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Synthesis, characterisation, design and study of magnetorheological property of nano fe$_2$O$_3$

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Abstract: Nano -Fe_2O_3 was synthesized by precipitation method using starch as the precipitating agent. The characterization of the samples by XRD (X-ray diffraction) technique confirmed the formation of Fe_2O_3..Analysis by SEM (scanning electron microscope) was carried out to study the morphology and particle size. The SEM image shows that the particles are spherical in nature. The as prepared samples contained rhombohedral phase. MR fluid was prepared using Fe_2O_3 nano powder, silicone oil and grease. The aim of the work was to determine the viscosity of the MR fluid, under the influence of different values of electromagnetic field. To determine the viscosity and damping coefficient a modified experimental setup was fabricated.
Keywords: Iron oxide, Rheological property, Viscosity,Grease.

Introduction

Magnetorheological (MF) fluids are class of smart fluid, which on response to an applied magnetic field exhibitsa change in their rheological properties [1]. Suspensions of ferromagnetic particles in a career fluid are MR fluids.Conventional viscosity based system can be easily replaced by a MR fluid [2].The main characteristics of MR fluid are fast response, simpleinterface between electrical power input and the mechanical power output, controllability and integration in complex system.MR fluids are changing from solid to liquid state and vice versa by the application of magnetic field whichemphasiswide applications in devices such as isolators, shock absorbers, clutches, engine mounts, alternators, power steering pumps, control valves, brakes and dampers etc[3-5].Effective change in the viscosity of the fluid to several orders of magnitude have created an curiosity to the researchers on MR fluid using nanoparticles. The most important problems of MR fluids are sedimentation and incomplete chain formation in response to magnetic field [6].The properties of the MR fluid are affected by various parameters such aspH, surfactants, solid content, viscosity and the size of the particles [7].Among these factors particle size was the major factor which influences the rheology of ferrofluids[8].

Iron oxides such as α-Fe_2O_3, γ-Fe_2O_3, β-Fe_2O_3 and $Fe_3O_4$are of great importance because of their potential applications [9].Various methods have been explored to prepare nano iron oxides such as coprecipitation, microemulsion, sol-gel,hydrothermal etc[10,11,12,13]. The present work focussed on synthesizing nano iron oxide by simple coprecipitation method in an alkali medium using starch as stabilizing agent.

Phule discussed the viscosity of the fluid at zero magnetic field and the sedimentation of the particles by using large particle size as the key points [7].Daniela et al has found enhanced relative viscosity with addition of nanoparticles as compared to conventional MR fluid [10].Bong et al used nanosized carbonyl iron particles in MR fluid and improved the yield behaviour [9]. Morozov found that on increasing the intensity of the magnetic field the viscosity of the ferrofluid increases [14].

This paper throws a light on the preparation of the nano $Fe_2O_3$by applying as magnetorheological fluid and investigating its response to applied electromagnetic field. An apparatus was designed and fabricated to determine the coefficient of viscosity.

Experimental

Materials

$FeCl_3$ $6H_2O$ (98 %), $FeCl_2$ $4H_2O$ (99 %), Sodium hydroxide (99%) and Starch (99.8%)were purchased fromSd fine Ltd and used. Distilled water was used as asolvent.

Synthesis of Fe_2O_3 nanoparticles

The procedure for preparing Fe_2O_3nanoparticles byco-precipitation method was as follows.$FeCl_3$ $6H_2O$ and $FeCl_2$ $4H_2O$ with a stoichiometric ratio of1:2 was dissolved in 50 ml distilled water and kept for heating at 60^0C. Starch solution of concentration 5% by weight was added to the mixture. Then, ammonium hydroxide was added drop wise to the solution heated at 80^0C with strong stirring. Just after the addition of ammonium hydroxide, the colour of the solution changed from light brown to dark black indicating the formation of Fe_2O_3 nanoparticles, which was allowed to homogenize by stirring for 10 minutes and kept for drying at 80º C for 4 hrs. The obtained powder was washed several times with water, dried and characterised.

Preparation of the MR fluid

The MR fluid was prepared by dispersing nano iron particles (Nano Fe_2O_3-38g) in a career fluid. Silicone oil (60ml) anorganic liquid was used as career fluid. The prepared MR fluid was agitated with mechanical stirrer for 4hrs. To avoid the sedimentation of the nano iron particles and to increase the viscosity, an additional component grease (2ml) was added as an additive.

Characterization

The nanopowder was characterised by Powder X-ray diffraction (CuKα, PANalytical) at room temperature. The average particle size of the iron oxide formed was determined using Debye- Scherer formula. Scanning Electron Microscopic analysis of the se sample was carried out using FEI QUANTA FEG 200 HR Scanning Electron Microscope. To determine the average size of the dispersed iron oxide particles suspended in the fluid, MR Fluid sample was analysed by optical scanning microscope.

MagnetoRheometer set up

Rheological properties of MR fluid sample are examined with a flow mode rheometer fabricated from a modified damper using a sinusoidal input dynamometer over a speed range of 12.7 to 177.8 mm/s (0.5 to 6 in/s) and an input current range of 0 to 2 A. The yield stress and plastic viscosity of the MR fluid were characterized using a Binghamplastic model.

Results and discussion

Preparation and Characterisation of nano Fe_2O_3

The X-ray powder diffraction pattern of the sample [Fig.1] was recorded on a *Bruker D8 Advance* diffractometer using CuKa (1.5406 Å).All the peaks appearing in the X-Ray diffraction patternare indexed based on Fe_2O_3 (JCPDS file # 89-8104).The analysis of the diffraction pattern showed the formation of rhombohedra phase. The average crystallite size of the powder was calculated using Debye-Scherrer Equation

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

(Where D is the crystallite size, λ is the wavelength of X-ray, β is the value of FWHM and θ is the Bragg's angle) and found to be around 20 nm. The surface morphology of the prepared sample was studied using scanning electron microscope, as shown in Fig.2. It is evident from the SEM images that the particles are spherical in nature and the average size of iron oxide particles are about 100-500nm respectively. The size and the shape of nanoFe_2O_3 are strongly dependent on the preparation technique [10].For the determination of the average size of the dispersed iron oxide particles suspended in the fluid, MR Fluid sample was analysed by optical scanning microscope.Figure.3 shows the optical image of the M R Fluid sample observed by Optical Scanning Microscope. The iron particles have found to be of regular shapes. The average size of iron particles is found to be about 100 nm.

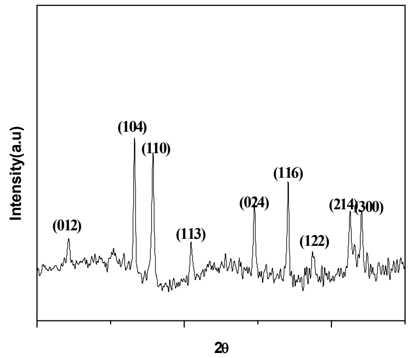

Fig.1. X-ray diffraction pattern of Fe_2O_3 nanoparticles

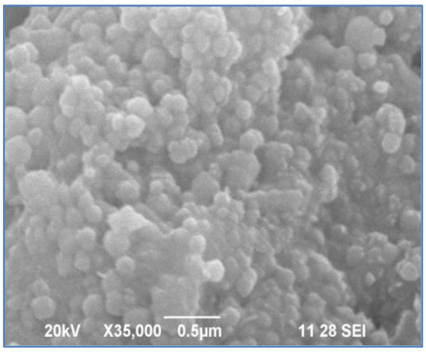

Fig.2. SEM image of Fe_2O_3 nanoparticle

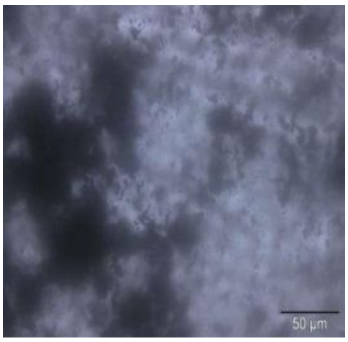

Fig.3. Optical Scanning microscope image of Fe_2O_3 nanoparticle

Magneto rheological property of MR fluid

The magnetorheological property of the MR fluid are studied by the modified experimental setup as shown in the figure.1

The viscosity of the prepared MR fluid was studied both in the presence and absence of current. Aknown amount of MR fluid (10g) was poured in to the beaker, with the outlet closed. By opening the outlet, the time taken for the flow was noted and the experiment was repeated thrice to have an average value. The observed results are shown in the Table1.

Table 1: Effect of Current Vs Viscosity

S.No	Current (Amps)	Time (sec)	Mean (sec)	VISCOSITY (Ns/m2)
1	Without current	137.43 120.88 114.31	124.20	14.91
2	0.02	122.24 130.30 129.28	127.27	15.28
3	0.04	126.80 131.01 135.38	131.06	15.74
4	0.06	122.12 134.35 155.18	137.21	16.47
5	0.08	184.31 157.52 227.50	189.77	22.79
6	0.10	291.29 288.08 210.02	263.13	31.60

Efffect of current vs viscosity

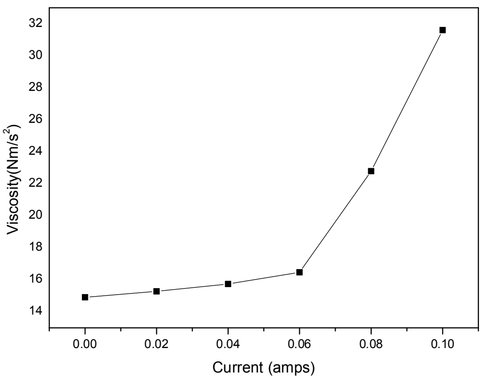

Fig.4.Effect of Current Vs Viscosity

The presence of current plays an important role in the viscosity of MR fluid. In order to understand the influence of current, the experiments were carried out by varying the current from0.02 to 0.10in amps. The Viscosity of the prepared MR fluid are calculated using the equation $\eta = \rho * t/(\rho_{g*}t_g) * \eta_g$. On increasing the current, the magnetic induction and magnetic flux of the coil are increased which attracts the prepared MR fluid and increases the viscosity. In the figure.4 we observe the increase in viscosity of the prepared MR fluid on increasing the current. The results of the experiments show that the viscosity of the prepared MR fluid was greater hence bigger magnetorheological effect.

Conclusion

In this study, synthesis of nano- Fe_2O_3 and their rheological properties insilicone oil and grease have been investigated. We then determined the viscosity of MR fluid at different values of electric field. The results reveal that the magnetic property of the MR fluid increases by applying the current, which in turn increases the viscosity of the nano-Fe_2O_3 Fluid. It is shown that even small electric field can make noticeable changes in viscosity of the MR fluid. Thus this nano MR Fluid gives out the good viscosity which can be used as an alternative damper fluid in automobiles.

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