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Preparation and characterization of nano magnetic fluid for automotive applications

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ABSTRACT

Purpose: of this paper is to prepare a nano magnetic fluids with nano additives to have the combined characteristics of high yield stress and better magnetic properties for smart vehicles. This study focuses on increasing the sedimentation time of the fluid using suitable nano additive nickel along with graphene as fillers.

Design/methodology/approach: Magnetic nano sized nickel particle based electro-magneto-rheological fluid was prepared and graphene nanoparticle with thickness less than 10nm was introduced as an additive to reduce its sedimentation. This added plate like graphene acts as filler which seals the interfaces of nickel particles and thereby it improves the resistance to sedimentation. Triton X 100 was added as the surfactant for the fluid to reduce the agglomeration of the particles.

Findings: Morphology of pure nickel and graphene were examined using scanning electron microscopy (SEM) images.

Research limitations/implications: The important limitations is that freely dispersed micron sized iron particles could settle over a period of time, in the form of cakes at the bottommost, and it is tedious to recuperate as dispersed phase. In this investigation, nano sized nickel particles were used as additive to reduce the sedimentation of micron sized iron particles so that, the mixture is homogeneous for extended period of time. In future, addition of different types composite additives in the magnetorheological fluid could be made for the better sedimentation control.

Practical implications: The sedimentation problem is one of the major drawback in the smart fluids, which can be eliminated by adding nano particles. For conventional fluid, the complete sedimentation will occur in 2 hours while the improved nano magnetic fluid with additive has good resistance to settle the micron sized iron particle up to 10 hours.

Originality/value: To prepare a low cost magnetorheological fluid with nano additives like nickel particles along with fillers as graphene nano particles. With this addition of nickel and inclusion of graphene, the sedimentation problem in magnetorheological fluids is significantly reduced. This magnetorheological fluids can be used in brakes and dampers of automobiles.

Keywords: Nano magnetic fluid, Yield stress, Nickel nano particles, Viscosity, Sedimentation rate

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MATERIALS

1. Introduction

Now a days, wide spectrum of studies were being done by researchers on nanomaterials, as they are having potential applicability in many areas such as automotive, biological, healthcare and almost in all distinct engineering environments. For any commercial implications of nano based magnetic fluid technologies, the material could be synthesized through a logical, very less cost and constraint manner, to get nanoparticles of desired size, composition with better properties and functionalities. The developed nanoparticles can be easily controlled with an aid of magnetic field for effective utilization of the same in any modern magnetorheological fluid technology [1,2].

The applications of magnetic rheological fluids have been greatly restricted due to agglomeration and sedimentation of magnetic particles dispersed in the carrier fluid [3].Both theoretical and experimental studies were made explicitly on magnetorhelogical fluids for its characteristics and its applications. In magnetic rheological fluids, the stability against sedimentation and higher saturation magnetization for better utilization are two important considerations. It is very difficult to achieve improvement in all these critical properties simultaneously. With recent development in synthesis of nanoparticles, based on the requirement, it is possible to overcome the shortcomings in magnetorhelogical fluids. Non uniform mixing, sedimentation in the carrier fluids are critical, which can be greatly reduced with magnetic nanoparticles as additive and fillers. This additives and fillers acts as the seal have ability to have dispersion of carrier fluids over micron sized iron particles [4,5]. Graphene exhibits exceptional physical, chemical properties and the oxides of graphene are highly recommended in super capacitor electrodes. The presence of graphite oxide improves the electrical conductivity which were confirmed by SEM, TEM and Raman analysis [7,8]. In addition to that, an analysis on viscoelastic properties was also made to make use of magnetorhelogical fluids in dampers during its operation [9]. A mathematical formalization was also studied to know the operational condition of the devise on shock absorbers [10]. XRD, FTIR and AFM were used predominantly to characterize the prepared nano particles using particle size analyser [11].

Non-uniform mixing that are manifest when nano sized particles are used. Nowadays magnetic rheological fluids are becoming increasingly important in applications concerning active control of vibrations, isolators, power steering pumps, control valves, clutches, brakes and dampers have been designed and used. The studies on synthesis of magnetic nano particles and its characterization were made, and the effective utilization of the modern magnetorhelogical fluids is investigated. The synthesized particles were mixed to form MR fluids. Nano sized magnetite particles with silicone oil. Magnetic Rheological fluids with different concentrations were prepared.

2. Preparation of nickel by chemical reduction method

2.5 g of nickel acetate in 60 ml of distilled water is heated to 50°C and then 1.1 ml of hydrazine hydrate liquid is added with vigorous stirring. The solution is then heated to 65°C, which results in a light violet precipitate. 3.18 g of solid sodium carbonate is added with thorough stirring to the somewhat cooled solution. To obtain the spherical nano particles, the solution is then further heated to 100°C on a hot plate, the product is nano sized nickel particles. The precipitated particles are isolated by filtration followed by washing with liberal amounts of distilled water. The yield of the overall synthesis is 60% base on the amount of nickel acetate. The formed black nickel nano particles are then dried at 40°C in an air oven overnight.

3. Preparation of nickel oxide

Nano crystalline NiO particles will be prepared by adopting the following procedure. Accurately weigh 5 g of pectin powder and dissolve in 60 ml of distilled water in a clean beaker. Weigh about 2.37 g of nickel chloride and dissolve in 20 ml of distilled water in another beaker. With gentle stirring, slowly add nickel chloride solution in to a pectin solution. During mixing, the contents should be stirred well and continue the process for about 30 minutes. On mixing the contents, slowly viscosity of medium is increases, indicates the formation of gel. after the addition, the reaction mixture is heated over water bath for about one hour. The obtained gel is then filtered, thoroughly washed with water in order to remove impurities like ammonium chloride and the other dissolved impurities. The purified gel is then taken on watch glass, further kept at 110°C for about 1 hour. Collect the dried gel in silica crucible, calcination it by keeping inside the muffle furnace at 600°C for about 1 hour. After cooling to room temperature, collect the NiO nano particles and stored in vacuum.

4. Preparation of graphene

Graphene is synthesized from graphene oxide. By adding hydrazine as reducing agent to graphene oxide, it yields graphene by eliminating oxygen contents of GO. 0.5 g of graphene oxide is taken in a beaker and it is mixed with 1 ml of hydrazine (N_2H_4).Then it is subjected to microwave treatment 50 W microwave power for 30 s. During the synthesis process, violent fuming was observed and the oxygen containing functional groups were reduced and produced graphene powder. A total of six samples were prepared for the characterization studies as shown in the Table 1.

Table 1.
Proportion of different fluid samples

Sample	Oil	Ni nano particles	Graphene	NiO nano particles	Tritonx 100	Ultrasonication
Sample 1	Silicone oil 38 g	0.3 g	0.2 g	_	_	15 min
Sample 2	Silicone oil 38 g	0.3 g	0.4 g	_	0.5 ml	15 min
Sample 3	Silicone oil 38 g	0.3 g	0.6 g	_	1 ml	15 min
Sample 4	Silicone oil 38 g	–	0.2 g	0.3 g	–	15 min
sample 5	Silicone oil 38 g	–	0.4 g	0.3 g	0.5 ml	15 min
Sample 6	Silicone oil 38 g	–	0.6 g	0.3 g	1 ml	15 min

5. Results and discussions

5.1. SEM image of nickel

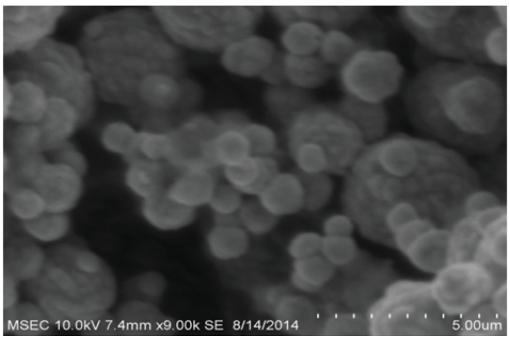

Fig. 1. SEM image of nickel nanoparticle

The morphology and the diameter of prepared nickel nano particles has been characterized by Scanning Electron Microscopy (SEM). Figure 1 shows spherical nano particles. The potential applied to the electron gun was 10 kV. The distance between the probe and the sample was 7.4 mm. The magnification used for observing the nano particles was about 9000x times, the original size. The size of the nickel nano particles is around 500 nm. From the image, it is inferred that maximum agglomeration of the Nickel nano particles is present.

5.2. XRD analysis of nickel

For metallic nickel nano particles, in the analysis of X-ray diffraction, strong peaks are to be observed at 2θ values of 43° and 51° according to the JCPDS reference 087-0712 (Fig. 2). The obtained two distinct peaks at 43.40° and 51.49° in the result shows the existence of metallic nickel nano particles. According to the Debye-Schrrer formula, the metallic nickel nano particles were found to be with a crystalline size of 33.08 nm.

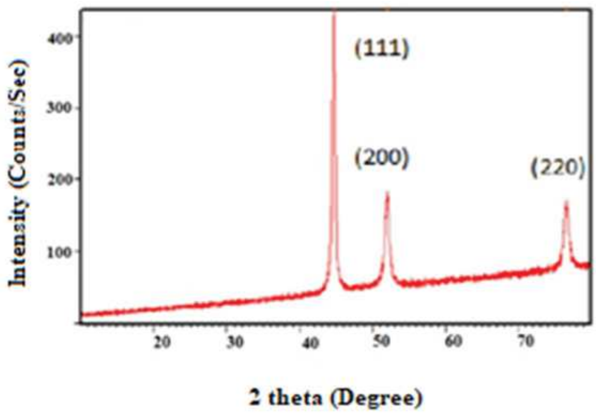

Fig. 2. XRD image of nickel

5.3. SEM image of nickel oxide

The morphology and the diameter of prepared nickel oxide nano particles has been characterized by Scanning Electron Microscopy (SEM). Figure 3 shows spherical nano particles and size of the nano particles is around 450 nm. The potential applied to the electron gun was 10 kV. The distance between the probe and the sample was 6.5 mm. The magnification used for observing the nano particles was about 9000x times, the original size. The size of the Nickel nano particles is around 500 nm. From the image, it is inferred that maximum agglomeration of the nickel oxide nano particles is present.

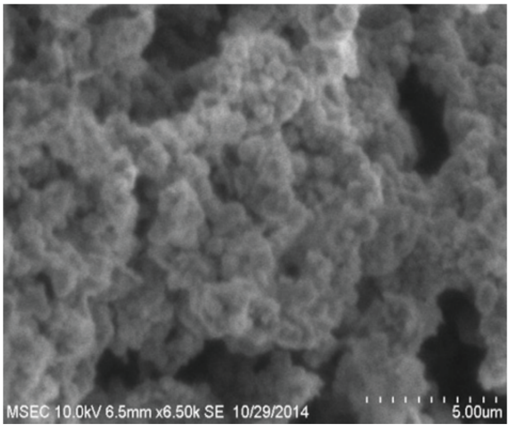

Fig. 3. SEM image of NiO nanoparticle

5.4. FTIR spectrum of nickel oxide

From FTIR spectra of Nickel oxide, the absorption peaks are significant in the region of 600-700 cm^{-1} which imitates the Ni-O stretches in vibration mode shown in Figure 4. The wideness in the absorption band conforms the presence of NiO powder as nanocrystals.

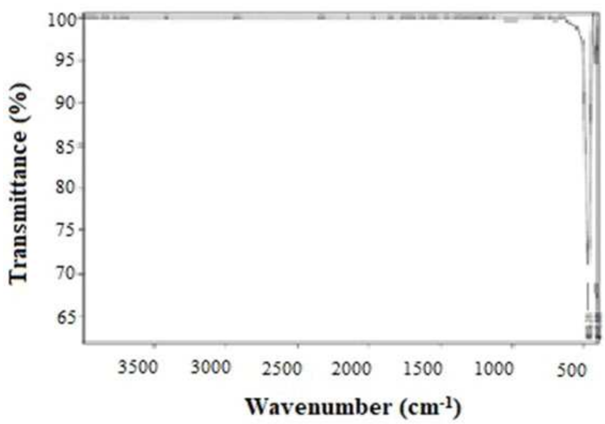

Fig. 4. FTIR image of nickel oxide

5.5. XRD analysis of nickel oxide

Figure 5 shows the X-ray diffraction grating of synthesized nickel oxide nano particles. Here, the purity of the synthesized nickel oxide and its crystallinity has been examined. The peak positions appearing at 2θ is 37.21°, 43.22°, 63.10°, 75.20°, and 79.39° can be readily indexed as (101), (012), (110), (113), and (006) which are the crystal planes with presence of bulk NiO. It is found that, the peaks of all diffraction grating are well matched with face centred cubic structure of nickel nanoparticles which are crystalline in nature (JCPDS, No. 04-0835). In addition to that, the result depicts the physical nature of the Nickel oxide nano particles of purity at highest level. The lattice constant of NiO manipulated from X-ray diffraction grating is found to be 4.1729 A°.

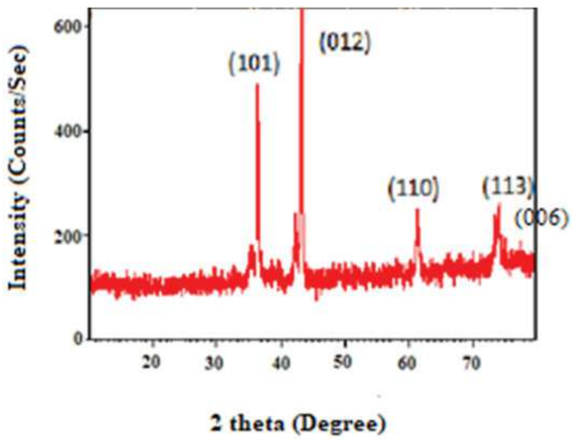

Fig. 5. XRD image of nickel oxide

5.6. SEM image of graphene

The dimension and surface morphology of prepared graphene has been characterized by Scanning Electron Microscopy (SEM). Figure 6 shows the SEM image of graphene, where it is clear that there is a continuous filament of nanographene is dispersed uniformly. This reveals the prevention in sedimentation on magneto-rheological fluid over longer period of time. The potential applied to the electron gun was 10 kV. The distance between the probe and the sample was 6.6 mm. The magnification used for observing the nano particles was about 9000X times, the original size.

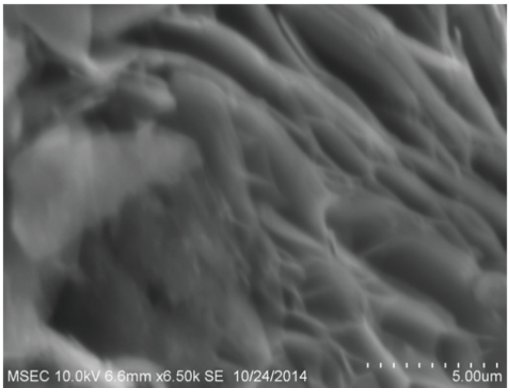

Fig. 6. SEM image of graphene

5.7. FTIR spectrum of graphene

FTIR image of graphene shows the clear transition of graphene oxide to graphene. This is evident from the disappearance of C-O and O-H bonds. A slight variation is seen at 1525 cm^{-1} which shows the presence of only C-C bond. This confirms the graphene formation using hydrazine as reducing agent.

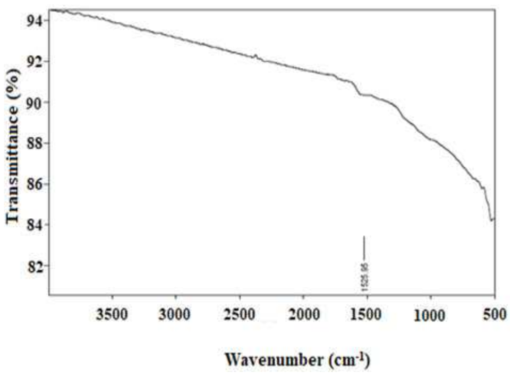

Fig. 7. FTIR image of graphene

5.8. XRD analysis of graphene

Graphene synthesized via microwave heat treatment method has a strong peak at 2θ = 25.91 with an interlayer distance of 3.57ºA which is closer to the interlayer distance of conventional graphene (Fig. 8). The crystal size is calculated using Debye-Scherrer formula:

$$D = \frac{0.9\lambda}{\beta cos\theta} \quad (1)$$

From Debye-Scherrer formula, graphene nanosheet, the crystal thickness is around 25 nm.

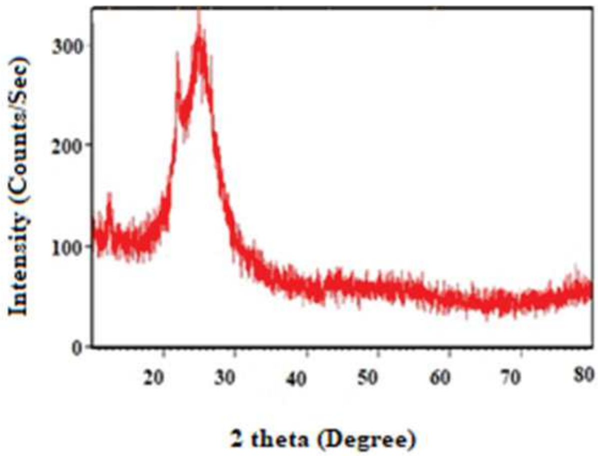

Fig. 8. XRD image of graphene

5.9. Viscosity measurement

The dynamic viscosities of the six different samples were measured using Brookfield viscometer DVT-3. The average dynamic viscosity values of magneto-rheological nano fluid were taken for convergence shown in the Table 2.

Table 2.
Viscosity measurement

SAMPLE	VISCOSITY
Sample 1	317 cP
Sample 2	287 cP
Sample 3	273 cP
Sample 4	253 cP
Sample 5	271 cP
Sample 6	398 cP

The graph plotted between the viscosity and time for three different oil based fluids are shown in the Figure 9. Here the sample 1 has higher viscosity compared to the other two samples. The above mentioned samples were made of transformer oil which naturally has less viscosity and higher thermal conductivity that are not suitable for automotive application. So the other three samples were again made using silicone oil which gives more viscosity.

5.10. Sedimentation ratio of silicone oil based fluid

Figure 10 shows the graph for sedimentation ratio of different silicone oil based fluids. Here the graph reveals that the fluids which consists of Nickel and nickel oxide nanoparticles and graphene shows very good stability. The other fluids with absence of surfactant shows greater sedimentation ratio.

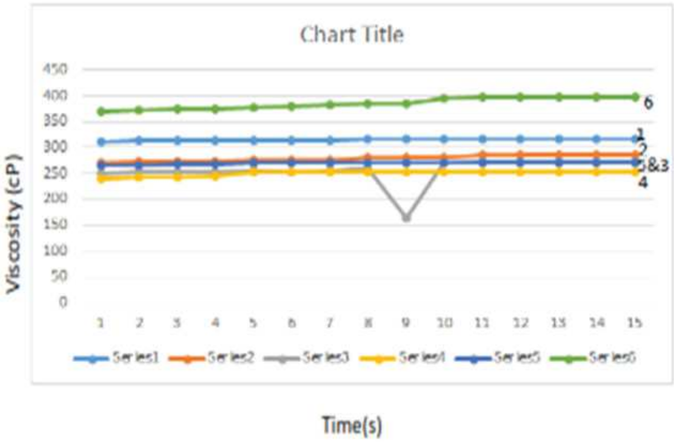

Fig. 9. Viscosity of silicon oil based nano magnetic fluid

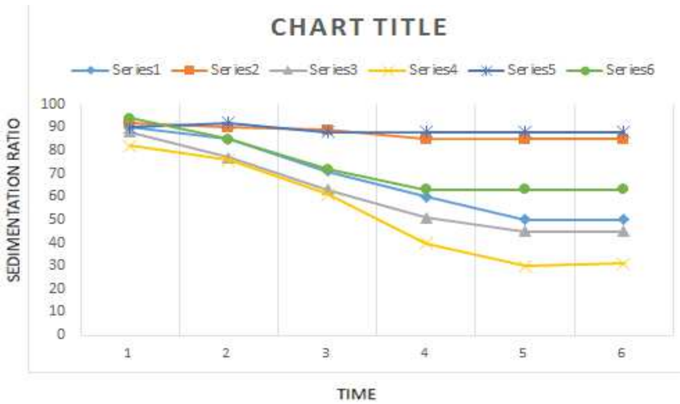

Fig. 10. Sedimentation ratio of different samples

6. Conclusions

In this study, six systems of nano magnetic fluids were prepared and compared with respect to the sedimentation characteristics based on magnetic particles and graphene additives. The successful addition of graphene nano particles among the magnetic particles was confirmed with SEM image. It was found from the experiment that, the addition of novel Graphene additives does not changes the properties of magnetorheological nano fluid, even at higher magnetic flux densities. It also rectified the serious sedimentation problem in the system. By the way, Sample of viscosity 271 cp is considered to be the best fluid due to higher sedimentation time, constitutes 0.4 gm of graphene and 0.3 g of nickel. Here, synthesis of nano particles and preparation of magnetorheological nano fluid has been successfully made and the best fluid for braking system was found by using experimental studies with higher braking torque.

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