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Experimental investigation the effect of nanoparticle concentration on the rheological behavior of paraffin-based nickel ferrofluid

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## ABSTRACT

The effect of Ni nanoparticles concentration on the rheological and magneto-rheological properties of Ni ferrofluid (FF) in liquid paraffin base were investigated. The FFs were prepared using a high energy milling. A high-energy mill was used to prepare these Ni based ferrofluids. By measuring the peak broadening of the (111) crystal plane for Ni from the X-ray diffraction spectrum of the dry nanoparticles, the mean crystallite size was 38 nm. Regardless of nanoparticle concentration, the fluids were found to have non-Newtonian shear thinning behavior. At a constant shear rate, the viscosity of the nanofluids increased as the Ni nanoparticle concentration was increased. This experimental data was then compared to existing models; none of which adequately described the measured rheological properties of the suspensions. When an external magnetic field was applied at a constant shear rate, the viscosity increased as the field strength was increased further. It is thought that as the magnetic field strength is increases, larger flocculated nanoparticle structures form within the fluid. Although there are larger flocculants, there are fewer of them resulting in a decrease in the viscosity of the suspension. © 2015 Elsevier Ltd. All rights reserved.

### 1. Introduction

A ferrofluid is a stable colloid of magnetic nanoparticles such as  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> [36], Fe<sub>2</sub>O<sub>3</sub> [22], Fe<sub>3</sub>O<sub>4</sub> [17] and [13], CoFe<sub>2</sub>O<sub>4</sub> [27], Mn–Zn [30], Co–Zn [29], and Li<sub>2</sub>O · Fe<sub>2</sub>O<sub>3</sub> [43] in a base liquid. The liquid base can be polar or non-polar [31], but the nanoparticles must be coated with a capping agent to prevent agglomeration of the individual particles [35]. The coating permits these nanofluids to be stable even in high-gradient magnetic fields [16]. This type of nanofluid is commonly used in the field of medicine and in industrial applications (e.g., drug targeting using magnetic nanoparticles; contrast enhancement in MRI; magnetic separation of cells; dynamic sealing, heat dissipation, inertial & viscous dampening applications, sensors, optics industries; etc.) [9,11,18,33,34].

Rheology is the study of how a fluid or soft solid flows when external forces are applied. The rheological and magnetorheological properties of nanofluids are an important, fundamental area of research which impacts our understanding and ability to use these materials. The most important problems of ferrofluids are sedimentation and incomplete chain formation in response to magnetic field [41]. The properties of the ferrofluid are affected by various parameters such as pH, surfactants, solid content, viscosity and the size of the particles [15]. Among these factors particle size was the major factor which influences the rheology of ferrofluids [26].

The rheological properties of various magnetic nanofluids have been previously studied and, in general, they exhibit a shearthinning behavior (i.e., their viscosity decreases with increasing shear rate) [10]. As the intensity of an externally applied magnetic field is increased, the viscosity of these fluids increases [20]. Recently, it has been suggested that experimentally observed magneto-viscous effects are due to nanoparticle chaining and aggregation in the presence of strong magnetic fields which affect the macroscopic properties of the fluid, even at low nanoparticle concentrations [2,4,5,24,37]. The rheological properties of a nanofluid are heavily impacted by particle-to-particle interactions within the fluid, which depend on the size and size distribution of the particles [8,38]. The complex nature of particle-to-particle interactions makes theoretical analysis difficult; these interactions are not even considered in the most commonly used models [6,7,23]. There are scanty literature to show the magneto-viscous effect of Ni ferrofluids. In this work, the Ni ferrofluid was prepared

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from an easy method. The effect of the nanoparticles concentration on the rheological and magneto-viscous properties of Ni ferrofluids in a liquid paraffin base, and the effect of agglomerated structures on the magneto-viscous effect has been investigated and discussed.

#### 2. Experimental

# 2.1. Materials

A magnetic Ni nanoparticle powder with a mean particle size less than 100 nm was obtained from Aldrich and pure liquid paraffin was provided by Arman Sina company of Iran. Oleic acid, which was used to cap the nanoparticles, was obtained from Merck. All of the chemicals used in this studied were analytical grade and were used as delivered.

#### 2.2. Preparation of the nanoparticle suspensions

Nanofluids were prepared by mixing 20 g of dry Ni nanoparticle powder with 50 cc of oleic acid and milled in a Fritsch Pulverisette planetary mill for 5 h. These suspensions were then centrifuged (Centurion Scientific Ltd., UK) at 12,000 rpm to separate uncapped, agglomerated Ni from the colloidal suspension of nanoparticles. The nanofluid suspensions with a variety of concentrations were made by: (1) diluting the colloid with an appropriate amount of liquid paraffin, (2) mixing the diluted colloids with a homogenizer (Weiggenhauser D130, Germany), and (3) putting colloids in a Fritsch ultrasonic bath (model: laborette 17) for 5 min. Using this method, stable colloids containing 5, 10, 20, 30 and 40 wt% of Ni nanoparticles capped with oleic acid in liquid paraffin were made.

# 2.3. Characterization of the dry Ni nanoparticles and the prepared nanoparticle suspensions

#### 2.3.1. X-ray diffraction (XRD) analysis of the dry Ni nanopowder

A Philips PW 1800 X-ray diffractometer equipped with a copper source and nickel filter ( $\lambda = 1.5418$  Å) was used to obtain the XRD spectrum for the dry nanoparticle powder. The sample was scanned in a continuous mode from 5° to 90° with a scanning rate

of 0.02° per second. The crystallite size of the nanoparticles was calculated using the following [28]:

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

where *D* is the average crystalline size,  $\lambda$  is the X-ray wavelength,  $\beta$  is the angular line width of half-maximum intensity, and  $\theta$  is the Bragg angle in degrees. Fig. 1 shows the X-ray powder diffraction spectrum of the dry Ni nanopowder. All of the peaks in this figure correspond to crystalline Ni. The slight background signal indicates that the Ni was likely developed from an amorphous phase. The angular line width of the half-maximum intensity of the (111) was measured to be 0.38, which indicates that the mean particle size is 38 nm. This value corresponds to the size reported from the supplier (i.e., <100 nm).

# 2.3.2. Magnetometer results from the dry Ni nanopowder

An Alternative Gradient Force Magnetometer (AGFM: model 155) was used to determine the hysteresis curve for the dry Ni nanopowder at room temperature. In Fig. 2, the magnetic hysteresis curve for the dry Ni powder is shown. The curve from this cycle shows that the magnetization is quite small. A constant magnetization value of 40 emu/g is reached when the applied field is greater than 4 kOe (or -40 emu/g below -4 kOe). This indicates that the nanoparticles have attained their saturated magnetization level at these field strengths and suggests that the nanoparticles are not super-paramagnetic, but are of a single domain type.

#### 2.3.3. Dynamic light scattering results for the prepared nanofluids

The hydrodynamic size distributions of the nanoparticles/ aggregates in the nanofluids were measured using a Malvern 3000 Dynamic Light Scattering (DLS) system. Fig. 3 shows the hydrodynamic diameters of the 40 wt% Ni nanofluid; this figure is representative of the other nanoparticle concentrations. The mean hydrodynamic diameter is 248 nm and diameters range from 175 to 350 nm.

In ferrofluids there is often a dipole–dipole interaction between individual magnetic particles [38] and it is likely that the nanoparticles have formed flocculated structures in the suspension. Since



Diffraction angle (degree)

Fig. 1. XRD pattern of Ni nanopowder.

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