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Elucidation of Physical Structural Stability of Nickel Nanoparticles Additive-Based Magnetorheological Grease

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Abstract

This study investigates the influence of nickel nanoparticles on the physical properties and structural stability of magnetorheological grease (NMRG) as a smart composite. The incorporation of nickel nanoparticles aims to mitigate issues commonly observed in conventional magnetorheological grease (MRG), including particle aggregation within the fibrous matrix, limited material performance, and deformation instability. NMRG samples containing 1–5 wt% nickel nanoparticles were prepared using a mechanical stirring method. The tests focused on physical properties, subjected to rheological analysis, chemical compound identification, and wettability characterizations. The results revealed that the NMRG exhibited enhanced hydrophobicity, increasing by approximately 8.4% with higher nanoparticle content, while no detectable changes occurred in the chemical composition of the compound. This indicates that the inherent chemical bonds in the grease dominate over the physical adsorption process. The inclusion of nickel nanoparticles also led to notable improvements in magnetic and rheological performance, characterized by higher viscosity, greater energy storage capability, an extended linear viscoelastic (LVE) range, and reduced damping behavior. These outcomes confirm that the incorporation of nickel nanoparticles enhances the physical properties and structural stability of NMRG, particularly in terms of elasticity and deformation resilience. The findings provide insight into how nanoparticle-induced surface modifications can significantly influence the performance of magnetorheological materials for advanced engineering applications.

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Keywords: Magnetorheological grease, nickel nanoparticles, physical properties, smart composite, structural stability

INTRODUCTION

Magnetorheological (MR) materials, a new class of smart composites, have attracted the attention of researchers in the medical device field due to their tunable rheological properties and promising high performance. Extensive studies related to the prototype of rehabilitation devices integrated with MR fluids have been developed [1]. However, due to the drawbacks of the MR fluids, such as sedimentation and leakage, MR grease (MRG) was introduced as a replacement [2]. The study found that the thixotropic properties of grease used as a medium in MRG can prevent the magnetic particles from depositing at the bottom by increasing the initial viscosity without exerting force on the material. As the external magnetic force is induced into the MRG, the magnetic particles will align along the direction of the induced fields, creating a chain-like structure in the grease medium. This formation enhances the MRG's performance, including material deformation, stability, strength, and energy storage.

Building upon these advancements, recent studies have focused on improving the magnetic properties of MRG by adding solid and liquid additives. Kim et al. studied the effect of dilution oil as a liquid additive on the MRG by using kerosene [3]. The result reported that the initial viscosity decrease aligns with material performance. The addition of dilution oil in the MRG helps to ease the particles' alignment during the applied magnetic field and simultaneously reduces the internal friction. The study proved the dynamic yield stress of MRG is dependent on the content of silicone oil as well as particle size. The results showed that the higher yield stress, 65.96 kPa was achieved [4]. In fact, Pang et al. found that the stability of the grease after a period of storage declined when kerosene was added, compared to hydraulic oil [5]. Besides, several solid additives, including magnetic and non-magnetic nanoparticles, have been studied by researchers. For instance, the utilization of nanoparticles such as chromium oxide (CrO2) [6], maghemite (Fe2O3) [7], graphite (Gr) [8], and cobalt ferrite (CoFe2O4) [9]. The viscosity trend of the MRG with nanoparticles showed a decreasing trend in off-state conditions, while increasing when the external magnetic force was applied, attributed to the nature of the thickener grease [6, 9]. Under on-state conditions, the performance of MRG containing nanoparticles improved by 277%, achieved by filling the gaps between magnetic particles and enhancing the particle chain structures [9]. Besides, the improvement in the response time of the MRG has also been reported [10]. Additionally, the energy storage of the MRG increased by approximately 33%, while conductivity improved significantly with rising temperature and applied magnetic fields [11]. Many researchers have concentrated on enhancing the performance of MRG, such as dynamic yield stress, MR effect and rheological behavior under the influence of magnetic fields. However, the correlation of the physical properties and structural stability of the MRG with the addition of magnetic additives remains incomplete, particularly in understanding long-term stability, particle dispersion uniformity, and the influence of varying magnetic field intensities.

Therefore, this paper focuses on investigating the characterization of the physical properties and structural stability of nickel nanoparticle additive-based MRG. The utilization of nickel nanoparticles is to enhance the magnetic response of the micron-sized carbonyl iron particles (CIP) in the MRG, which allows better tunable towards their viscosity characteristics and improves the mechanical properties such as strength and durability. The addition of nickel nanoparticles is also expected to exhibit strong interaction behavior between microns and nanoparticles, which improves the chain strength and stability by creating more surface force in between particles under the influence of a magnetic field [12]. In this study, a series of NMRG samples are fabricated and analyzed using contact angle, FTIR, and magnetic rheometer. The results are discussed in the following section of the paper.

METHODOLOGY

MRG samples were fabricated by a mechanical stirrer utilizing three components: CIPs, grease, and nickel nanoparticles. The CIPs purchased from Sigma Aldrich were spherical with an average diameter of 5 µm. The lithium grease with NLGI 2 (UMW GRANTT International Sdn. Bhd.) was chosen as a suspension medium. This grade is selected due to its consistency at room temperature and is applicable for a wide range of applications. Additive nickel nanoparticles with a diameter of 100 nm were purchased from ACS Material LLC. The composition used for CIPs was 70 wt% [13], while nickel nanoparticles were 1, 3, and 5 wt%. The composition of the additive is limited to 5 wt% to represent a significant improvement in the performance without drastically altering the material's characteristic [9, 10, 14]. This also shows a significant enhancement of the materials' property, and at the same time avoids particle agglomeration [15]. All the components are mixed for 1 hour at 300 rpm under room conditions to obtain homogeneity before further characterization. Table 1 is the summary of the sample composition.

Table 1. Sample composition.

Sample	CIP, wt%	Grease, wt%	Nickel, wt%
NMRG0	70	30	0
NMRG1	70	29	1
NMRG3	70	27	3
NMRG5	70	25	5

The physical properties testing including identification of compound class, hydrophobicity behavior, and viscoelastic properties of the samples, was analyzed. The functional group of chemical compounds was identified using Fourier Transform Infrared Spectroscopy (FTIR) in the wavelength range between 500–4000 cm^{-1}. The hydrophobicity behavior was characterized using Attension Theta Tensiometer equipment. The 5 µl of distilled water was dropped on the base of 1 ml of NMRG. The image angle of the water droplet was captured and calculated using the embedded software. A magneto shear rheometer was utilized to study the viscoelastic properties of the NMRG. The rheometer was equipped with an MR device (MRD70/1T) to generate magnetic fields varied from 0–0.6 T by adjusting the input current from 0–3 A. For each measurement, 1 ml of NMRG was filled on the parallel plate with a diameter of 20 mm and 1 mm constant gap was applied. The viscous properties were determined by manipulating the shear rate from 0.01–1000 s-1 under rotational mode. The viscoelastic and deformation of the NMRG were evaluated under oscillatory mode through sweep strain and frequency tests. The strain was varied from 0.001%–10% at a constant frequency of 1 Hz. The frequency was varied from 0.1–100 Hz at linear viscoelastic (LVE) strain. The temperature of the rheometer was set at 25 °C and controlled by a Viscotherm VT2 to maintain the temperature throughout the experiment. Every single test was repeated several times to ensure consistency.

RESULTS AND DISCUSSION

Physical Properties

Physical properties play a fundamental role in determining the functional performance and stability of the smart composites under external-field stimuli. In this study, the wettability analysis of the samples was characterized by using the contact angle method. By definition, wettability is the ability of a liquid to maintain contact with a solid surface, and the balance between the intermolecular interactions of adhesive force controls it. The wettability property is important to enhance the NMRG functionality and potential technical applications, while at the same time encountering friction, cavitation, surface corrosion, and removing wear and contamination issues in the existing equipment [14, 16, 17]. A hydrophilic or a wetting liquid has a contact angle with a solid of less than 90°, while a non-wetting or hydrophobic liquid has a contact angle with a solid between 90°–180°. The summarized results are exhibited in Figure 1. It is acknowledged that initially, grease has intrinsic hydrophobic characteristics while metallic particles (nickel and CIP) do not. However, these metallic particles are suitable for creating superhydrophobicity adsorbent particles in the mixture. Analysis shows that all samples have inherent hydrophobicity properties with a contact angle >90°. The increasing wetting behavior on hydrophobicity was recorded by increasing nickel nanoparticle concentrations. The results show that contact angle readings are 90.5°, 93.4°, 95.3° and 98.1° for MRG0, NMRG1, NMRG3, and NMRG5, respectively. In fact, the hydrophobicity effect of NMRG5 increased by 8.4% compared to that of MRG0. A high wettability is essential to allow the lubricant to quickly spread and penetrate the narrow gaps between metal particles and the grease surface, improving the tribological performance. This involves physical adsorption and colloidal forces due to the interactions between polar and non-polar molecules, with minimal or absent water droplets in the mixture contributing to strong viscoelastic behavior [14, 16, 18]. When the colloidal forces are optimally balanced, the CIP and nickel nanoparticles will remain well-dispersed, resulting in strong surface adherence [19]. This condition allows the grease to spread uniformly without obstruction of the particle agglomeration [14]. The low wettability in the NMRG will disrupt the internal structure of the grease fibrous, leading to particle agglomeration and weak structural stability.

Figure 2 shows the FTIR result analysis for NMRG samples. In general, a similar absorbance frequency trend was observed for all samples. The medium sharp peak of C-H stretching is vibrated at the wavelength range between 3000–2800 cm^{-1}, representing the alkane compound class. A strong binding energy formed from the stable alkane group will lead to high resistance in the sample. A change in the absorbance is observed for the wavelength between 1550–1300 cm^{-1}. A strong absorbance peak was observed at a frequency of 1550 cm^{-1}, representing a stretching nitro compound (N-O). Besides, a weak alcohol compound, O-H bending appeared at a frequency range of 1370 $^{cm-1}$. A medium-light C-H was absorbed at 1450 cm^{-1} due to the presence of an alkane group. A weak vibration at the wavelength of 700 cm^{-1} depicted a bending alkene (C=C). It shows insignificant changes in the absorbance peak, although there is an increase in the nickel nanoparticle concentrations. The stable groups that appear in the graph have weakened the physical adsorption of the metallic particles to the grease medium. It is also believed that the agglomeration structures between metallic particles have formed and contributed to the inactive electron transfer process, as most chemical groups appear as stable bonds. This condition has caused the formation of stable surface adhesion of the mixture, indirectly increasing the hydrophobicity effect [20]. The microscopic image observed in Figure 3 depicts the agglomeration of spherical nickel and CIP before mixing with the grease. Thus, the addition of liquid additives in the NMRG is appropriate, as mentioned by [3], to loosen the grease fibrous and cluster formation between nickel and CIP for better particle interaction.

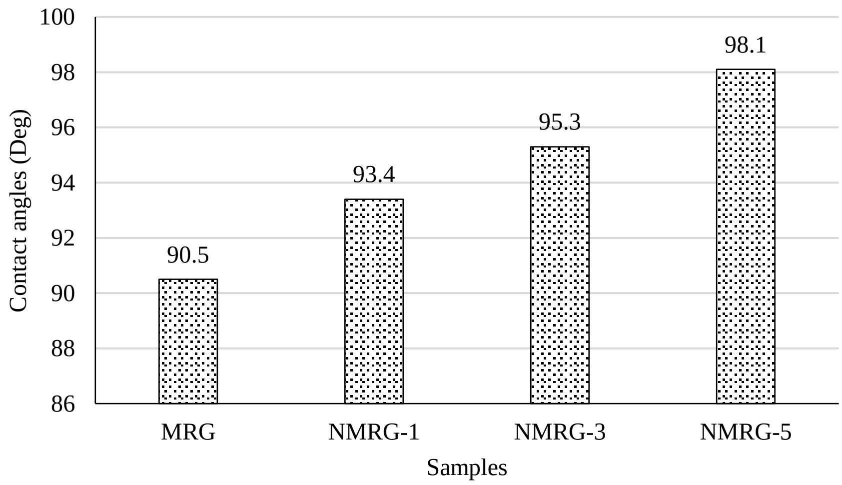


Figure 1. Wettability analysis.

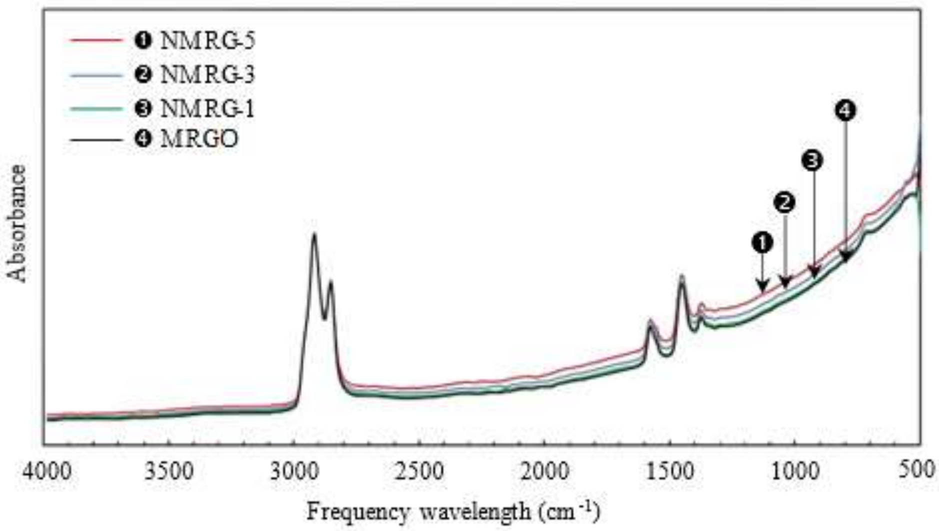


Figure 2. FTIR analysis.

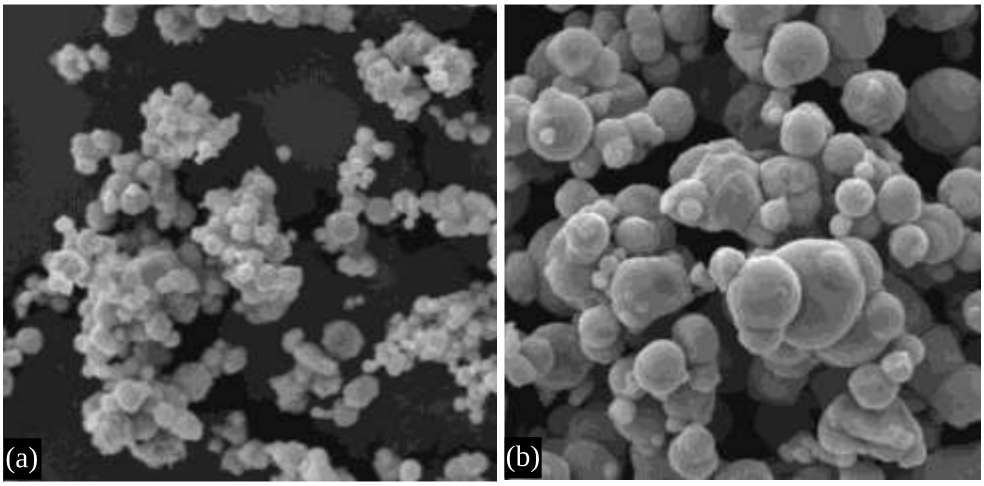


Figure 3. Microscopic image (a) nickel and (b) CIP.

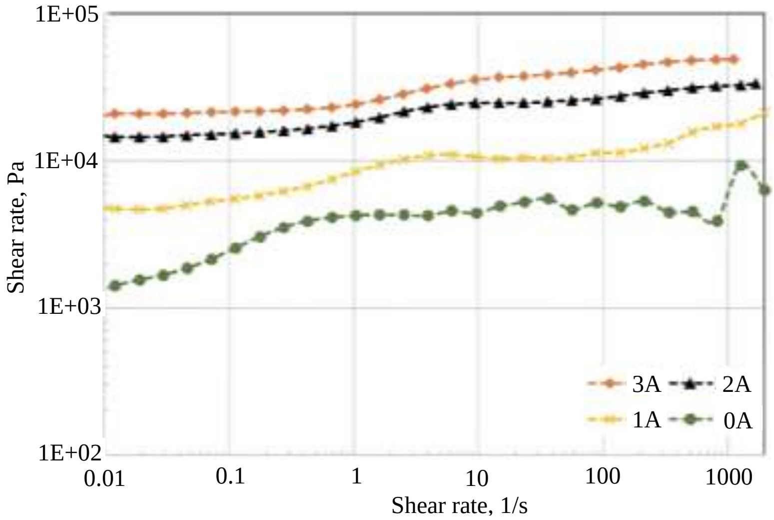
Rheological Properties

Structural stability in the NMRG is indicated by the interactions among the CIP and nickel nanoparticles and their dispersion within the grease. The addition of nickel nanoparticles in the NMRG is to strengthen the colloidal forces, improve the surface compatibility and prevent particle clustering between metallic particles and the grease network. Therefore, the magnetic properties of the NMRG samples are investigated using a rheometer. The results of shear stress dependent on the shear rate are depicted in Figure 4. The increasing pattern is examined from all NMRG samples aligned with the increasing applied magnetic field and behaves as a non-Newtonian fluid. The value of shear stress is also slightly increased over shear rates, which indicates the shear thickening effect. The increasing value of shear stress over NMRG samples resembles a high internal resistance, which requires a higher torque and leads to poor mechanical efficiency.

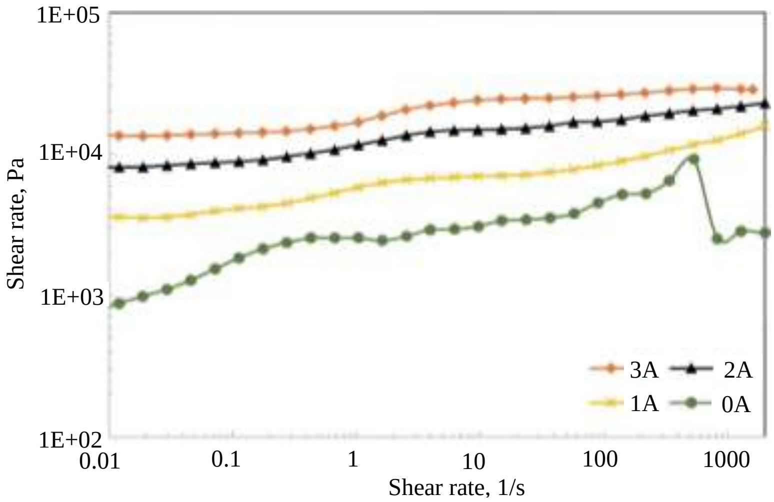
In contrast, a viscous mixture has less internal leakage through the clearance in rotating equipment, which results in better volumetric efficiency. Hence, the development of the NMRG with an optimal or higher shear stress value is necessary to achieve a higher load-carrying capacity under difficult tribological conditions [17]. As a rule, material friction depends on the molecular structure and the interaction force between metallic particles and fibrous grease. The entanglement of the fibrous grease is loosening over shear rate; however, the metallic particles remain in the strong chain network as the magnetic field is applied. Consequently, the shear stress is increased, and the tribological conditions, including wear and friction, are still under pulling action [21].

The viscous property of NMRG is further examined under the rotational motion mode of the rheometer. It is observed that the viscosity of the NMRG samples underwent shear-thinning behavior. This proves that the NMRG samples inherit the lubricant properties which become viscous over shear rate. The shear force has weakened the larger entanglement of the grease fibrous and destroyed the magnetic columnar constructions to achieve successive shear flow. These results are in line with the outcomes by Wang et al [21].

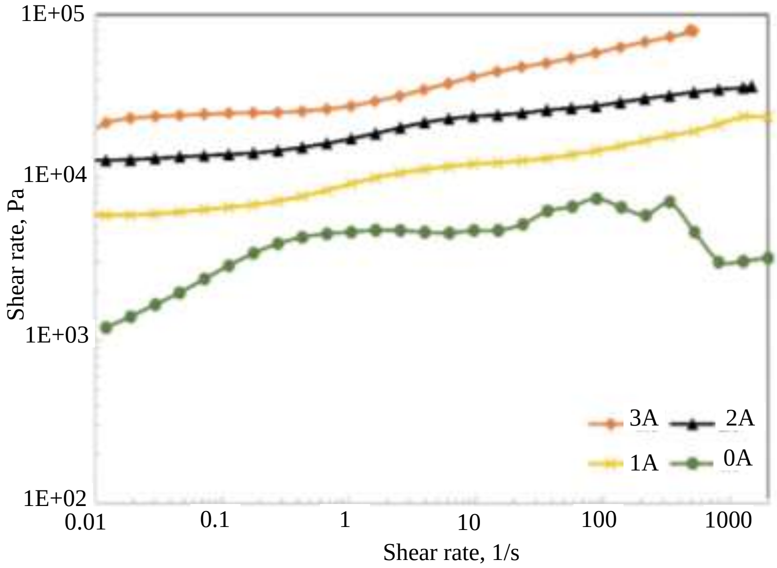
Figure 5 shows that the viscosity values of the NMRG samples increased with the addition of nickel nanoparticle concentrations; however, they decreased with the shear rate. The consistent fluctuation in the NMRG samples at 0A shows the metallic particles dependent on the shear rate direction and speed. By increasing the magnetic field, the consistent pattern of the graph is depicted. The presence of metallic particles in the grease structure behaves as a thickener. The aggregated metallic particles in the grease fibrous have slightly increased the NMRG viscosity.



NMRG0



NMRG1



NMRG3

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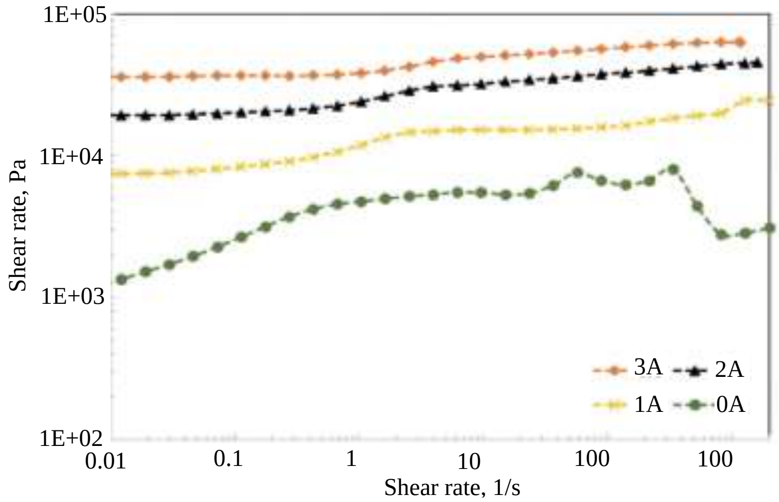
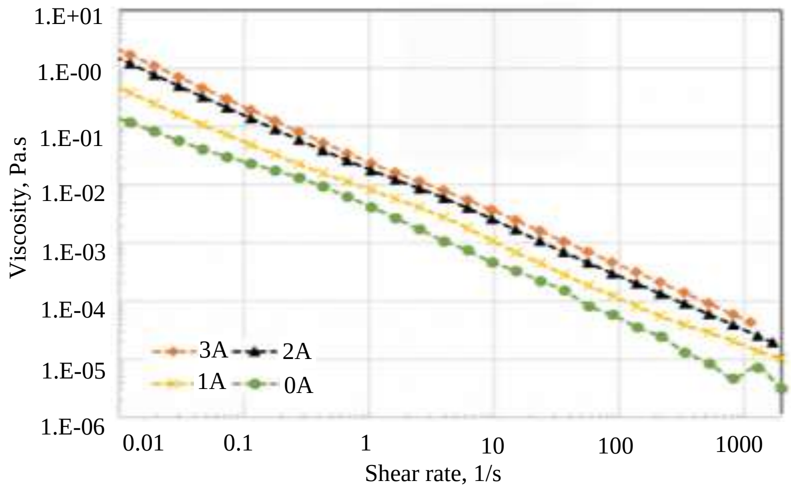
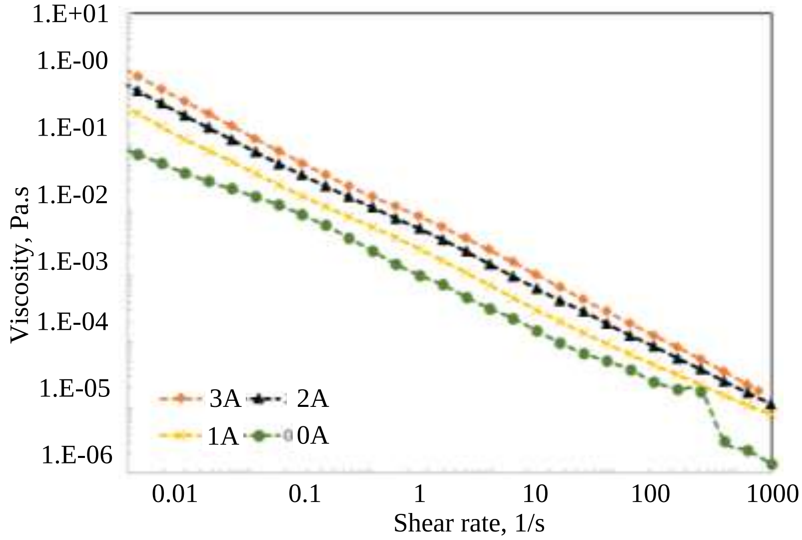


NMRG5

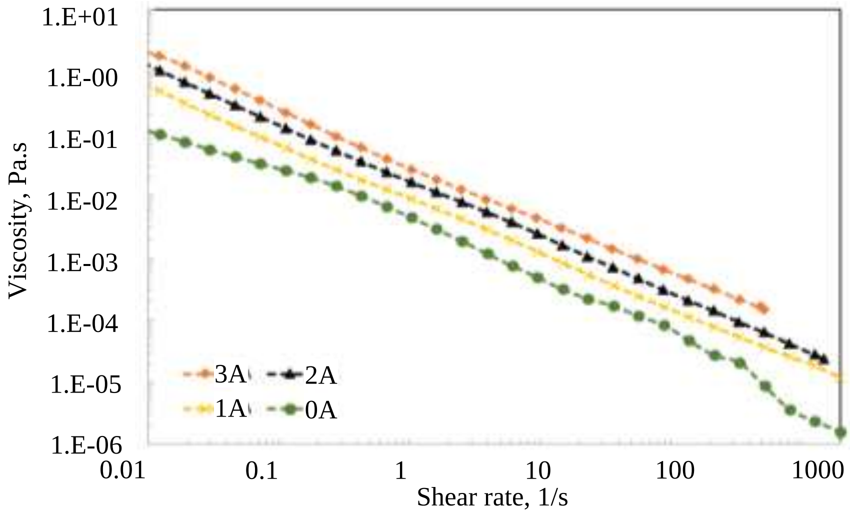
Figure 4. Shear stress of NMRG with different nickel nanoparticle concentrations and magnetic fields applied.



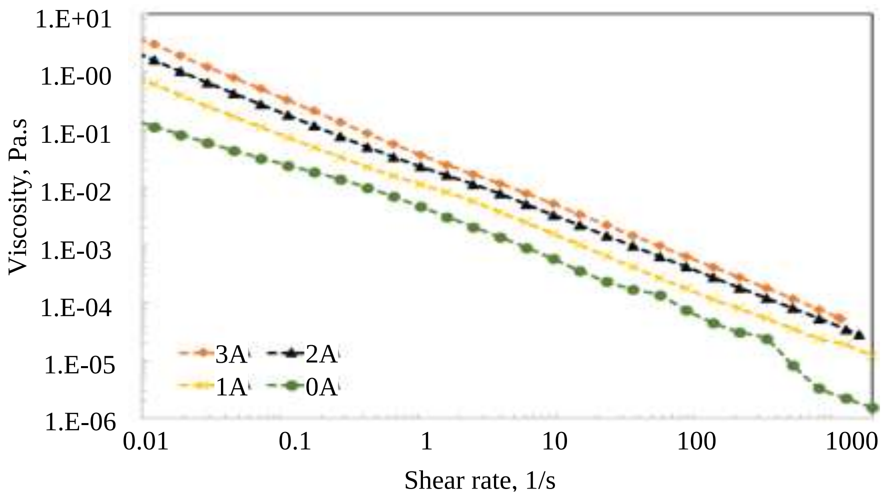
NMRG0



NMRG1



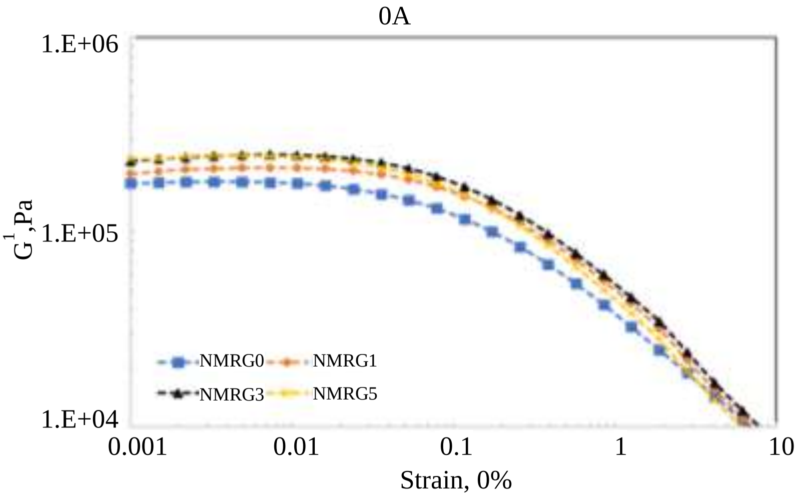
NMRG3



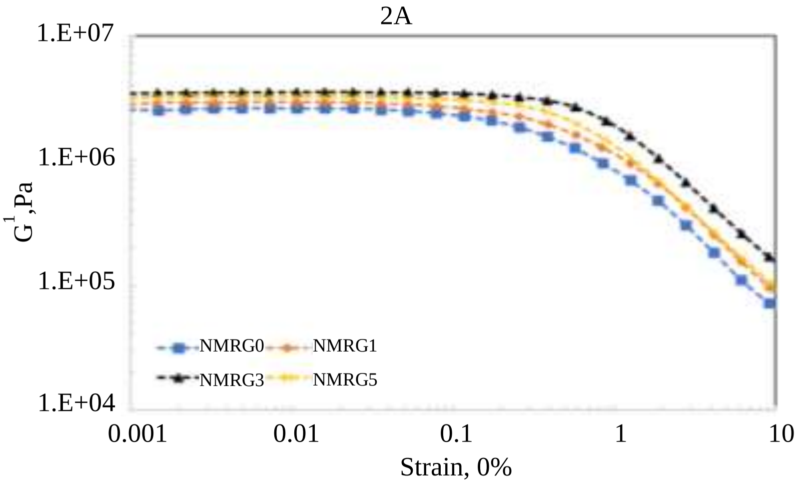
NMRG5

Figure 5. Viscosity of NMRG with different nickel nanoparticle concentrations and magnetic fields applied.

Figure 6 depicts the storage modulus, G', of NMRG samples under different nickel nanoparticle concentrations at off-state and on-state conditions. It is observed that the pattern is increasing for both conditions with increasing nickel nanoparticle concentrations. The G' value is enhanced by 57% from the initial condition compared to after applying the magnetic field for MRG0. Meanwhile, the G' value of NMRG samples increases by about 33% after the magnetic field is applied. Additionally, the linear viscoelastic (LVE) region of the NMRG samples extends further than the initial state as the nickel nanoparticle concentrations increase. For instance, the LVE of NMRG0 improves from a strain of 0.008% to 0.07%, while NMRG5 demonstrates the longest LVE region at on-state conditions, reaching 0.3% strain. This is attributed to the synergistic combination of micron and nano-sized metallic particles, which reinforce the grease-fibrous structures by filling the gaps within them. The breaking and reorganization interaction between metallic particles was developed during a magnetic field was applied. Hence, the ability to absorb the energy for self-healing characteristics is improved, and the structures are strengthened and stiffened. This molecular structure and internal networks have contributed to the strong viscoelastic behavior, which allows the sample to store more energy within its structure [14]. The results prove that the correlation between the elasticity, strength, and structural stability of the samples is significantly improved by adding nickel nanoparticles to the grease medium.



Off-state condition.



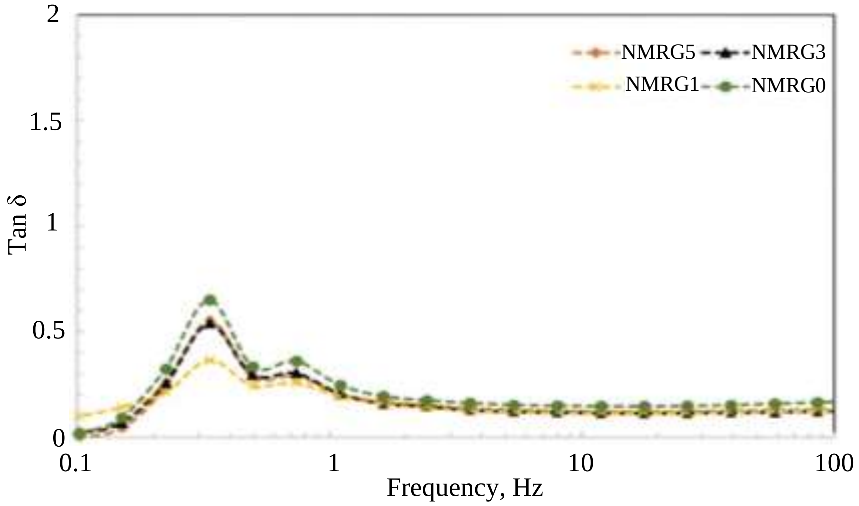
On-state condition.

Figure 6. Storage modulus of NMRG under different nickel nanoparticle concentrations.

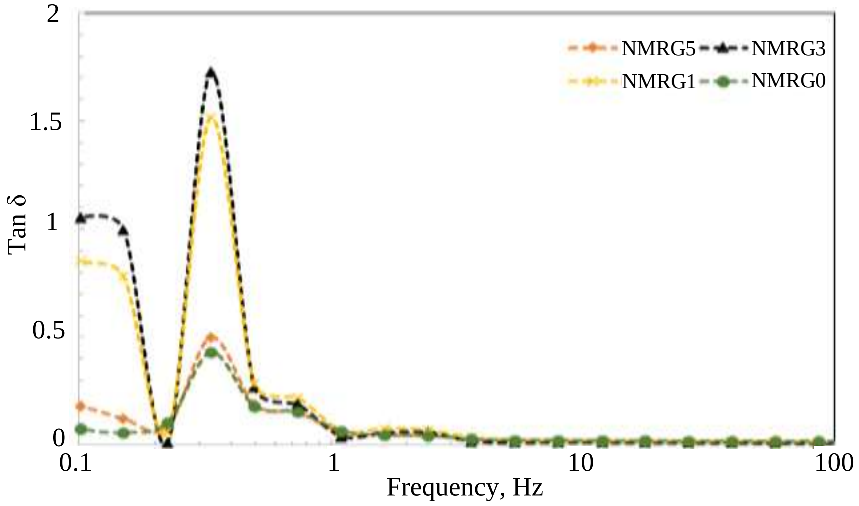
Further studies on the damping properties of the NMRG samples under the frequency domain are investigated, as depicted in Figure 7. The loss factor is calculated for each sample to identify how much the energy dissipates and the stability of the NMRG. It is observed that the fluctuating trend is happening at a frequency below 1.0 Hz at all applied magnetic fields. According to Mohamad et al [13]., this behavior is associated with the breaking of metallic chain structures. The process of breaking and reorganizing into thicker and stronger chain structures is primarily driven by high energy dissipation, represented by G". It is confirmed that the energy dissipation is higher at this state compared to the energy absorbed and only achieved linearity at a frequency above 1.0 Hz. With the addition of nickel nanoparticles, the storage and loss modulus of NMRG samples increases further.

In addition, pores between CIPs have been filled by the nickel nanoparticles, and the polar interaction between metallic particles, such as hydrogen bonding interaction, has made the samples denser. It is noted that the combination of different sizes of nickel nanoparticles and micro-sized CIPs tends to form agglomeration by increasing their wt%.

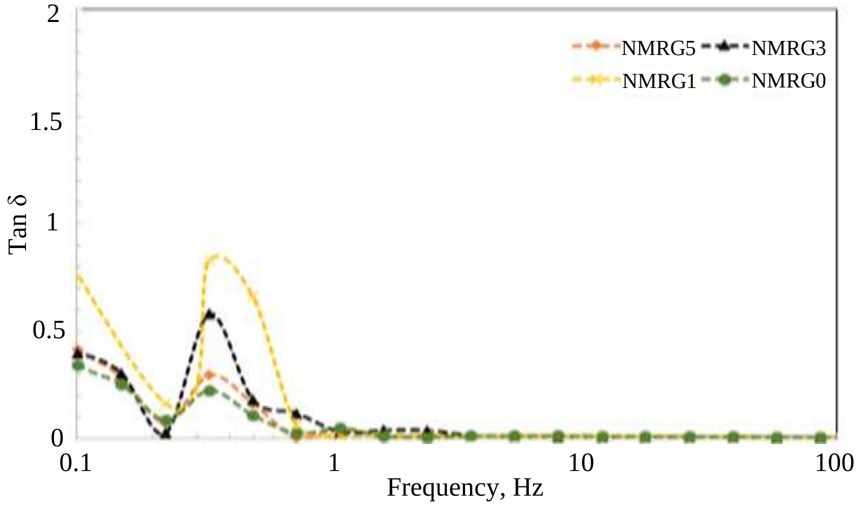
This agglomeration, which has reduced the distance between metallic particles and grease medium, will have higher interfacial friction due to the sliding motion during shearing. As a result, the loss factor decreases with increasing nickel nanoparticle content over the applied magnetic field. Therefore, it can be concluded that the presence of nickel nanoparticles has improved the deformation ability of the NMRG. To correlate, the rheological properties of NMRG enhance in line with the increase of surface wettability properties as mentioned by Huang et al [22].



Off-state condition, 0A



On-state condition, 1A



On-state condition, 2A

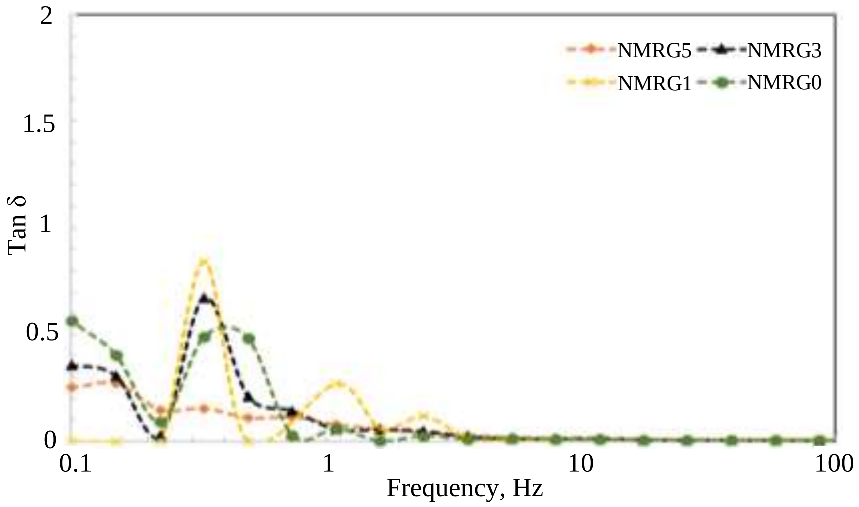


Figure 7. Damping properties of NMRG under different nickel nanoparticle concentration.

CONCLUSIONS

In this study, the physical properties and structural stability of nickel nanoparticle-based magnetorheological grease (NMRG) was investigated through a series of characterizations, including wettability, chemical composition, and rheological behavior. The findings demonstrate that the addition of nickel nanoparticles enhances the smart composite's hydrophobicity, viscosity, energy storage, and deformation characteristics. Notably, an increase of approximately 33% in energy storage was observed under an applied magnetic field, while the damping properties were optimized due to improved structural stability. These results suggest that nickel nanoparticle-enhanced NMRG holds significant potential for use in advanced damping and actuation systems, where tunable mechanical properties are essential. However, further studies are needed to evaluate long-term stability, sedimentation behavior, and performance under varying operational conditions such as temperature fluctuations and prolonged shear stress. Additionally, the effect of dilution oils on improving the dispersion and movement of metallic particles should be explored to further enhance the efficiency of the material.

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