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RESEARCH PAPER

Nano-Clay Enhanced Demulsification of Oxidized Turbine Oils for Improved Water Separation Efficiency

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ABSTRACT

Water-oil emulsification, especially in steam turbine engines, is very common and degrades the performance characteristics of oils, causing salting, clogging of pipes, and apparatus corrosion due to water salts. This work will be interested in the effect of adding nano-clay on the prevention of emulsification for Iraqi turbine oils, enhancing the separation of water from oil. Turbine oil 4006 was oxidized at 150°C for different periods (0, 2.5, 5, 10, and 15 hours) and the emulsification characteristics by the ASTM D 1401 method were researched. The results are that with the prolongation of oxidation time, the emulsification layer is increased, and the separation of water and oil is delayed. Base oil 60 was also oxidized at 120°C for 3 hours. In these, nano-clay treatments at 0%, 1%, 2%, and 4% w/w were given. Addition of nano-clay reduced the emulsification layer and enhances the water separation mainly with 4% nano-clay treatment. Comparison between the two oils revealed that base oil 60 exhibited faster water separation than turbine oil. Treatment with nano-clay reduced the surface tension of both the oils and coalesced the water droplets more, thus enhancing the efficiency of separation. These findings imply that nano-clay could be used as an efficient demulsifier for improving the performance of turbine oils.

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INTRODUCTION

Steam turbine oils play a critical role in ensuring optimal turbine performance, with demulsibility standing out as a key parameter. Contamination of oils with water, often exacerbated by certain additives, can lead to the formation of stable emulsions that compromise turbine efficiency [1,2]. Although turbine oils are formulated with corrosion inhibitors, aging stabilizers, extreme pressure agents, and anti-wear additives to enhance reliability and longevity, these compounds can inadvertently promote emulsification [3]. Among indigenous compounds, asphaltenes are considered major contributors to emulsion formation, producing complex colloidal systems characterized by the immiscibility of the liquid phases involved [4]. Emulsions are thermodynamically unstable and gradually undergo destabilization through physicochemical processes such as gravity separation, droplet coalescence, flocculation, phase separation, and Ostwald ripening [5,6]. Efficient demulsification, therefore, is essential and can be achieved through chemical, biological, or physical methods [7-11]. Chemical demulsification involves introducing specialized demulsifiers to weaken the stability

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of emulsions and accelerate film thinning at the water–oil interface. These demulsifiers are typically classified into ionic liquids, polymeric surfactants, and nanoparticles based on their chemical structure and application [12,13,7]. Nanotechnology has significantly expanded its reach into multiple sectors, including energy, by offering innovative solutions to the challenges faced by the oil and gas industry [11]. Notably, nanoparticles have shown remarkable potential as demulsifiers, effectively enhancing emulsion breaking processes [12,13]. Recent research has demonstrated that nanoparticles not only act as efficient demulsifiers but also serve as deposition inhibitors and selective adsorbents for asphaltenes. Their adsorption capacity increases with the aromaticity and polarity of asphaltenes, and their interaction with other oil components, such as resins, further improves their demulsification performance [7]. In particular, composite nanoparticles combining inorganic cores with hydrocarbons exhibit synergistic effects in preventing asphaltene deposition. Typical sorbents used in such applications include clays, rock minerals, glass, alumina, silica, carbon materials, polymers, metals, and metal oxides [3]. Owing to their nanoscale dimensions, high surface functionality, and targeted interaction with asphaltene molecules, metal oxide nanoparticles have been increasingly adopted for emulsion control [14]. In this study, nano-clay was employed as a demulsifier to mitigate emulsification in Iraqi turbine oils, with a focus on evaluating the key factors influencing the emulsification behavior and assessing the effectiveness of the nano-clay treatment.

MATERIALS AND METHODS

CharacterizationofNano-Clay

To verify the efficiency of nano-clay as a demulsifying agent, the material was characterized using Transmission Electron Microscopy (TEM) and Dynamic Light Scattering (DLS) techniques. The particle size analysis indicated an average diameter of 48 ± 5 nm with a polydispersity index (PDI) of 0.21, suggesting good dispersion homogeneity. TEM imaging revealed a lamellar sheet morphology, with an average sheet length of approximately 1.5 µm and a thickness ranging from 30 to 70 nm. These structural properties contribute to a high surface area-to-volume ratio, facilitating strong interactions with polar asphaltenes and resin components within turbine oils.The inhibition of emulsification in turbine oils was pursued by either deep filtration of base oils or the introduction of nano-clay as a barrier to emulsion formation.

Oils Used

This study was performed using turbine oil with code number 4006 and base oil 60, both produced by the Dora Refinery. The physicochemical properties of the oils are summarized in Tables 1 and 2, respectively.

Nano-Clay Specifications

The nano-clay utilized in this study was obtained from Sigma-Aldrich and characterized as a white

Table 1. Specifications of Turbine Oil Code No. 4006.

Property	Value
Density (g/cm³) @ 15°C	0.860
Viscosity (cSt) @ 40°C	74
Viscosity Index (min)	95
Flash Point (C.O.C.), °C (min)	210
Pour Point, °C (max)	-12
Neutralization Value (mg KOH/g oil)	0.5

Table 2. Specifications of Base Oil 60.

Property	Value
Density (g/cm³) @ 15°C	0.830
Viscosity (cSt) @ 40°C	65–75
Flash Point (C.O.C.), °C (min)	201
Pour Point, °C (max)	-10
Neutralization Value (mg KOH/g oil)	0.2

to tan powder. The particle diameter ranged from 30 to 70 nm, with a pH value between 4.5 and 7.0 and a particle length between 1–3 µm.

Experimental Procedure

Oxidative degradation is a major factor influencing the emulsification tendency of turbine oils. To assess this phenomenon, oxidation experiments were conducted under controlled, accelerated conditions to evaluate the stability of additive-containing oils compared to untreated base oils.

1.Turbine Oil Oxidation: Turbine oil 4006 was oxidized at 150°C using a copper wire as a catalyst. Air was supplied at a flow rate of 7 liters per minute. Emulsification behavior was evaluated according to ASTM D1401 after 0, 2.5, 5, 10, and 15 hours of oxidation. The separation indices were recorded and are presented in Table 3.

2.Base Oil Oxidation: Base oil 60 was subjected to similar oxidation conditions, with modifications: the oxidation temperature was set at 120°C, and the oxidation duration was limited to 3 hours. Emulsification characteristics were similarly assessed using ASTM D1401.

3.Nano-Clay Treatment: The oxidized turbine oil (after 15 hours) and oxidized base oil 60 were treated with nano-clay at concentrations of 0%, 1%, 2%, and 4% w/w. The emulsification.

RESULTS AND DISCUSSION

Effect of Oxidation Time on Emulsification and Viscosity of Turbine Oil

The emulsification behavior of turbine oil 4006 was markedly influenced by oxidation time. As oxidation progressed, the volume of the residual emulsion layer after 30 minutes of separation increased significantly, rising from 7 mL at 2.5 hours to 64 mL after 15 hours of oxidation, as depicted in Fig. 1. This increase is attributed to the formation and agglomeration of polar oxidation products—resins, asphaltenes, and acidic compounds—which stabilize water droplets within the oil phase.

Additionally, oxidation substantially impacted oil

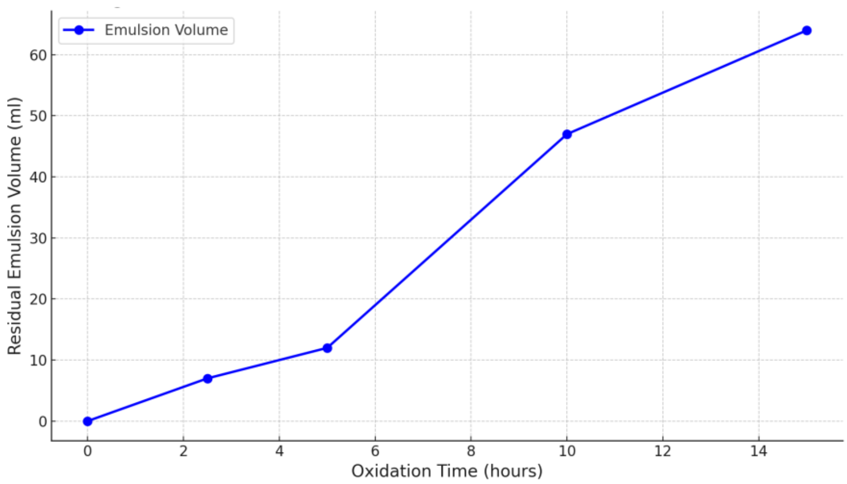

Fig. 1. Relationship between Oxidation Time and Residual Emulsion Volume for Turbine Oil 4006.

Table 3. Viscosity and Emulsion Volume of Turbine Oil 4006 at Different Oxidation Times.

Oxidation Time (hours)	Viscosity at 40°C (cSt)	Emulsion Volume (mL)
0	74	0
2.5	78	7
5	82	12
10	90	47
15	97	64

viscosity. As presented in Table 3, viscosity at 40°C increased progressively with oxidation time. The rise in viscosity is likely due to polymerization and cross-linking reactions of oxidized components, leading to enhanced resistance to water separation. Collectively, these findings indicate that oxidation not only augments emulsification but also alters the physical properties of the oil, thereby complicating water-oil separation.

Notably, a sharp increase in emulsion volume and viscosity was observed between 5 and 10 hours of oxidation, suggesting the presence of a critical point beyond which oil degradation and emulsification accelerate rapidly. This finding highlights the necessity for proactive maintenance schedules to prevent severe oil deterioration under oxidative conditions.

Comparison Between Turbine Oil 4006 and Base Oil 60 in Oxidation Resistance

Base oil 60, oxidized at 120°C for 3 hours, exhibited a significant susceptibility to emulsification, forming a 60 mL emulsion layer after 30 minutes of separation (Table 4). In contrast, turbine oil 4006, even after 15 hours of oxidation at 150°C, showed comparable emulsification volumes. This discrepancy indicates that the absence of antioxidant and emulsification-inhibiting additives in base oil 60 renders it more prone to rapid oxidative degradation and emulsion formation.

Interestingly, the demulsification efficiency of

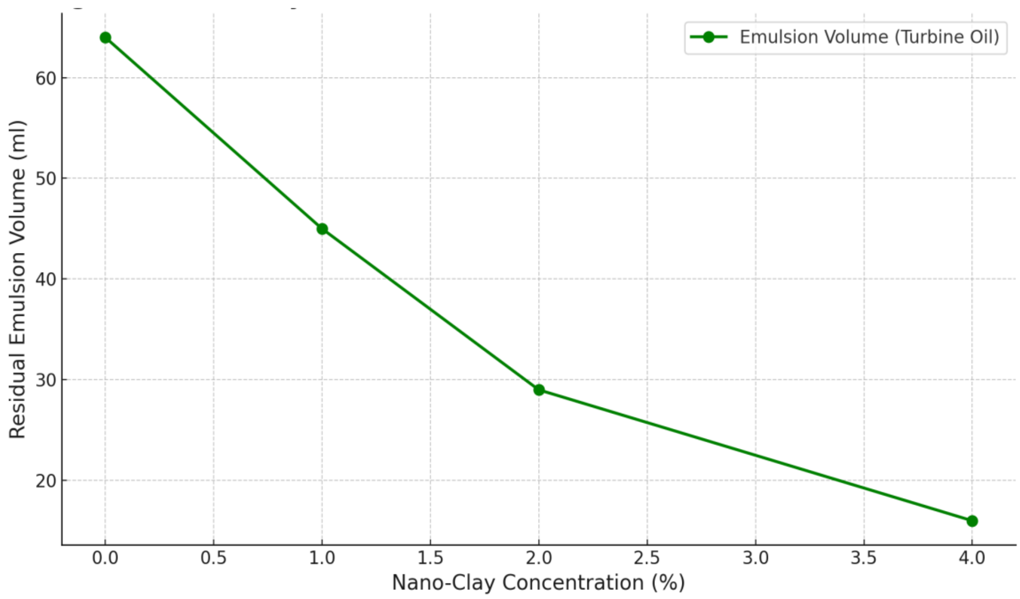

Fig. 2. Effect of Nano-Clay Concentration on Emulsion Volume in Turbine Oil 4006 after 30 min Separation.

Table 4. Emulsification Properties of Base Oil 60 under Oxidation Conditions.

Oxidation Time (hours)	Oxidation Temperature (°C)	Emulsion Volume (mL)
0	120	0
3	120	60

Table 5. Emulsification Properties of Oxidized Turbine Oil 4006 with Nano-Clay Treatment.

Nano-Clay Concentration (%)	Separation after 5 min	Separation after 30 min	Emulsion Layer Volume (mL)
0	1	14	64
1	6	24	45
2	13	30	29
4	19	39	16

nano-clay was more pronounced in base oil 60, likely due to reduced competition from other emulsifying agents present in turbine oil.

Effect of Nano-Clay Concentration on Water-Oil Separation Efficiency

Nano-clay was added at concentrations of 0%, 1%, 2%, and 4% (w/w) to both oxidized turbine oil and base oil 60. As shown in Figs. 2, Fig. 3, and Table 5 increasing nano-clay concentration markedly improved the water-oil separation efficiency. The emulsion layer volume decreased with higher nano-clay loadings, with the optimal separation achieved at 4% nano-clay addition.

For oxidized turbine oil 4006, the emulsion volume reduced to 16 mL at 4% nano-clay, whereas for base oil 60, it dropped to 7 mL under the same conditions.

Also, changes was noticed with different concentration as seen in Table 6 and Fig. 3

Structural Characterization of Nano-Clay by SEM

To validate the morphological features of the nano-clay, a Scanning Electron Microscope (SEM) analysis was conducted. The resulting micrograph (Fig. 4) reveals the characteristic layered and flaky sheet structure of the particles, with variable thickness between 30–70 nm and lateral dimensions reaching up to 1–3 µm. This structural trait increases the surface-to-volume ratio, enabling effective interaction with asphaltenes and resins that contribute to emulsion stability.

Structural Analysis of Nano-Clay by XRD

To further assess the structural properties of the nano-clay used in this study, X-ray diffraction (XRD) analysis was performed. The XRD pattern shown in Fig. 5 reveals a characteristic diffraction

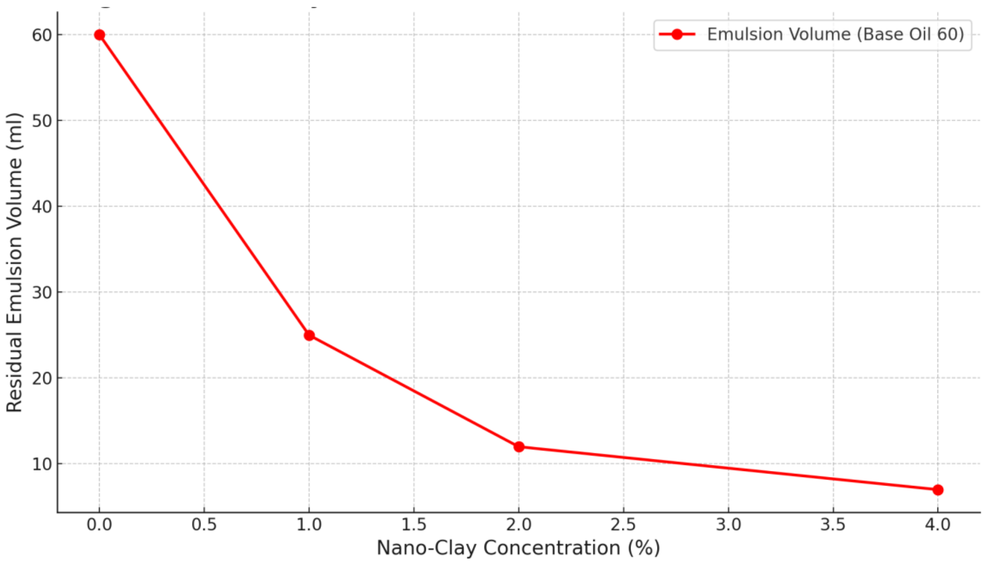

Fig. 3 Effect of Nano-Clay Concentration on Emulsion Volume in Base Oil 60 after 30 min Separation.

Table 6. Emulsification Properties of Oxidized Base Oil 60 with Nano-Clay Treatment.

Nano-Clay Concentration (%)	Separation after 5 min	Separation after 30 min	Emulsion Layer Volume (mL)
0	1	16	60
1	12	31	25
2	15	39	12
4	20	41	7

peak centered at 6.9°, corresponding to the (001) plane of the clay layers. This peak is indicative of the interlayer spacing between silicate sheets, confirming the nanolayered structure of the clay particles. Additional minor peaks observed in the range of 10° to 30° suggest a semi-crystalline structure commonly associated with natural clay minerals.

Surface Tension Reduction with Nano-Clay Treatment

A notable effect of nano-clay treatment was the reduction in surface tension, which facilitates the coalescence of water droplets, promoting faster

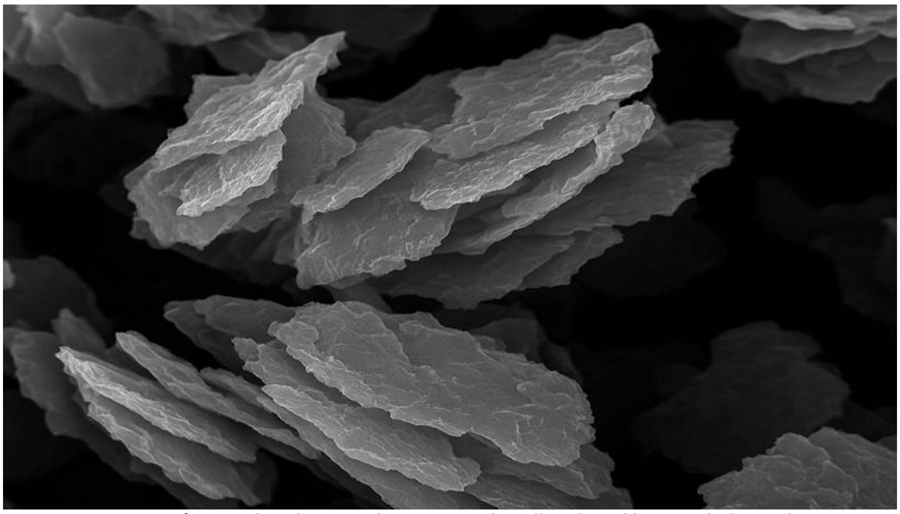

Fig. 4. SEM image of nano-clay shows a characteristic lamellar sheet-like morphology. This structure provides a high surface area that enhances adsorption of polar components in turbine oils, contributing to its demulsification efficiency.

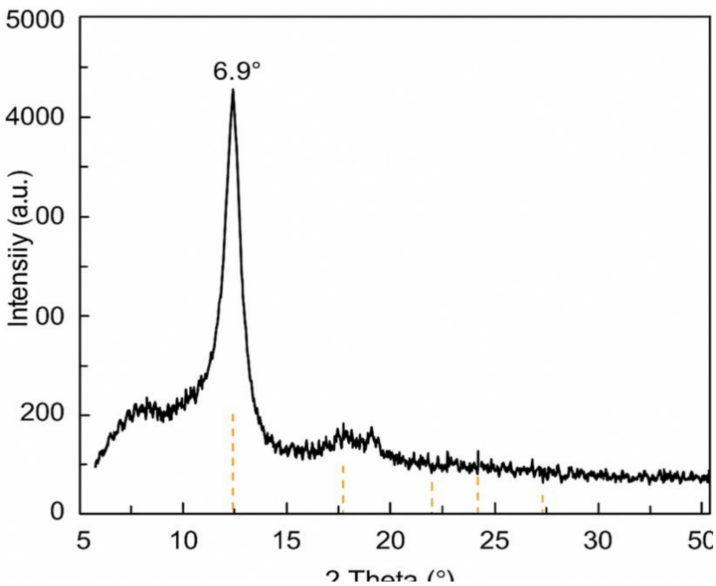

Fig. 5. XRD pattern of nano-clay displaying a prominent diffraction peak at 6.9°, which corresponds to the basal spacing of the layered silicate structure. The sharpness and intensity of this peak confirm the crystalline lamellar morphology of the nano-clay.

separation. As reported in Table 7, nano-clay more effectively reduced the surface tension in base oil 60 compared to turbine oil 4006, likely due to the simpler composition of base oil.

Water Droplet Size Distribution Before and After Nano-Clay Treatment

Nano-clay treatment not only enhanced separation but also altered the droplet size distribution. As shown in Table 8 and Fig. 6, the average water droplet size increased significantly post-treatment, thereby promoting droplet coalescence and more efficient water-oil separation.

Effect of Particle Size and Morphology on Demulsification Performance

The nanoscale dimensions and lamellar morphology of the nano-clay were pivotal in enhancing demulsification performance. Particles smaller than 50 nm provided higher surface energies and greater active sites for the adsorption of emulsifying compounds, facilitating rapid droplet coalescence. The observed lamellar structures and low polydispersity indices contributed to stable dispersions, ensuring consistent demulsifier performance through combined physical and chemical interactions.

Mechanism of Nano-Clay as a Demulsified

Nano-clay functions as a demulsifier by adsorbing polar compounds such as asphaltenes and resins that stabilize water droplets. By disrupting the interfacial film surrounding droplets, nano-clay promotes coalescence and accelerates separation. Van der Waals forces and

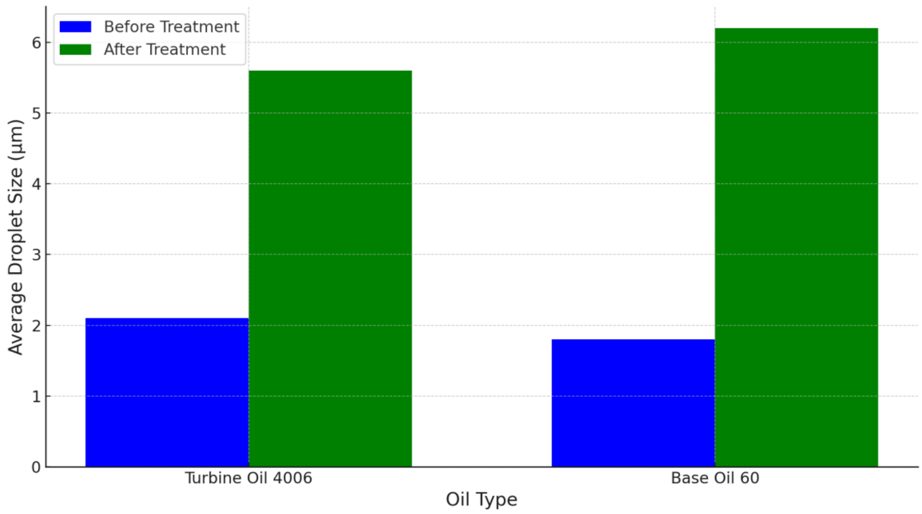

Fig. 6. Average Water Droplet Size Before and After Nano-Clay Treatment for Turbine Oil 4006 and Base Oil 60.

Table 7. Surface Tension Reduction with Nano-Clay Treatment.

Nano-Clay Concentration (%)	Surface Tension Reduction (mN/m) for Turbine Oil 4006	Surface Tension Reduction (mN/m) for Base Oil 60
0	0	0
1	2.5	3.0
2	4.2	5.0
4	6.7	7.5

Table 8. Average Water Droplet Size Before and After Nano-Clay Treatment.

Oil Type	Average Droplet Size Before Treatment (µm)	Average Droplet Size After Treatment (µm)
Turbine Oil 4006	2.1	5.6
Base Oil 60	1.8	6.2

hydrogen bonding further enhance this process, as evidenced by the reduction in surface tension and increased droplet sizes post-treatment. Thus, nano-clay effectively enhances both the interfacial and bulk properties of emulsified oil-water systems, establishing it as a promising candidate for demulsification applications.

CONCLUSION

The findings of this study clearly demonstrate the effectiveness of nano-clay in reducing emulsification and enhancing water-oil separation in both oxidized turbine oil 4006 and base oil 60. Based on the experimental results, the following conclusions can be drawn:

1. Turbine oils exhibit a critical oxidation threshold, beyond which both emulsification and viscosity increase sharply, significantly hindering water separation.
2. Nano-clay significantly reduces surface tension and promotes the coalescence of water droplets, thereby accelerating phase separation.
3. The demulsification performance of nano-clay is more pronounced in base oils lacking additives, suggesting that the presence of emulsifying agents in turbine oils may interfere with nano-clay's interaction with polar oxidation products.
4. Further research is recommended to explore the long-term stability and industrial applicability of nano-clay treatments. In addition, investigating the synergistic or antagonistic interactions between nano-clay and various oil additives in complex formulations could provide deeper insights into optimizing demulsifier formulations for diverse operational environments.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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