

# Material-Level Degradation of Magnetorheological Fluids Under Long-Term Cyclic Shear

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## Abstract

*The long-term functional stability of magnetorheological fluids (MRF) remains a key limitation for their reliable use in adaptive systems and continuously operated magnetorheological devices. In this study, the intrinsic evolution of rheological properties in a commercial MRF (MRC-CIL) is systematically examined under prolonged cyclic loading, with the aim of isolating material-level degradation mechanisms independent of device-related effects. The fluid was subjected to 120,000 low-strain oscillatory shear cycles under a constant magnetic field, simulating extended service conditions while avoiding macroscopic structural failure. Rheological measurements revealed a progressive decrease of approximately 13 percent in magnetically induced shear stress, indicating partial irreversibility in field-induced particle chain formation and microstructural densification within the suspension. Microstructural characterization using scanning electron microscopy confirmed the morphological integrity of the carbonyl iron particles, with no evidence of fragmentation or severe wear. However, energy-dispersive X-ray spectroscopy detected a measurable increase in surface oxygen content from 1.4 percent to 1.5 percent, suggesting the onset of mild oxidative processes at the particle-fluid interface. These findings indicate that early-stage oxidative interfacial modification, rather than mechanical damage, is the dominant degradation pathway under low-strain cyclic loading, promoting irreversible particle clustering and a gradual loss of magnetorheological performance. The results provide new insight into the physicochemical aging behavior of MRF and offer practical guidance for the design of oxidation-resistant formulations with enhanced durability for demanding automotive and aerospace applications.*

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## INTRODUCTION

The advancement of smart material technologies has significantly enhanced the role of MRF in adaptive and controllable mechanical systems. First introduced by Rabinow, MRF exhibit a rapid, reversible transition from a low-viscosity Newtonian state to a semi-solid structure when subjected to an external magnetic field. This transition arises from the field-induced alignment of dispersed ferromagnetic particles, producing a tune-able change in rheological properties. Such real-time controllability has enabled their integration in diverse actuation and damping systems, including automotive brakes and clutches, prosthetic devices, aerospace actuators, and adaptive vibration absorbers [1–3].

Extensive studies have characterized the performance of MRF under post-yield or high-shear

conditions, where frictional wear, particle abrasion, and thermal effects dominate [4, 5]. These investigations have highlighted shear-induced heating and torque loss as principal causes of mechanical degradation. However, material deterioration often originates earlier which happen under near-static or pre-yield conditions and then through slow physicochemical processes such as oxidation, surface modification, and gradual interparticle bonding. Understanding these early-stage mechanisms is essential for bridging the gap between intrinsic material aging and the macroscopic performance degradation observed in practical actuator systems.

A recurring phenomenon associated with long-term service is In-Use Thickening (IUT), characterized by an irreversible increase in off-state viscosity that reduces magnetic responsiveness, increases energy consumption, and diminishes actuation efficiency [6]. Prior studies have linked IUT and related degradation effects to fatigue, thermal cycling, and oxidative reactions. For instance, Dang et al. [7]. IUT adversely impacts the field-responsiveness and functional efficiency of MRF-based systems, eventually leading to diminished device performance and failure. In operational terms, increased viscosity not only raises energy demands but may cause unanticipated mechanical resistance or functional delays in actuators and valves.

Several empirical investigations have documented these long-term degradation phenomena. For instance, Dang et al. [7] reported that MR seat dampers exhibited increased shear stress and viscosity after prolonged vibrational loading, with consequential declines in damping efficiency. Liang et al. [8] observed early mechanical stiffening in prosthetic knees embedded with MRF, primarily attributed to rheological aging effects. Similarly, Chen et al. [9] identified oxidation-driven surface roughening of magnetic particles as a key contributor to performance decay in MR fan clutches, noting that oxidation modifies particle surface energy, leading to stronger interparticle attraction and chain formation even in the absence of a magnetic field. Recent studies by Johari et al. [10] and Kikuchi et al. [11] further emphasized that environmental and operational conditions such as humidity, temperature variations, and vibrational stress significantly accelerate microstructural degradation, thereby altering the long-term rheological behaviour of MRF. Johari's work [10] demonstrated that even minor environmental changes in tropical climates could promote oxidation, while Kikuchi et al. [11] developed a specialized long-term test apparatus that revealed torque losses and microstructural failure in MRF formulations subjected to repeated cycling. However, a critical review of these studies reveals a persistent methodological limitation which is the difficulty of isolating intrinsic material aging from device-specific influences. Embedded systems inherently introduce complexities such as geometric confinement, field inhomogeneities, and thermal gradients, all of which confound the identification of pure material degradation pathways. As a result, interpretations of MRF degradation often merge system-level effects with fundamental material changes, limiting the generalizability of findings across applications. These confounding factors make it challenging to design fluids with universal long-term stability. Recognizing this challenge, researchers have progressively attempted to simplify experimental setups to better isolate material behaviours. Utami et al. [12] provided an important contribution by evaluating the long-term performance of MRF within damper systems operating under prolonged flow-mode conditions. Their work highlighted the significant roles of mechanical friction, particle oxidation, and carrier fluid breakdown in MRF degradation. However, while their study advanced the understanding of degradation under operationally relevant conditions, it was still constrained by the intrinsic complexities of device architecture and fluid dynamics, which could not be entirely eliminated. Building on this foundation, recent advances in material testing have emphasized the importance of conducting investigations under highly controlled rheometric environments. Kikuchi et al. [11] developed a specialized durability test apparatus that successfully minimized mechanical artifact influences, thus allowing more reliable rheological assessments. This enabled the controlled study of degradation over thousands of cycles without interference from device-specific parameters.

Nonetheless, comprehensive studies coupling rheological evolution with in-depth microstructural and chemical analyses under purely material-level conditions remain relatively scarce. For example, the

study conducted by Dhiman et. al. [13], investigate the rheological behaviour of magnetorheological elastomer through prediction using machine learning. The current understanding of MRF degradation further indicates that multiple intertwined mechanisms such as oxidation, sedimentation, particle agglomeration, and shear-induced structural rearrangements contribute to long-term performance loss [14, 15]. Oxidation not only alters the surface chemistry of magnetic particles but also facilitates particle fusion and hard agglomerate formation, which increases off-state viscosity and hinders field-induced structure formation. Sedimentation and density-driven particle settling disrupt homogeneity, while mechanical fatigue promotes irreversible microstructural changes detrimental to dynamic response characteristics. Additionally, carrier fluid breakdown under thermal stress may alter dispersion quality and surface tension, further destabilizing the colloidal structure of MRF. Given these complex degradation pathways, there is a compelling need for systematic investigations that isolate MRF material behaviour under operationally representative but simplified conditions. In particular, there remains a notable gap in studies that directly correlate long-term rheological property evolution with microstructural and chemical transformations in the absence of device-induced artifacts. Few works have simultaneously employed scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX) alongside rheological analysis to build a holistic view of aging processes at both macro and micro levels.

Accordingly, while prior studies such as those have provided critical preliminary insights into MRF long-term performance within operational devices like in prosthetic knee device [16], a deeper material-level understanding remains necessary. Expanding on this foundation, the present study aims to evaluate the intrinsic rheological and microstructural evolution of a commercial MRF subjected to prolonged cyclic oscillatory shear under controlled magnetic fields. A dynamic shear rheometer setup, devoid of complex device geometries and hydrodynamic effects, is employed to isolate pure material degradation mechanisms. Complementary characterization techniques, including SEM and EDX, are utilized to investigate particle morphology, surface oxidation, and elemental composition variations associated with aging processes.

Unlike prior studies limited to device-level degradation, this work isolates intrinsic material aging under controlled cyclic shear, providing the first correlation between rheological softening and surface oxidation in commercial MRF. Through this integrated approach, the study seeks to elucidate the dominant pathways governing MRF degradation, offering generalizable insights applicable across diverse smart material applications. The findings of this research are expected to advance the scientific understanding of long-term MRF behaviour, inform material optimization strategies aimed at enhancing durability, and guide the development of predictive models for performance degradation. By establishing a clearer linkage between rheological property shifts and microstructural changes, this study addresses a critical and largely underexplored dimension of smart material science, ultimately contributing to the commercialization and reliability improvement of next-generation MRF-based systems.

## **MATERIAL AND METHODOLOGY**

This section outlines the experimental procedures employed to evaluate the long-term rheological behaviour of a commercial MRF under controlled oscillatory stress conditions.

### **Material Selection**

The material selected for this study was a commercially available MRF, designated as MRC-C1L, supplied by CK Material Laboratory, Seoul, Korea. This fluid is commonly used in automotive applications, including brakes, dampers, and shock absorbers, and is composed of carbonyl iron particles (CIPs) suspended in a hydrocarbon-based carrier medium. The solid content is approximately 78 wt.%, ensuring a high magnetic response, while the hydrocarbon carrier fluid contributes to thermal and chemical stability across a wide operational temperature range. Key physical and rheological properties of MRC-C1L are summarized in Table 1.

The relatively high shear stress and moderate off-state viscosity of MRC-C1L make it suitable for long-term rheological and mechanical loading studies, providing a representative assessment of fluid durability in dynamic environments.

### Sample Preparation and Initial Characterization

Prior to testing, the MRF sample was mechanically stirred at 250 rpm for 15 minutes to ensure homogeneous dispersion of the magnetic particles and minimize sedimentation effects. This preconditioning step was essential for maintaining consistency across all measurements. Baseline material characterization was conducted using SEM and EDX. SEM analysis provided insights into particle morphology and distribution, while EDX identified elemental composition, focusing on iron and oxygen content. These initial assessments established a reference point for evaluating subsequent microstructural and compositional changes after long-term cyclic testing.

### Rheological Testing and Long-Term Oscillatory Experiment

Rheological measurements were performed using a dynamic shear rheometer (Anton Paar MCR 302, Graz, Austria) equipped with a 20 mm parallel-plate magnetorheological device (PP20/MRD/TT). All tests were conducted at a controlled temperature of 25 °C. Initial viscosity measurements were performed under both off-state (0 A) and on-state (1–2 A) magnetic field conditions.

For on-state characterization, a magnetic field was generated by applying electrical current through the magnetorheological device, with an estimated magnetic flux density of approximately 0.2 T at 1 A. Viscosity profiles were recorded over a shear rate sweep from 0.01 to 1000 s<sup>-1</sup>. Following the initial characterization, the MRF was subjected to a long-term oscillatory shear experiment designed to simulate extended mechanical cycling. Oscillatory loading was applied continuously under a constant magnetic field at 1 A. Test parameters are listed in Table 2.

After completion of 120,000 oscillatory cycles, the sample was re-evaluated using the same rotational shear test protocol as applied initially. Post-test viscosity data were compared to the pre-test results to assess changes in rheological properties. Unlike previous studies that evaluated MRF degradation within assembled devices such as dampers, where device-specific factors such as valve geometry and flow dynamics influenced the results, e.g., in prosthetic knee device [16], this methodology isolates material-level effects by eliminating external system interactions. The use of a controlled rheometric environment provides a direct and reproducible measure of intrinsic fluid degradation under cyclic mechanical stress.

**Table 1.** Properties of MRC-C1L from CK Laboratory, Korea.

Property	Value
Density (g/cm <sup>3</sup> )	2.75 ~ 2.95
Flash point (°C)	>140
Operating Temperature (°C)	-40 ± 140
Shear stress (kPa @ 1500/s, 570 mT)	68.0 ± 7
Solid Content (wt%)	77 ~ 80
Sedimentation Stability (vol%, 30 days)	4.00
Viscosity (Pa.s @ Calculated as slope 800-1200/s, 0T, 40 °C)	0.106 ± 0.020

**Table 2.** Oscillatory Test Parameters

Parameter	Value
Frequency, Hz	2
Applied Current, A	1
Strain, %	0.000001
Initial gap, mm	1
Remarks	Continuously

## RESULTS AND DISCUSSION

### Rheological Response under Long-Term Oscillatory Test

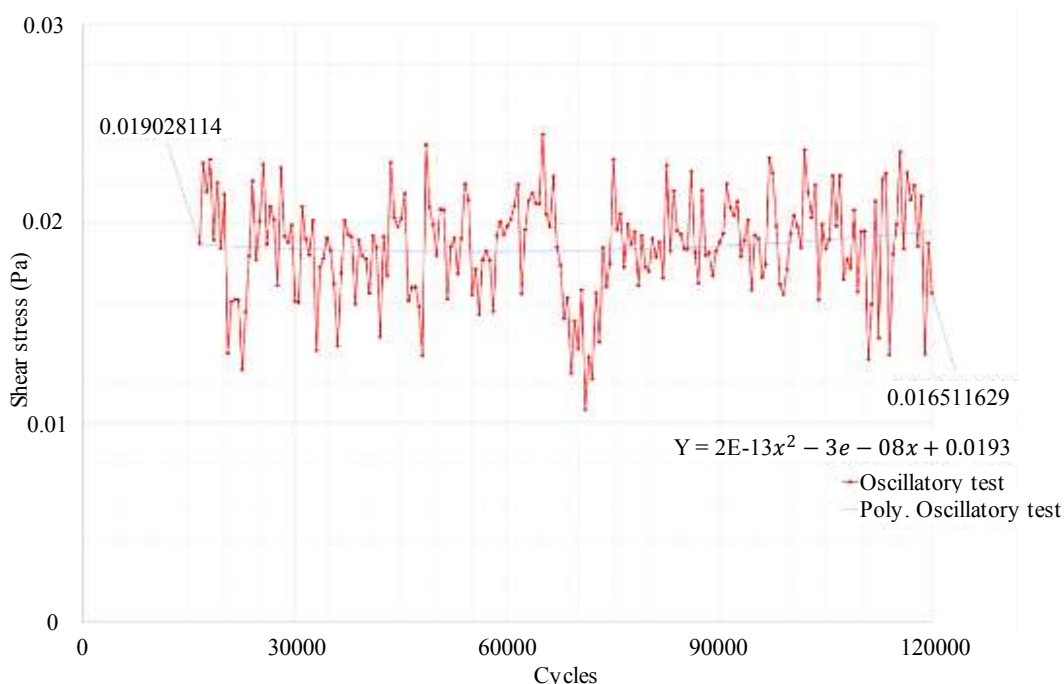
The rheological performance of MRF is fundamentally governed by yield stress, viscosity, and shear-dependent flow characteristics. In this study, shear stress and viscosity were selected as the primary indicators to evaluate the influence of long-term oscillatory shear on fluid performance. These parameters are particularly sensitive to changes in the microstructure and interparticle interactions within the MRF.

Figure 1 presents the evolution of shear stress as a function of cycle count during the application of oscillatory stress over 120,000 cycles. The test conditions were kept constant, where the applied current of 1 A, frequency of 2 Hz, strain amplitude of 0.000001%, and a gap of 1 mm are set. The results demonstrate a notable and progressive decrease in shear stress, which follows a linear trend modelled by the equation:

$$y = 2 \times 10^{-13}x^2 - 3 \times 10^{-8}x + 0.0193$$

where  $y$  is the shear stress (Pa), and  $x$  is the number of cycles. The shear stress decreased from an initial value of 0.0190 Pa to approximately 0.0165 Pa by the end of testing with a decrease of nearly 13%. This finding indicates that shear stress in magnetorheological (MR) fluids subjected to long-term oscillatory tests decreases due to particle structure evolution over time. This phenomenon is influenced by factors such as magnetic field strength, shear rate, and test duration [17, 18].

Higher magnetic fields and shear rates can accelerate particle structure evolution, reducing hysteresis and the shear history effect [18]. Unexpectedly, in dilute MR fluids, shear stress may decrease with increasing magnetic field at constant shear rates [19]. Long-term operation can alter MR fluid rheological properties, as observed in continuous-load experiments using MR dampers [12]. The time required to reach steady-state shear stress is inversely proportional to magnetic field strength and directly proportional to shear rate [17]. These findings have implications for the design and performance of MR fluid-based devices in various applications.



**Figure 1.** Shear stress variation of MRC-C1L over 120,000 oscillatory cycles at 1 A.

Previous studies have reported several findings. For instance, [17] observed that repeated shear can enhance stress-bearing capacity due to the strengthening of chain structures in concentrated MRF. Likewise, [18] found that MRF exposed to long-term fatigue exhibited a gradual rise in yield stress due to increased internal particle bonding and oxidative effects. These observations support the current study's finding that cyclic mechanical excitation, when combined with continuous magnetic activation, can promote a change of force in high-solid-content MRF. It is important to note, however, that not all long-term MRF studies report an increase in rheological parameters. In contrast, [19] found that in low-viscosity, dilute MRF subjected to flow-mode operation, shear stress tended to decrease over time due to structural collapse and particle migration. The divergence in outcomes may be attributed to differences in fluid composition, test geometry, and operational mode. Whereas flow-mode tests often induce internal turbulence and chain fragmentation, the oscillatory mode used in this study operates at low strain and high particle confinement, furthermore with constant magnetic applied in long term will promote particle surface structure disruption.

Therefore, the observed decrease in shear stress in MRC-C1L suggests that under the tested conditions, such as low strain, high solid loading, and moderate magnetic field, the fluid's microstructure becomes more robust over time. This behaviour may reflect a transition toward irreversible particle clustering or agglomeration, which, although improving stiffness, may eventually compromise the fluid's reversibility and dynamic responsiveness. This hypothesis is further evaluated through microstructural and elemental analysis in later sections.

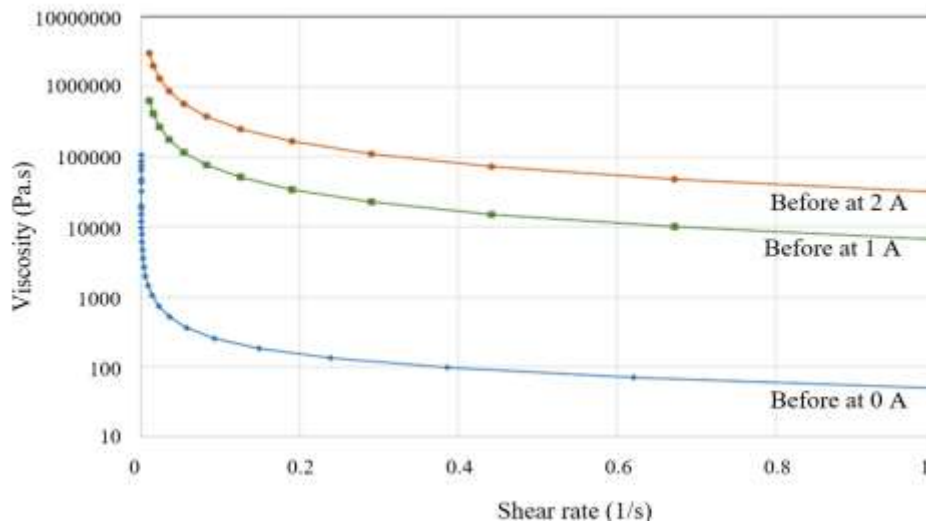
### **Viscosity Evolution Before and After Long-Term Oscillatory Test**

The viscosity-shear rate behaviour of MRC-C1L was assessed before and after 120,000 cycles of oscillatory loading at magnetic field intensities of 0, 1 and 2 A. These results, presented in Figures 2 to 4, provide a detailed view of the fluid's rheological response under static and magnetically activated conditions, as well as the evolution of its flow behaviour.

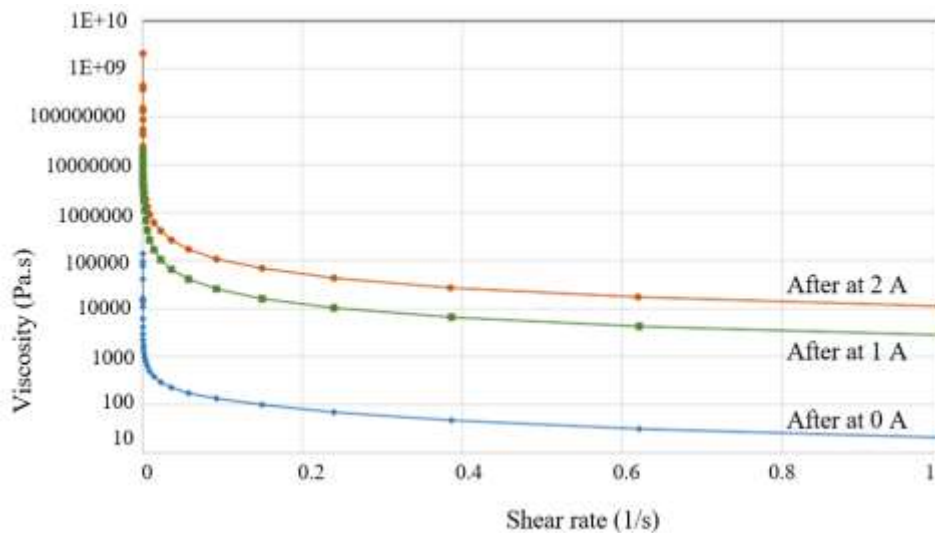
Before oscillatory shear testing, the MRF exhibited nearly Newtonian behaviour in the absence of a magnetic field as shown in Figure 2, with viscosity remaining relatively constant across the range of shear rates. This flow behaviour is characteristic of the base hydrocarbon carrier fluid, where the randomly dispersed CIPs contribute minimally to flow resistance due to the lack of an applied field. Under magnetic excitation at 1 and 2 A, the viscosity increased markedly at low shear rates, and the flow profile transitioned to shear-thinning behaviour, similar to previous study reported by Nagirella, et. al [20]. This effect is a direct result of the magnetorheological response, where magnetic field-induced dipole interactions cause the CIPs to align into chain-like structures, resisting shear and increasing the apparent viscosity [5, 21]. As shear rate increases, these structures are disrupted, leading to a reduction in viscosity and a more fluid-like response.

Following 120,000 cycles of oscillatory shear at 2 Hz and 1 A, a substantial increase in on-state viscosity was observed at both 1 and 2 A as shown in Figure 3, while the off-state viscosity (0 A) remained relatively unchanged. This trend suggests that significant structural changes occurred within the magnetic field-activated state. The prolonged exposure to shear stress and magnetic alignment appears to have facilitated microstructural rearrangement, likely through the formation of denser or more cohesive particle networks that resist flow more effectively. Such changes are indicative of in-use thickening mechanisms driven by field-dependent fatigue, where interparticle friction and chain stabilization evolve over time [12, 22].

The comparative analysis of pre- and post-test viscosity profiles at each field intensity, shown in Figure 4 (a–c), provides further clarity. At 0 A (Figure 4a), the viscosity showed only a slight increase, suggesting minimal structural alteration in the absence of a magnetic field. This outcome implies that the carrier fluid remained chemically stable and that sedimentation or particle aggregation was not a dominant factor under these test conditions. A more pronounced change was recorded at 1 A (Figure 4b), where viscosity increased across all shear rates after fatigue testing.



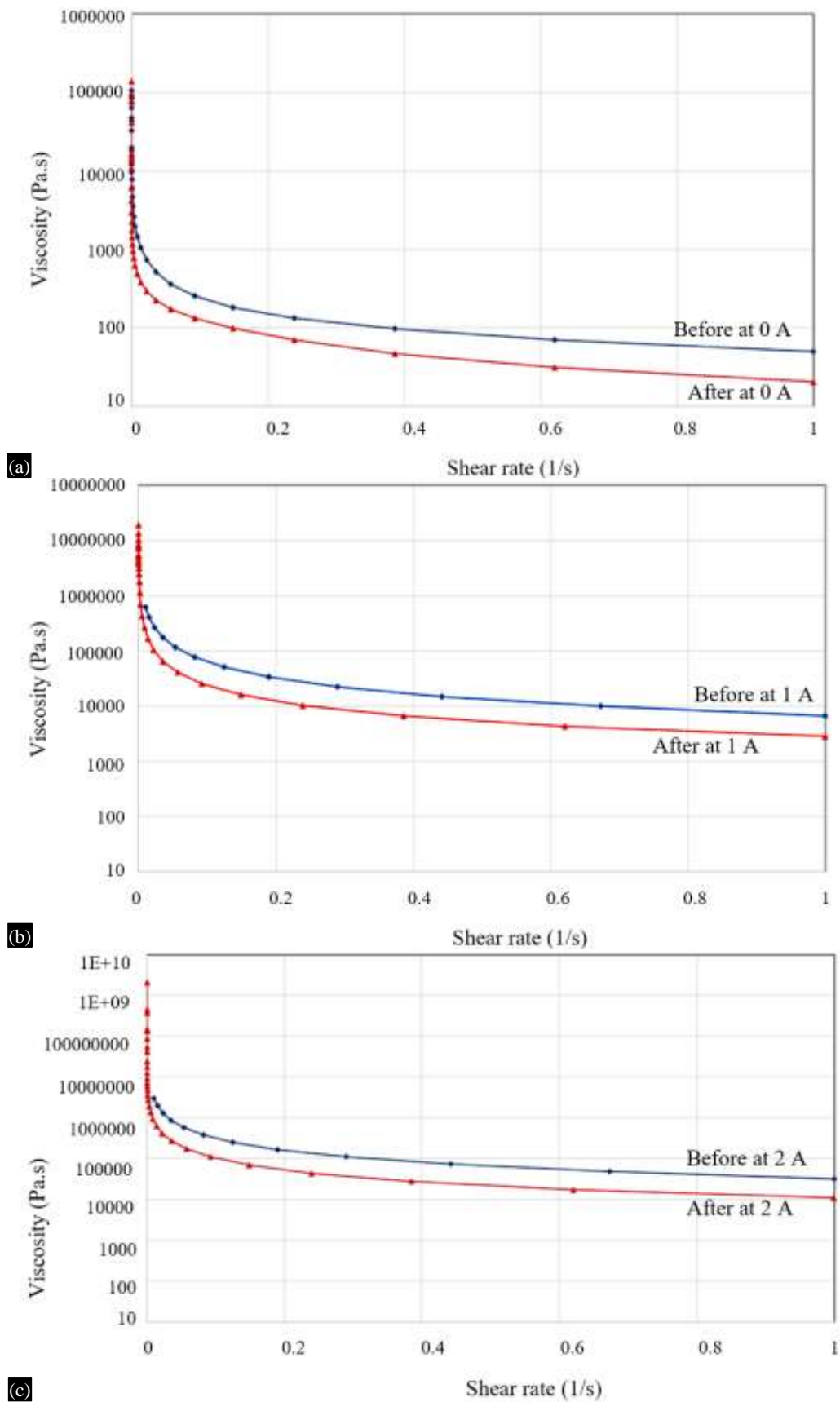
**Figure 2.** Viscosity-shear rate profile of MRC-C1L before oscillatory cycling at 0, 1 and 2 A.



**Figure 3.** Viscosity-shear rate profile of MRC-C1L after 120,000 oscillatory cycles at 0, 1, and 2 A.

This behaviour reinforces the notion that magnetic activation, when coupled with cyclic shear, promotes the accumulation of internal particle structures and enhances resistance to flow. The findings are consistent with the decrease in shear stress observed in Section 3.1, supporting the conclusion that MRC-C1L undergoes structural rearrangement rather than degradation under these loading conditions. At 2 A (Figure 4c), the post-test viscosity also increased, though the difference compared to pre-test values was less substantial than at 1 A. This outcome may be explained by the onset of magnetic saturation, where most of the CIPs are already aligned in strong chains, and additional field strength contributes marginally to further microstructural build-up [11, 23]. Once saturation is reached, the fluid exhibits limited capacity for additional thickening, even under prolonged mechanical cycling.

These results demonstrate that the viscosity changes observed in MRC-C1L after prolonged oscillatory loading are closely associated with magnetic field exposure, with more pronounced structural evolution occurring under on-state conditions. The increase in viscosity, particularly at 1 and 2 A, reflects a cumulative rearrangement of particle structures rather than degradation of the carrier fluid. This trend is consistent with the progressive drop in shear stress discussed previously in Section 3.1 and instigated the interpretation that rheological aging in this fluid is primarily driven by field-dependent microstructural consolidation.

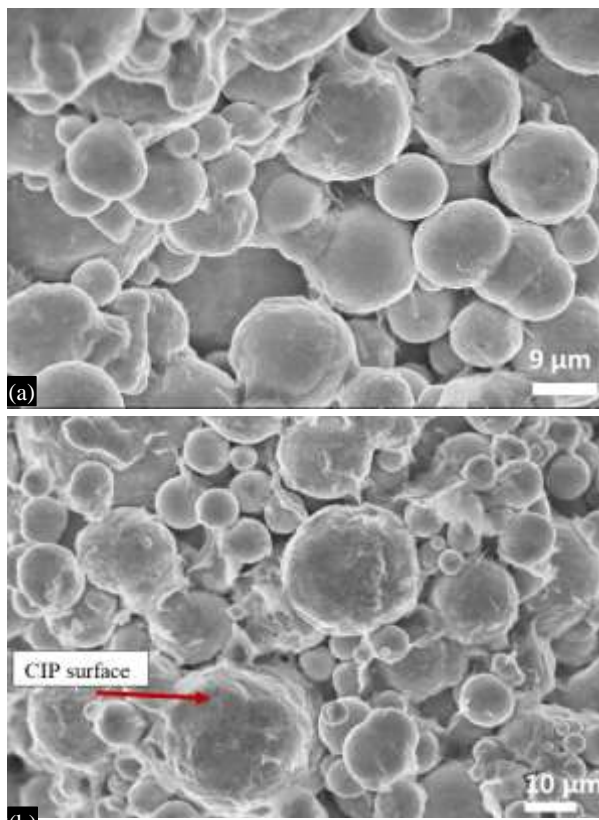


**Figure 4.** Viscosity comparison at (a) 0, (b) 1 and (c) 2 A, before and after long-term oscillatory loading.

### Microstructural and Elemental Analysis

Understanding the microstructural integrity and chemical stability of MRF under cyclic shear loading is essential for evaluating long-term rheological performance. This study incorporated SEM and EDX to examine the physical and compositional evolution of MRC-C1L after 120,000 oscillatory cycles.

SEM images of the CIPs extracted from the fresh MRF, shown in Figure 5(a) revealed a generally uniform spherical morphology, with diameters ranging from 1 to 5  $\mu\text{m}$ . The particles exhibited smooth surface features indicative of minimal oxidation or contamination at the initial stage. This morphology is characteristic of CIPs widely used in commercial MRF due to their magnetic responsiveness and flow compatibility [24]. Following the long-term oscillatory shear test, SEM analysis of the aged MRF sample, Figure 5(b), indicated no substantial change in particle shape or size. The particles retained their spherical geometry and displayed no apparent signs of mechanical fragmentation or agglomeration. This observation implies that the test conditions, moderate magnetic field strength and room temperature, were not sufficient to cause degradation through abrasion or fatigue. Similar results were obtained in the work of [22] who observed morphological preservation of MRF particles after damper-based fatigue cycling under operational conditions.



**Figure 5.** SEM images of MRC-C1L CIPs; (a) fresh sample before testing, and (b) after 120,000 oscillatory cycles; particles retained spherical morphology with minimal change.

Despite the morphological stability, chemical surface changes were detected through EDX spectroscopy. The oxygen content in the as-received MRF was measured at 1.4%, while post-test analysis showed a slight increase to 1.5%. Although minor, this increase is indicative of progressive oxidation at the surface of the iron particles. The initiation of oxidation is a known degradation mechanism in MRF, often driven by exposure to ambient oxygen, moisture, and repeated mechanical activation, which can disrupt surfactant layers and increase surface reactivity [23, 24]. Surface oxidation is of particular concern because it can lead to increased interparticle friction and reduce magnetic responsiveness. Over extended periods or under elevated temperatures, oxidation contributes to the

formation of rigid agglomerates and can impair field-induced chain formation. Study by Ulicny., et. al [22] reported similar trends in automotive-grade MRF, emphasizing that even slight changes in oxygen content can correlate with diminished field sensitivity and elevated off-state viscosity.

Furthermore, [11] developed an accelerated test apparatus for MRF and reported that under elevated thermal and magnetic stress, the growth of oxide layers was significantly correlated with rheological aging, particularly in systems with high solid content. While the current experiment did not introduce severe oxidative conditions, the observed elemental variation aligns with early-stage material degradation pathways.

The complementary SEM and EDX analyses collectively demonstrate that the prevailing mode of structural changes under the applied testing conditions was governed primarily by subtle surface-level chemical alterations rather than by physical fragmentation or particle aggregation. This observation is consistent with the rheological evidence discussed earlier, suggesting that long-term cyclic loading can initiate internal material transformations at the chemical interface, even when no apparent morphological degradation is observed.

## CONCLUSION

This study systematically examined the intrinsic rheological stability and degradation behavior of a commercial magnetorheological fluid (MRC-C1L) under prolonged cyclic shear in a controlled magnetic field environment. By eliminating device-related influences, the work isolated the material's inherent degradation mechanisms and established a clear linkage between rheological evolution and surface chemical modification. The observed 13% decrease in magnetically induced shear stress reflects progressive microstructural rearrangement and densification within the field-induced particle chains. Despite this rheological softening, SEM analysis confirmed that particle morphology remained intact, suggesting that mechanical fatigue and fragmentation were not dominant factors.

EDX analysis revealed a slight increase in oxygen content from 1.4% to 1.5%, identifying the initiation of surface oxidation as the primary early-stage degradation pathway. These findings demonstrate that even mild oxidative modification can promote irreversible clustering and alter long-term flow characteristics, contributing to a deeper understanding of performance deterioration mechanisms in magnetorheological fluids.

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## Authors' Contributions

- *Dewi Utami*: Conceptualization, Methodology, Investigation, Data Curation, Visualization, Formal analysis, Writing – original draft
- *Saiful Amri Mazlan*: Visualization, Supervision, Funding acquisition, Project administration, Writing – review & editing
- *Nur Azmah Nordin*: Methodology, Investigation, Supervision
- *Mohd Aidy Faizal Johari*: Methodology, Visualization, Supervision, Writing – review & editing
- *Ubaidillah*: Methodology, Investigation, Supervision

## Data Availability Statement

The datasets generated during this study are not publicly available because this research is part of an ongoing project.

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### Conflicts of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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