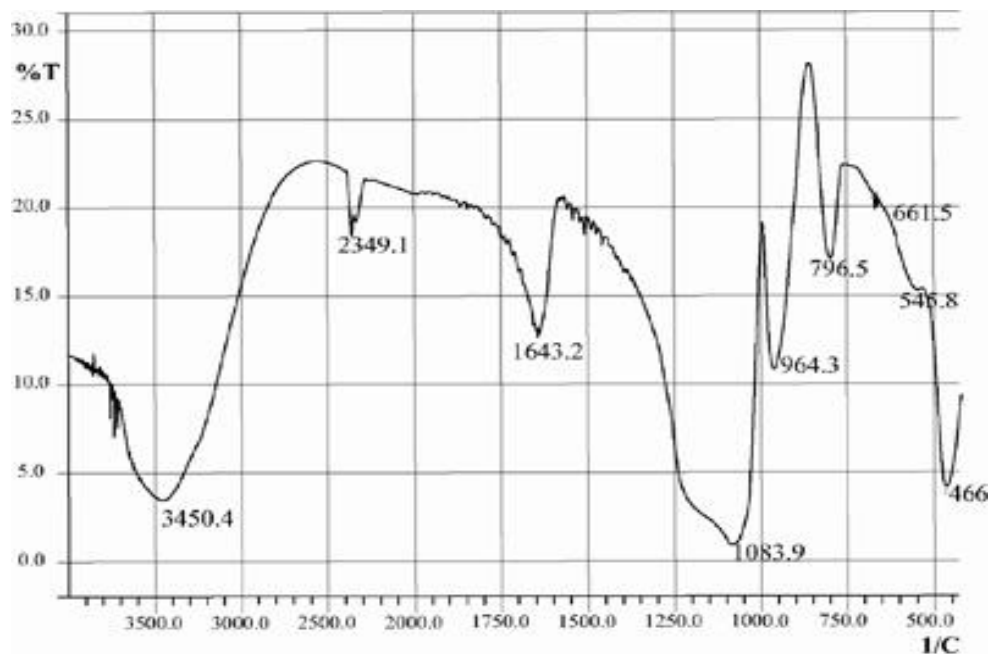


Figure 3(a). FT-IR spectrum of SiO₂

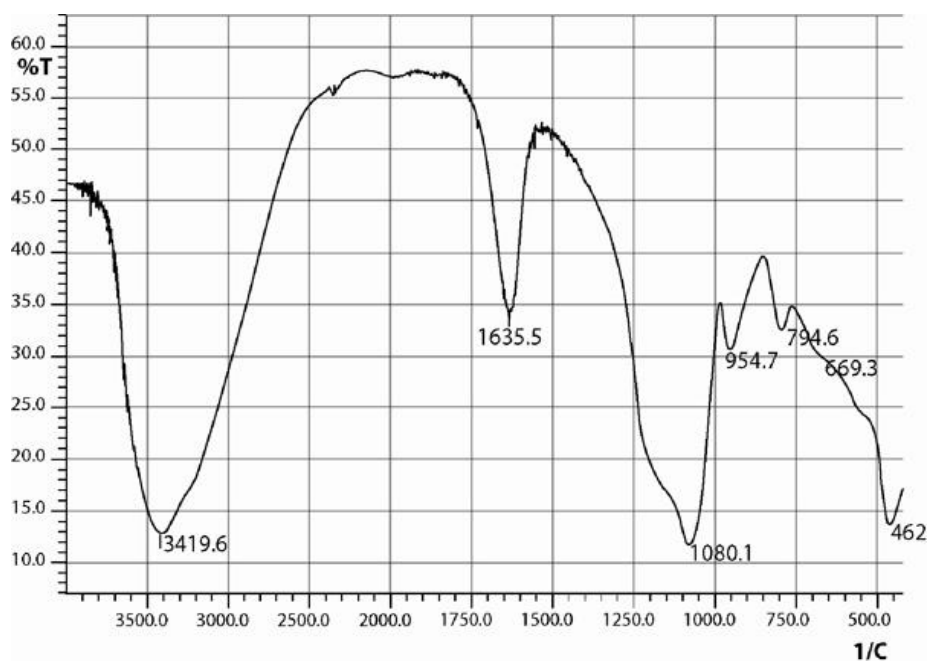


Figure 3(b). FT-IR spectra of Iron-oxide

Study of the **Figure 3 (b)** reveals that the bands at 457 cm⁻¹ and 580 cm⁻¹ were assigned to the stretching and torsional vibration modes of the hematite Fe-O bonds in the tetrahedral and octahedral sites respectively, which are in good agreement with the results reported by Mollah M.Y.A [22]. The absorption bands at 3420 cm⁻¹ and 1630 cm⁻¹ are due to stretching and bending vibrations of O-H bonds in surface water molecules. Thus the absorption bands shown by the synthesized iron oxide are in good agreement with the reported absorption bands of hematite type of iron oxide. Therefore, the FT-IR analysis provides the evidence that hematite type of Iron oxide was electrochemically generated

in this research.

Close examination of **Figure 3 (c)** shows that major absorption bands at 462 cm⁻¹, 795 cm⁻¹, 955 cm⁻¹, 1080 cm⁻¹, 1636 cm⁻¹, 2360 cm⁻¹ and 3420 cm⁻¹ shown by the Silica coated Iron-oxide composite were in good agreement with those shown by its constituents Silicon (IV) oxide and Iron oxide. These FT-IR results indicate the presence of both silicon (IV) oxide and Iron oxide in the composite.

FT-IR results of the composite (3:7) and (7:3) are shown in the **Figure 3 (d)** and **Figure 3 (e)** respectively which also indicate the presence of both silicon (IV) oxide and Iron oxide in the composite.

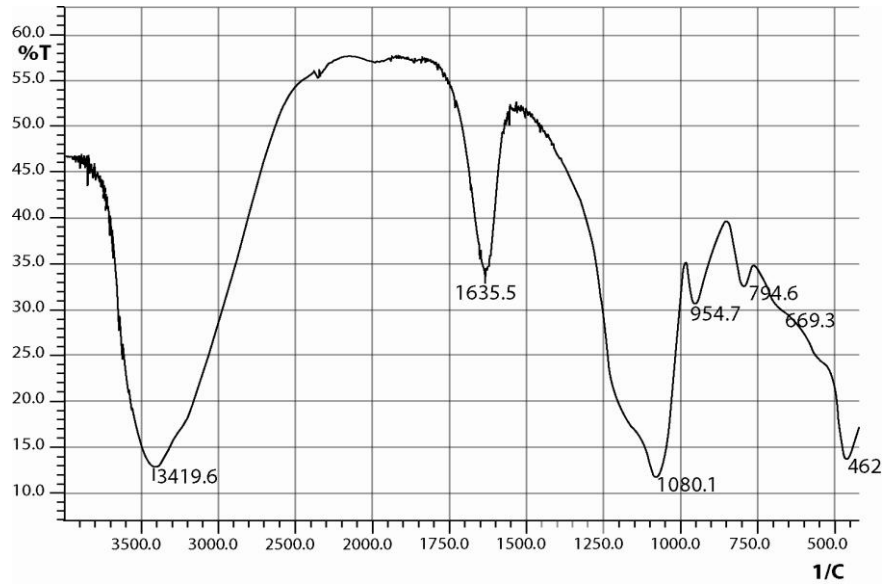


Figure 3(c). FT-IR of the Silica coated Iron oxide (1:1)

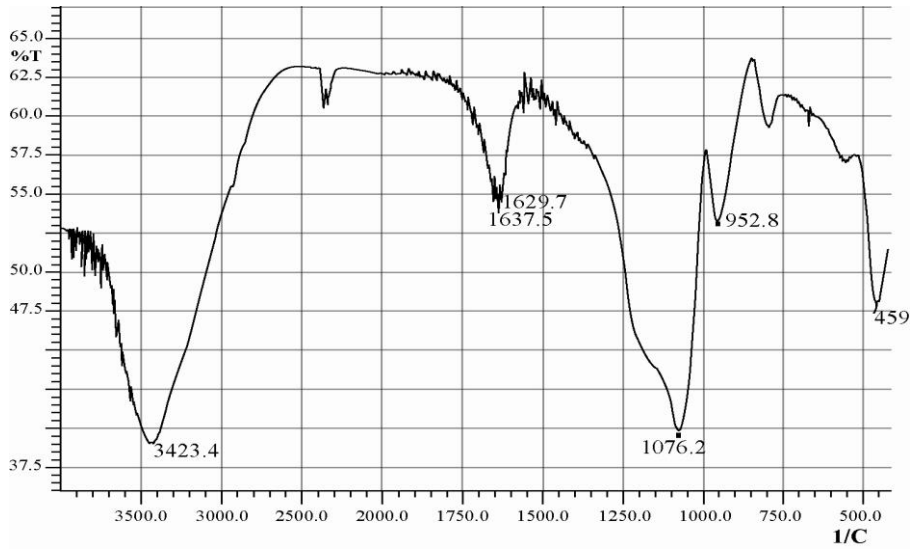


Figure 3(d). FT-IR of Silica coated Iron-oxide composite (3:7)

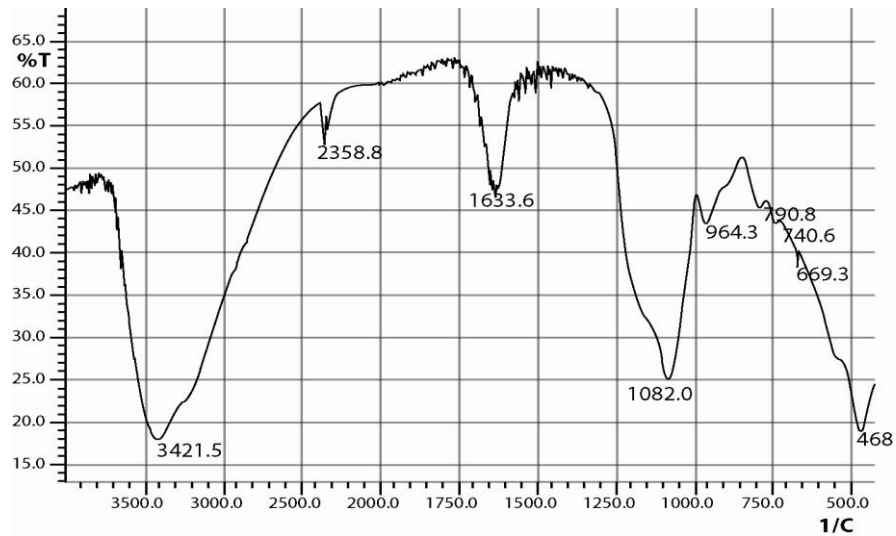


Figure 3(e). FT-IR of Silica coated Iron-oxide composite (7:3)

3.2. XRD Analysis

Figure 4 (a) shows the XRD analysis of the prepared Silica which indicates characteristics diffuse peak at about 20° . The result reveals that the silica particles are amorphous. This demonstrates that a high percentage of these particles are amorphous, but a few of them are crystalline. The result is in good agreement with the result reported by Meera basa [24] and Tabatabaei [26].

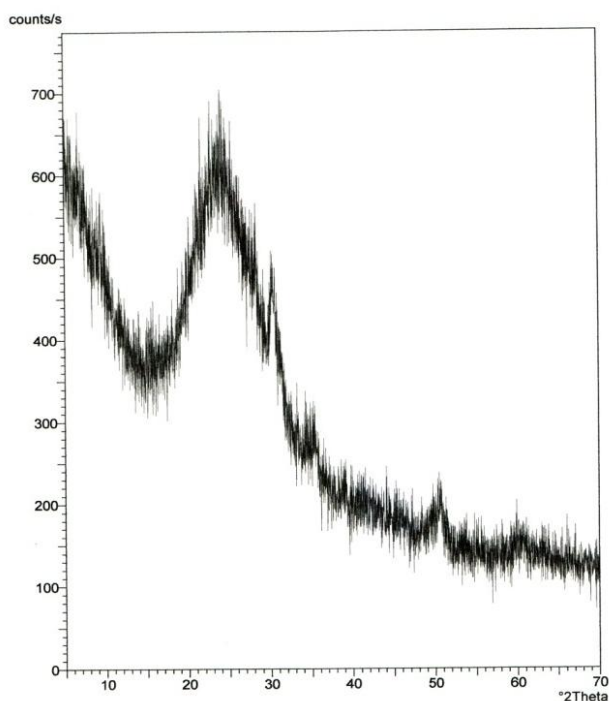


Figure 4(a). XRD of Silicon (IV) oxide

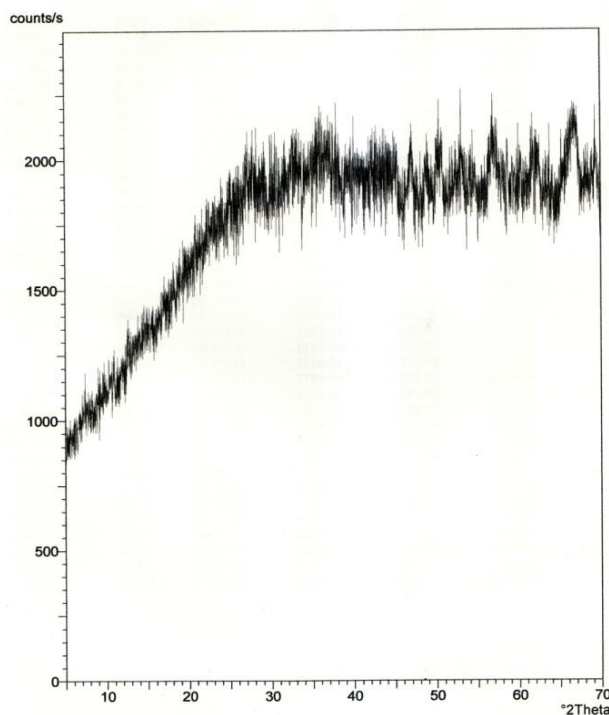


Figure 4(b). XRD of Iron oxide

Iron-oxide prepared by electrochemical method were characterized by XRD technique. **Figure 4 (b)** represents the X-ray diffraction spectra of iron oxides. The 2θ value of iron oxide sample is found to match with JCPDS-33-0664, confirming the formation of iron oxide.

Figure 4 (c) shows the XRD patterns of the thermally treated Silica coated Iron oxide composite. In the sample, besides the broad band characteristic for the presence of the amorphous silica gel, the amorphous iron-oxide was evidenced. This could be assigned to the bonding of the iron into Si-O-Fe-O-Si polymeric bonds that do not break at low temperatures of the thermal treatment. The results are in good agreement with the result reported by Raileanu *et al* [27].

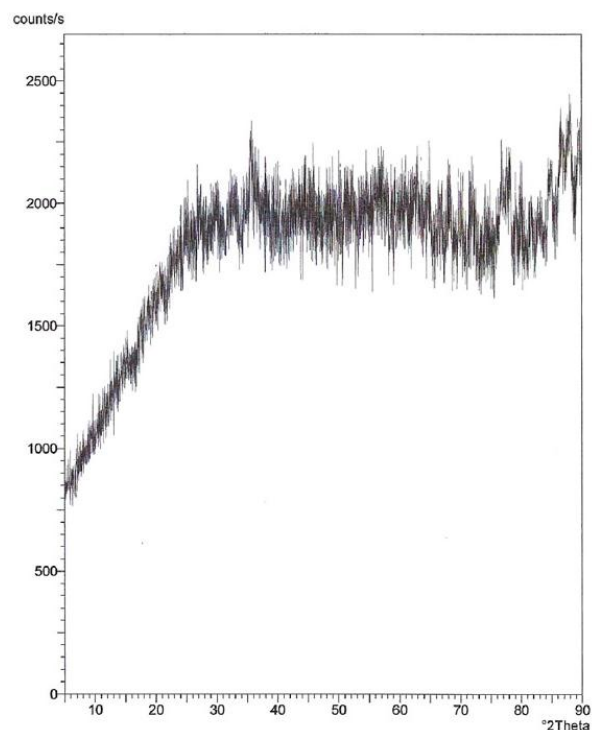


Figure 4(c). XRD Pattern of Silica coated Iron oxide composite

3.3. TGA-DTA Analysis

TGA of SiO_2 (**Fig.5a**) shows gradual weight loss from 40° to 160°C (11.0%), almost a steady state from 160°C onward due to removal of free water (absorbed from atmosphere) and presence of any structural water.

Thermogram of Differential Thermal Analysis (DTA) (**Figure 5a**) shows an endothermic effect between 86 - 150°C temperature ranges due to removal of structural water. The result is in good agreement with the result reported by Qingyin Wu [28].

Thermogram of iron oxide (**Figure 5b**) shows gradual weight loss from 26° to 176°C (10.4%) and 176° to 378°C (8.2%), almost a steady state from 378°C onward. Weight loss may be due to removal of free water absorbed from atmosphere and presence of any structural water. Gradual weight loss from 250° to 400°C (8.2%) was due to dehydroxylation for structural OH.

Differential Thermal Analysis (DTA) (**Figure 5b**) shows two endothermic peak at 94°C and 246°C temperature. The endothermic effect at 94°C may be due to removal of physically bound water and the endothermic effect at 246°C is due to removal of structurally bound water. Exotherm between 250 ° to 300°C is due to recrystallization process involving transformation of smaller crystals into larger one.

The DTA/TG result shown above for iron oxide is in good agreement with the data reported by Balek et al [29].

Thermograph of the silica coated iron-oxide (1:1) (**Figure 5c**) shows gradual weight loss from 26°C to 290°C (11.0%), almost a steady state from 290°C onward. Weight loss may be due to removal of free water absorbed from atmosphere and presence of any structural water.

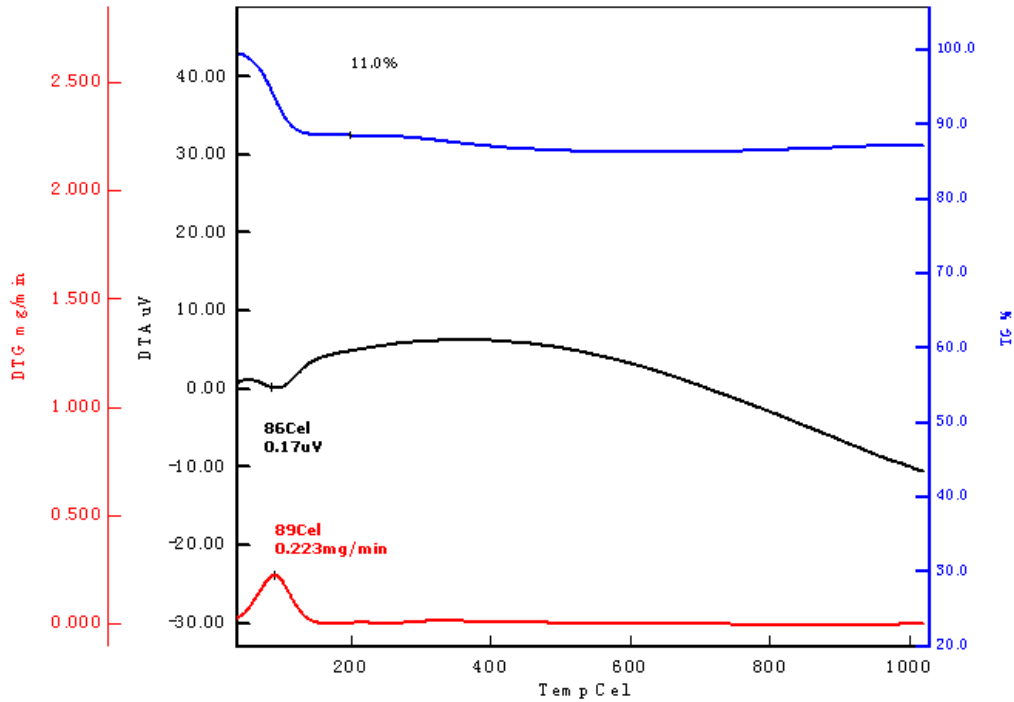


Figure 5(a). TGA/DTA of Silicon (IV) oxide

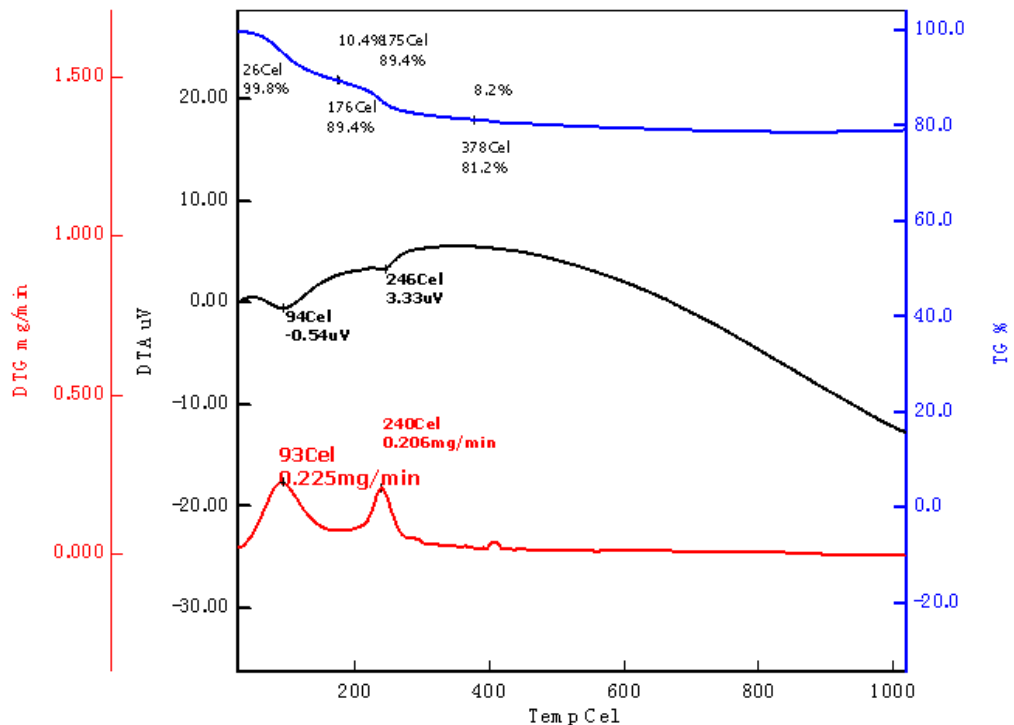


Figure 5(b). TGA/DTA of Iron Oxide

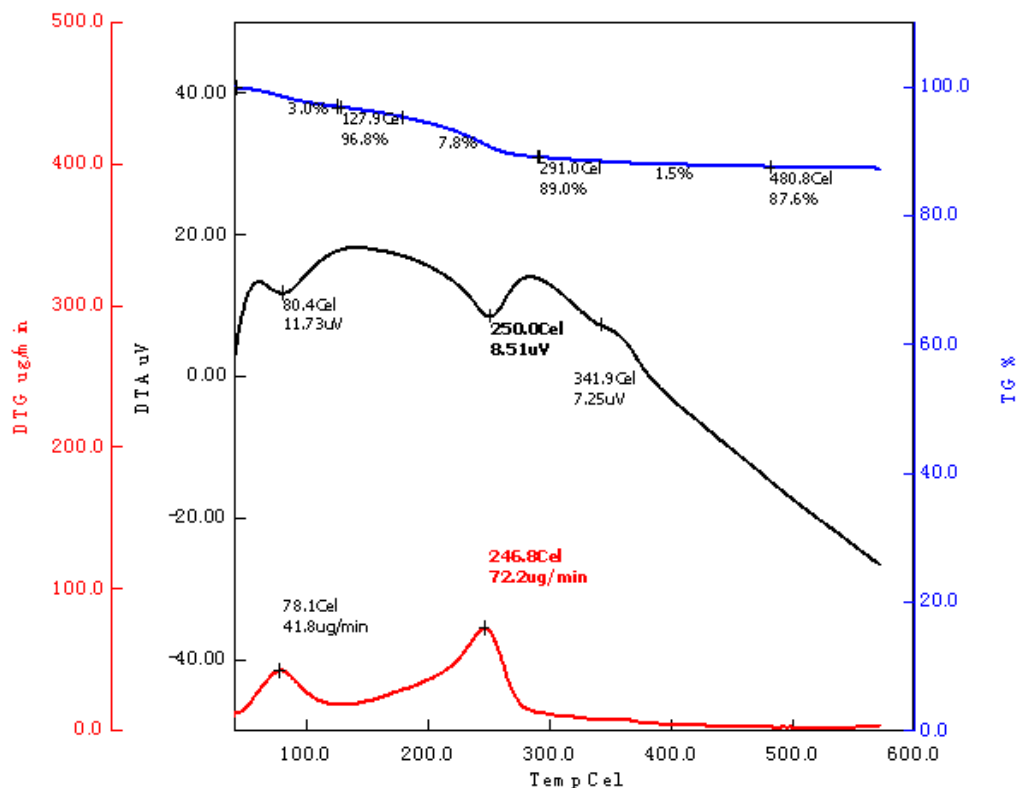


Figure 5(c). TGA/DTA of Silica coated composite

Differential Thermal Analysis (DTA) (Figure 5c) shows two endothermic peak at 80.4°C and 250°C and three exothermic peak at 160°C, 300°C and 360°C. The endothermic effect at 80.4°C and 250°C may be due to removal of structural water. Endothermic peak at 341.9°C corresponds to decomposition of goethite phase. Exothermic peak at 300°C can be considered to be due to the growth of goethite (α -FeOOH), an intermediate step of hematite. Other exothermic peaks are due to recrystallization process involving transformation of smaller crystals into larger one. The DTA/TGA result shown above are in good agreement with the result shown by Ennas *et al* [30] and Palomares [31].

3.4. SEM and EDS

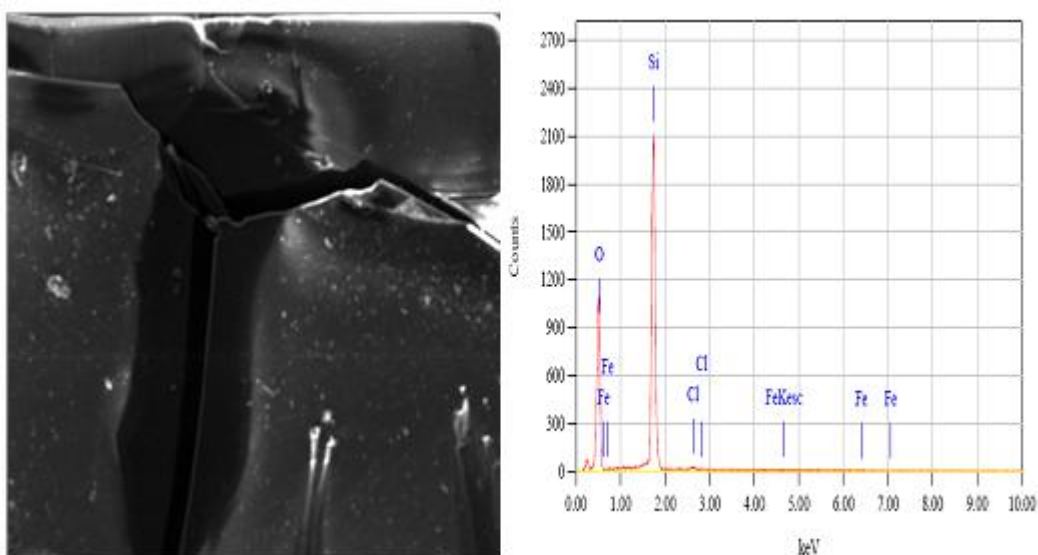


Figure 6(a). SEM & EDS image of Silicon (IV) oxide

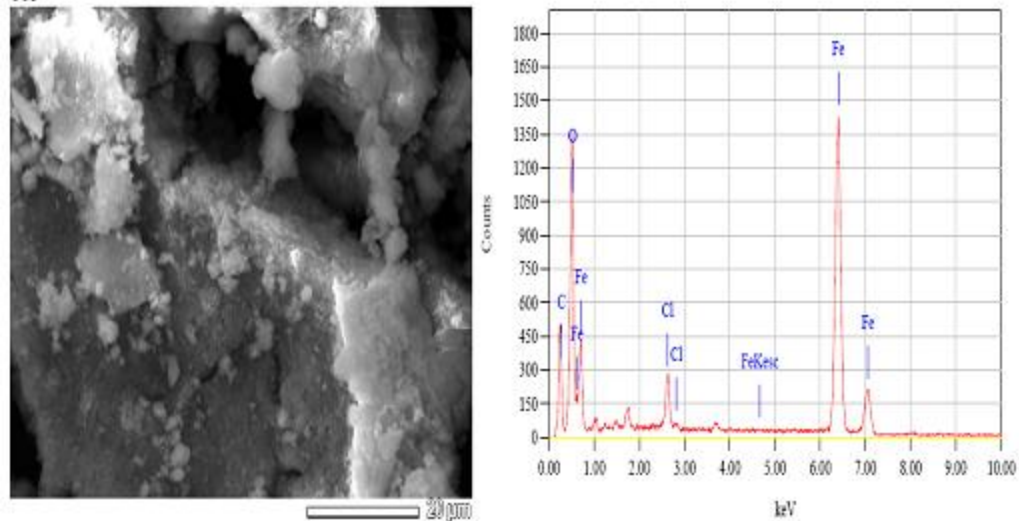


Figure 6(b). SEM & EDS image of Iron oxide

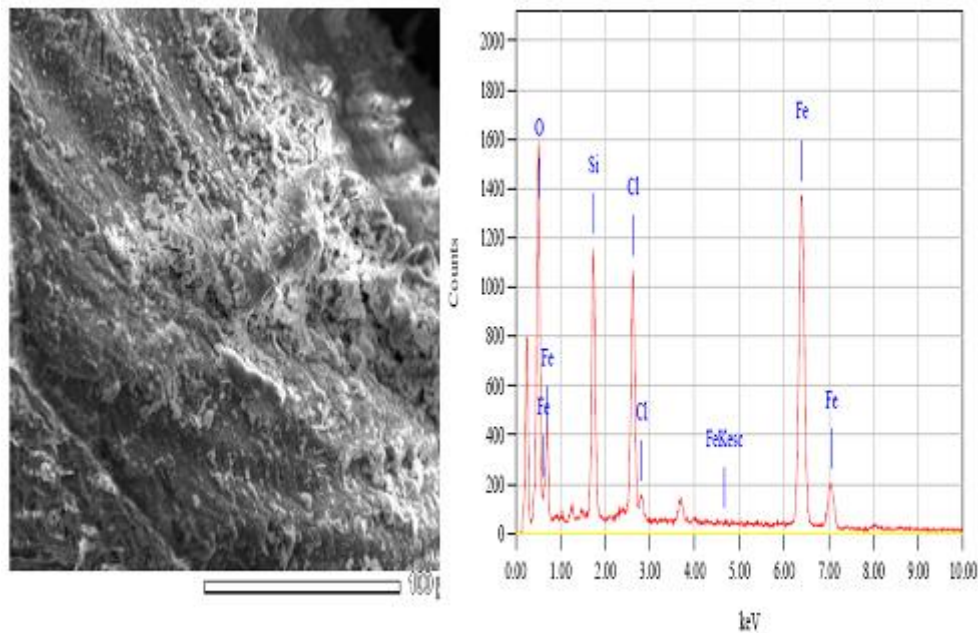


Figure 6(c). SEM & EDS image of Silica Coated iron oxide composite

The SEM result of Silicon (IV) oxide shows that the particles are in aggregated form. EDS result suggests the presence of Si and oxygen elements, confirming the formation of SiO_2 particles. Theoretically Si-O ratio is 0.875 and experimentally the value is 0.72, which is very close to the expected value. EDS result indicate the presence of trace amount of Cl as impurities.

The above **Figure 6 (b)** represents the SEM and EDS images of iron oxide which suggests the presence of aggregated particles and small grains at the surface. However the elemental analysis suggests the presence of Fe and O atomic percentage that indicates the formation of the iron oxide. Theoretically Fe-O ratio is 2.33 and experimentally the value is 2.04, which is very close to the expected value. EDS result indicate the presence of small amount of Cl as impurities.

The SEM and EDS results of the prepared Silica coated Iron-oxide composite, shown in the above **Figures 6 (c)** indicate the formation of the composite. The EDS result indicates the formation of the composite of specific composition. Theoretically Fe-Si to O ratio is 1.75 and experimentally it is found as 1.99, which is close to the expected value, confirming the formation of Silica coated Iron oxide composite. The EDS result also indicate the presence of small amount of Cl as impurities.

4. Conclusions

Silica coated iron oxide composite of different ratios (3:7, 1:1 and 7:3) were prepared by Sol-Gel method. This chemical method provides a simple, general and cost effective process for the synthesis of Silica coated Iron-oxide.

XRD and FT-IR results confirm that *Hematite* type of iron oxide and *Tridymite* (SiO_2) type of Silicon (IV) oxide were prepared in this research. Silica coated Iron oxide composites of varied ratios prepared by different method have been characterized by FT-IR, XRD, TGA/DTA, SEM and EDS techniques. Analytical results obtained by the above techniques lead us to conclude that Silica coated Iron-oxide composites were formed due to interaction between Silicon (IV) oxide and Iron oxide.

The composites synthesized in this work will have important applications not only in advanced materials but also in environmental fields such as adsorbent to remove toxic heavy metals from wastewater.

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REFERENCES

- [1] Charlie Sia Chin Voon, "Development and fabrication on the oil palm fiber/glass reinforced in aluminium composite laminates" M.Engr. thesis, University of Malaysia Sarawak, 2008.
- [2] (a) Matthew Lee Clingerman "Development and Modelling of electrically conductive composite material" Ph.D. thesis, Michigan Technological University, 2001.
(b) ZHENG Jing, SONG Wen-juan, MA Guang, JIA Zhi-hua, "Thermal expansion and mechanical properties of Al/Si composites fabricated by pressure infiltration", *Trans. Nonferrous Met. Soc. China*, vol. 17, p 326-329, 2007.
(c) Gupta, V.K., et al., Chromium removal by combining the magnetic properties of iron oxide with adsorption properties of carbon nanotubes, *Water Research*, 2011.
(d) A Lj Bojic, M Purenovic and D Bojic "Removal of chromium (VI) from water by micro-alloyed aluminium composite (MAIC) under flow conditions" *Water SA* Vol. 30, No. 3, p 353-359, 2004.
(e) Hossein Eisazadeh "Removal of Arsenic in Water Using Polypyrrole and its Composites" *World Applied Sciences Journal*, vol. 3 (1), p 10-13, 2008.
(f) Mahoko Murakami, "Surface properties of an indirect composite polymerized with five laboratory light polymerization systems," *Journal of Oral Science*, Vol. 51, No. 2, p215-221, 2009.
(g) Qingfeng Tan, Xiaou Bao, Tengchun Song, Yu Fan, Gang Shi, Baojian Shen, Conghua Liu and Xionghou Gao, "Synthesis, characterization, and catalytic properties of hydrothermally stable macro-meso-micro-porous composite materials synthesized via in situ assembly of preformed zeolite Y nanoclusters on kaolin" *Journal of catalysis*, vol.251,1, p 69-79, 2007.
(h) Shuilin Zheng et al., "Preparation and Photocatalytic Property of TiO_2 /Diatomite-Based Porous Ceramics Composite Materials" *International Journal of Photoenergy*, Article ID 264186, 2012.
- (i) L.C.Davis, J. Chen and M.F.Thorpe, "Predicting the elastic properties of Composite materials", *Proceedings of the American society for composites-seventh technical conference*, oct.13-15, 1992.
- [3] C. Cannas, D. Gatteschi, A. Musinu, G. Piccaluga, C. Sangregorio, "Structural and Magnetic properties of Fe_2O_3 Nanoparticles Dispersed over a Silica Matrix", *J. Phys. Chem. B*, vol. 102, p7721,1998.
- [4] C. Cannas, G. Concas, A. Musinu, G. Piccaluga, G. Spano, "Mossbauer spectroscopic study of Fe_2O_3 nanoparticles dispersed over a silica matrix", *Naturforsch.*, vol.54a, p513, 1999.
- [5] D. D. Awschalom, D. P. DiVincenzo, "Complex Dynamics of Mesoscopic Magnets", *Phys. Today*, vol. 48, 4,p 43 1995.
- [6] R. F. Ziolo, E. P.Giannelis, B. A. Weinstein, M. P. O'Horo, B. N. Ganguly, V. Mehrotra, M.W. Russell, D. R. Huffman, "Matrix-Mediated Synthesis of Nanocrystalline $\alpha\text{-Fe}_2\text{O}_3$: A New Optically Transparent Magnetic Material", *Science*, vol. 257, p219, 1992.
- [7] R. D. McMichael, R. D. Shull, L. J. Schwartzendruber, L. H. Benett, R. E. Watson, "Magnetocaloric effect in superparamagnets" *J.Magn.Magn.Mater.*, vol. 111,29, 1992.
- [8] T. Ida, H. Tsuki, A. Ueno, K. Tohoji, Y. Udagawa, K. Iwai, H. Sano, "characterization of iron oxide in Fe_2O_3 SiO_2 catalyst" *J. Catal.*, vol. 106, p428 ,1987.
- [9] C. Cantalini, M. Pelino, "Microstructure and humidity-sensitive characteristics of $\alpha\text{-Fe}_2\text{O}_3$ ceramic sensor" *J.Am. Ceram. Soc.*, vol.75, p546, 1992.
- [10] C. Cantalini, H. T. Sun, M. Faccio, G. Ferri, M. Pelino, "Niobium-doped $\alpha\text{-Fe}_2\text{O}_3$ semiconductor ceramic sensors for the measurement of nitric oxide gases" *Sensors and Actuators B*, vol.24-25, p673 1995.
- [11] H. T. Sun, C. Cantalini, M. Faccio, M. Pelino, M. Catalano, L. Tapfer, "Porous silica-coated $\alpha\text{-Fe}_2\text{O}_3$ ceramics for humidity measurement at elevated temperature" *J. Am. Ceram. Soc.*, vol.79, p927, 1996.
- [12] S. Roy, D. Das, D.Chakravorty, D. C. Agrawal, "Magnetic-properties of glass-metal nanocomposites prepared by the sol-gel route and hot-pressing" *J. Appl. Phys.* Vol.74, p4746, 1993.
- [13] Le Zeng, "A method for preparing silica-containing iron (III) oxide adsorbents for arsenic removal" *Water Research*, vol.37, p 4351-4358, 2003.
- [14] T. Tago, T. Hatsuta, K. Miyajima, M. Kishida, S. Tashiro and K. Wakabayashi, "Novel Synthesis of Silica-Coated Ferrite Nanoparticles Prepared Using Water-in-Oil Microemulsion," *Journal of the American Ceramic Society*, Vol. 85, No. 9,p. 2188-2194, 2002.
- [15] A.-L. Morel, S. I. Nikitenko, K. Gionnet, A. Wattiaux, J. Lai-Kee-Him, C. Labrugere, B. Chevalier, G. Deleris, C. Petibois, A. Brisson and M. Simonoff, "Sonochemical Approach to the Synthesis of Fe_3O_4 @ SiO_2 Core-Shell Nanoparticles with Tunable Properties," *ACS NANO*, Vol. 5, No. 2, p. 847-856,2008.

- [16] Z. I. Lei, Y. L. Li and X. Y. Wei, "A Facile Two-Step Modifying Process for Preparation of Poly (SStNa)- Grafted $\text{Fe}_3\text{O}_4/\text{SiO}_2$ Particles," *Journal of Solid State Chemistry*, Vol. 181, No. 3, p. 480-486, 2008.
- [17] Meizhen Gao, Wen Li, Jingwei Dong, Zhirong Zhang, Bingjun Yang " Synthesis and Characterization of superparamagnetic $\text{Fe}_3\text{O}_4@/\text{SiO}_2$ Core-Shell Composite Nanoparticles" *World Journal of Condensed Matter Physics*, vol. 1, p49-54, 2011.
- [18] C. Z. Huang and B. Hu, "Silica-Coated Magnetic Nanoparticles Modified with Gamma-Mercaptopropyltrimet hoxysilane for Fast and Selective Solid Phase Extraction of Trace Amounts of Cd, Cu, Hg, and Pb in Environmental and Biological Samples Prior to Their Determination by Inductively Coupled Plasma Mass Spectrometry," *Spectrochim Acta Part B: Atomic Spectroscopy*, Vol. 63, p. 437-444, 2008.
- [19] Surender Duhan & Sunita Devi "Synthesis and Structural characterization of Iron Oxide-silica Nanocomposites Prepared by the Solgel Method" *International Journal of Electronics Engineering*, vol.2(1), p. 89-92,2010.
- [20] Zulfikar, M.A., A.W. Mohammad and N. Hilal. Preparation and characterization of novel porous PMMA- SiO_2 hybrid membranes. *Desalination* vol.192,p262-270,2006.
- [21] S. Duhan, P. Aghamkar, M. Singh, N. Kishore, P. K. Sen, *J. Sol-Gel Sci Tech.*, vol.46, 17,2008.
- [22] Mohammed Jasim uddin, Muhammed Shah Miran and Mohammad Yousuf Ali Mollah "Electrochemical Synthesis and characterization of iron oxyhydroxide" *Journal of the Bangladesh Chemical Society*, vol. 20(1),p39-45,2007.
- [23] Piccaluga G., Corrias A., Ennas G., Musinu A., "The Sol-Gel Method: Choice of the experimental conditions", *Materials Science Foundations*, Vol.13
- [24] Meera Basa, "Synthesis & Characterization of Silica Coated Iron oxide Nanoparticles by Sol-Gel Technique" M.Sc. Thesis, National institute of Technology, Rourkela 2007.
- [25] Lenza. R.F.S., Vasconcelos W.L. "FT-IR of Silicon (IV) oxide" *Mat. Res.*, vol. 4(3), 2001.
- [26] Tabatabaei,S., Shukohfar, A., Aghababazadeh, R., Mirhabibi, A., "Experimental study of the synthesis and characterisation of silica nanoparticles via the sol-gel method" *Journal of Physics: Conference Series* 26, p 371–374,2006.
- [27] Raileanu, M., Crisan, Petrache C., Crisan D., Zaharescu M. " $\text{Fe}_2\text{O}_3\text{-SiO}_2$ Nanocomposites obtained by different sol-gel routes" *Journal of Optoelectronics and Advanced Materials* Vol. 5, No. 3, p. 693 – 698 ,2003.
- [28] Qingyin Wu, "Preparation and proton-conductibility of silica gel containing 64 wt.% undecatungstocobaltoaluminic heteropoly acid", *Materials Letters*, vol. 56 , p19– 23,2002.
- [29] Balek,V.,Subrt,J., " Thermal behaviour of iron (III) oxy hydroxide", *Pure & App. Chem.*, Vol. 67, No. 11, p 1839-1842, 1995.
- [30] Guido Ennas, Maria F. Casula, SergioMarras, Gabriele Navarra, Alessandra Scano, and GiaimeMarongiu "Characterisation of FeOOH Nanoparticles and Amorphous Silica Matrix in an FeOOH- SiO_2 Nanocomposite" *Journal of Nanomaterials* Vol.2008.
- [31] Palomares, S.A., Anchez, S., Ponce-Castaneda, S. , Mart_inez, J.R., Facundo Ruiz, Yurii Chumakov, Dom_inguez, O. , "Quantitative analysis of iron oxide particles Embedded in an amorphous xerogel matrix" *Journal of Non-Crystalline Solids*,vol.325,p251– 257, 2003.