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DESIGN AND EVALUATION OF A NANOFLUID CONTAINING MAGNETITE AND GRAPHENE OXIDE NANOCOMPOSITES TO PREVENT ASPHALTENES PRECIPITATION IN POROUS MEDIA

DISEÑO Y EVALUACIÓN DE UN NANOFLUIDO CON NANOCOMPOSITOS DE OXIDO DE GRAFENO Y MAGNETITA PARA PREVENIR LA PRECIPITACIÓN DE ASFALTENOS EN EL MEDIO POROSO

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ABSTRACT

The precipitation and destabilization of asphaltenes in mature oil fields pose significant challenges to the oil industry, leading to damage on the producing formation and the potential blockage of production facilities. Conventional removal and inhibition methods have proven to be costly and temporary, with no guarantee of preventing new asphaltene deposits. In this study, our focus is on the design and evaluation of a stable nanofluid composed of Fe_3O_4 (Magnetite) and GO (Graphene Oxide) nanocomposites as an effective asphaltene stabilization agent in Colombian crude oil. By functionalizing magnetite nanoparticles with graphene oxide, we achieved a significant improvement in their stability. Various surfactants were tested to enhance the nanoparticle stability, followed by viscosity modification using commercial additives to attain the desired stability. The stability of the nanofluid was thoroughly evaluated through Oliensis, SARA, flocculation, rheology, and physicochemical analyses. Additionally, fluid-fluid and fluid-rock interaction tests were conducted under reservoir conditions. The results demonstrated an impressive 48% reduction in asphaltene-induced damage and a positive change in wettability, leading to a remarkable 38% increase in the recovery factor observed in core analyses. This study provides a promising approach to mitigate asphaltene-related challenges in mature oil fields, ensuring formation integrity and enhancing production efficiency.

RESUMEN

La precipitación y desestabilización de asfaltenos en campos petroleros maduros representan desafíos significativos para la industria petrolera, porque conlleva daños a la formación productiva y el potencial bloqueo de las facilidades de producción. Los métodos convencionales de eliminación e inhibición de asfaltenos han demostrado ser costosos y temporales, sin garantía de prevenir nuevos depósitos de estos. En este estudio, nuestro enfoque se centra en el diseño y evaluación de un nanofluido estable compuesto por nanocompuestos de Fe_3O_4 (magnetita) y GO (óxido de grafeno) como un agente eficaz de estabilización de asfaltenos en un crudo colombiano. Al funcionalizar nanopartículas de magnetita con óxido de grafeno, logramos una mejora significativa en su estabilidad. Se probaron varios surfactantes para mejorar la estabilidad de las nanopartículas, seguido de una modificación de la viscosidad mediante aditivos comerciales para lograr la estabilidad deseada. La estabilidad del nanofluido y su capacidad de inhibición fue evaluada exhaustivamente a través de análisis Oliensis, SARA, flocculación, reología y fisicoquímicos. Además, se realizaron pruebas de interacción fluido-fluido y fluido-roca bajo condiciones de yacimiento. Los resultados demostraron una impresionante reducción del 48% en los daños inducidos por asfaltenos y un cambio positivo en la mojabilidad, lo que llevó a un notable aumento del 38% en el factor de recobro, que fue observado en análisis de núcleos (Coreflooding). Este estudio proporciona un enfoque prometedor para mitigar los desafíos relacionados con asfaltenos en campos petroleros maduros, garantizando la integridad de la formación y mejorando la eficiencia de producción.

KEYWORDS / PALABRAS CLAVE

Magnetite | asphaltene inhibition | nanoparticles | nanocomposites | porous media | iron oxide | graphene oxide | increase viscosity | coreflooding.

Magnetita | inhibición de asfaltenos | nanopartículas | nanocompuestos | medios porosos | óxido de hierro | óxido de grafeno | aumento de viscosidad.

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1. INTRODUCTION

As global energy demand continues to grow, efforts are intensifying to explore novel renewable energy sources, including nuclear, solar, and wind power. However, their collective contribution remains insufficient to fulfill current global energy needs. In the forthcoming two decades, renewable energy is anticipated to complement existing sources (Rezvani et al., 2018). Consequently, securing an adequate energy supply is a substantial challenge for the hydrocarbons industry.

Nonetheless, traditional methods of crude oil production, along with primary and secondary recovery techniques, have encountered vulnerabilities in recent years. Furthermore, the recurring issue of asphaltene destabilization and precipitation in mature oil fields has conventionally been managed through expensive and temporary removal or inhibition approaches. Unfortunately, these methods can induce undesirable alterations in fluid behavior and do not assure prevention of future asphaltene deposition. Moreover, these procedures often entail intricate and costly equipment, potentially failing to adequately safeguard the producing formation and thus resulting in severe and intricate damage.

Asphaltenes, the heaviest constituents of petroleum fluids, exhibit insolubility in light paraffins while being soluble in light aromatic hydrocarbon solvents like toluene, benzene, or pyridine (Akbarzadeh, 2007). Addressing these challenges, this study zeroes in on the functionalization of Fe_3O_4 nanoparticles with GO (graphene oxide) nanoparticles and assesses their potential as agents for stabilizing asphaltenes in oil fields. Metallic nanoparticles such as Fe_3O_4 are susceptible to magnetic instability, leading to agglomeration and sedimentation, thereby hindering their uniform application in oil formations. However, by leveraging graphene oxide GO as a stabilizer for Fe_3O_4 nanoparticles, their suspension stability is augmented, effectively curbing agglomeration and precipitation. Furthermore, graphene oxide GO amplifies the capacity of these nanocomposites to stabilize asphaltenes (Setoodeh et al.,2018).

This research means a promising stride towards enhancing the hydrocarbons industry, tackling energy supply predicaments, and mitigating the recurring uncertainty linked to asphaltene destabilization in mature oil fields. The utilization of functionalized nanoparticles introduces new avenues for sustainable and efficient oil production, concurrently minimizing formation impairment, and ensuring stability amidst the fluctuations in energy requirements.

2 THEORICAL FRAMEWORK

Three mechanisms of formation damage have been discerned, elucidating how asphaltenes curtail the effective mobility of oil. The first mechanism stems from the augmentation of reservoir fluid viscosity, attributed to the concurrent production of water and oil, which results in the formation of water-in-oil emulsions. These emulsions cause an upsurge in viscosity. Moreover, experimental measurements have substantiated that asphaltene flocculation leads to a rise in viscosity (Li, X., Chi, P., Guo, X., & Sun, Q. 2019).

The second mechanism involves altering the formation's wettability, transforming it from water-wettable to oil-wettable. This transformation arises from the adsorption of asphaltenes onto the surface of high-energy rock minerals, as shown in Figure 1. Processes governed by ionic interaction and surface precipitation have been identified as the primary contributors to the modification of mineral surface wettability when subjected to asphaltene precipitation in the presence of water (Al-Maamari, R. S., & Buckley, J. S. (2003). Nonetheless, this phenomenon of wettability alteration is improbable in asphaltene fluid reservoirs already exhibiting mixed or oil wettability. This is due to the prolonged adsorption of asphaltenes on the mineral surface over geological timescales prior to well drilling for production.

Wetting within Pores: In a water-wet scenario (left), oil remains confined to the center of the pores. The converse holds true if all surfaces are oil-wet (right). In the case of mixed-wettability (middle), oil has displaced water from some surfaces but retains its position at the centers of water-wet pores. Despite these conditions,
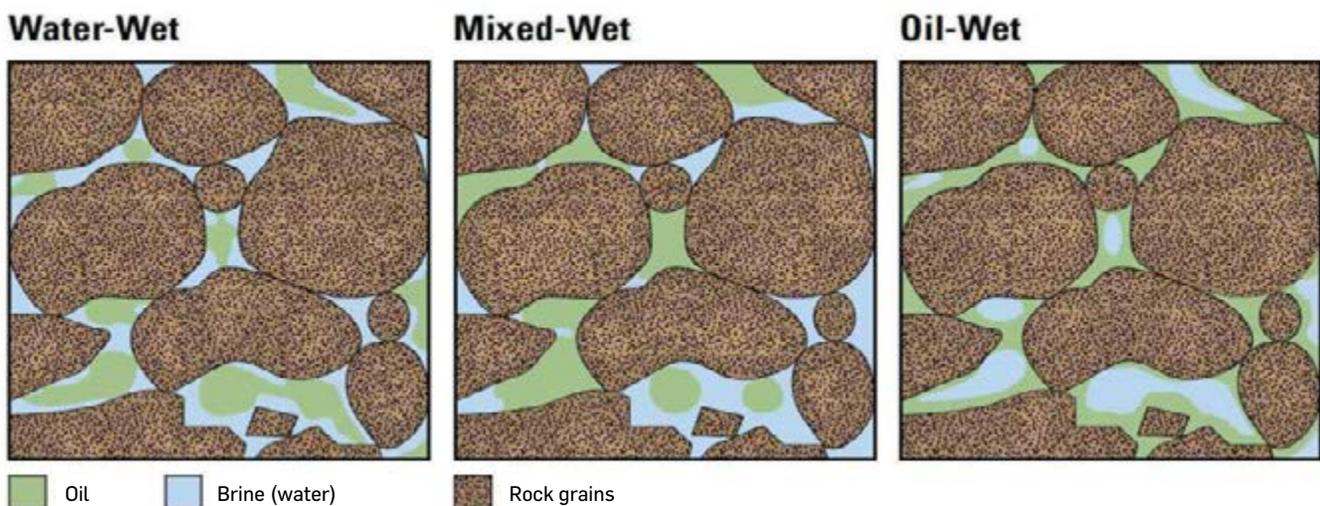

Oil　　Brine (water)　　Rock grains

Figure 1. Rock Formation Wettability Types

the three scenarios exhibit comparable water and oil saturations. (Source: https://www.slb.com/resource-library/oilfield-review/defining-series/defining-wettability).

The third most likely mechanism of formation damage entails a reduction in formation permeability due to pore throat obstruction by growing asphaltene particles.

Furthermore, aside from clogging porous media, asphaltenes lead to obstructive issues in pipelines, wellheads, and valves, significantly impeding the flow of crude oil. These challenges carry significant economic repercussions for the oil industry (Maqbool et al., 2009).

Numerous approaches have been proposed to avert asphaltene precipitation, particularly in proximity to wells (Mansoori, G. A., & Elmi, A. J. S. P. (2010). Some of these methods involve intermittent solvent treatments or continuous chemical injections. These techniques span chemical, mechanical, and biological processes, among others (Civan, F., 2023).

Chemical interventions encompass the addition of dispersants, coagulants, flocculants, and non-polar solvents, aimed at managing asphaltene precipitation (Mansoori, G. A., & Elmi, A. J. S. P. (2010). Various surfactants and solvents have been tested, including toluene (T), nonylphenol (NP), dodecyl benzene sulfonic acid (DBSA), and dodecyl resorcinol (DR). Among these, the order of inhibitory potency against asphaltene precipitation with solvent addition was found to be: DR > DBSA > NP > R > T. The strong inhibitory impact of surfactants was attributed to the interaction between the acidic components of the molecules and asphaltenes (Ghloum et al.,2010). In another study, the effect of three commercial and three non-commercial inhibitors was investigated on four crude samples. The results indicated that basic inhibitors were more effective in most crudes due to their high resin content (75%), while acid inhibitors showed greater efficacy in other crudes due to their high polarity and compatibility with the asphaltene fraction.

Mechanical interventions include manual cleaning, scraping, and mechanical vibration, among others. Manual cleaning is among the oldest known methods for removing heavy hydrocarbon deposits (Mansoori, G. A., & Elmi, A. J. S. P. (2010) Pigs can effectively clean tubes and lines, but are less effective for eliminating heavy organic deposits in the formation. These treatments are often costly and limited to production facilities.
Heat treatment techniques encompass on-site combustion, steam injection, hot water injection, hot gas injection, hot chemical injection, microwave heating, and the use of exothermic chemical reactions, particularly in heavy and extra heavy crudes (Mansoori, 2010). Nonetheless, heat treatments have limitations. Elevated temperatures can cause formation damage if the temperature returns to its initial value post-treatment, leading to significant deposits. Downhole heaters are constrained by maintenance expenses and energy availability. Additionally, heat-releasing chemicals are often costly.

Biological approaches involve on-site application of anaerobic, aerobic bacteria, and other microorganisms, including fungi. The biodegradation mechanism of asphaltenes is the least understood reaction, proceeding relatively slowly and potentially requiring months or years for microorganisms to degrade a substantial amount of asphaltenes (Iraji, S., & Ayatollahi, S. (2019).

The aforementioned methods are typically expensive, yield temporary effects, may alter fluids, and do not assure prevention of new asphaltene deposition. Moreover, they require intricate and often costly equipment, additional personnel on-site, and, occasionally, fail to safeguard the producing formation, exacerbating severe formation damage which is even more challenging to rectify (Civan,F., 2023).

3. STATE OF THE TECHNIQUE

Recently, the utilization of nanoparticles has gained traction as an alternative chemical approach to address issues associated with asphaltene precipitation. Several researchers (Nassar, 2010) (Franco et al., 2013) (Al-Jabary et al., 2007) (Mohammadi, et al., 2011) (Cortes et al., 2012) (Kasemzadeh, et al., 2015) have investigated the adsorption of asphaltenes utilizing nanoparticles. Nassar explored the adsorption of asphaltenes on colloidal - Al2O3 nanoparticles. Various batch adsorption experiments were conducted at different asphaltene concentrations and temperatures. The author observed rapid adsorption, with equilibrium achieved after approximately 2 hours. However, these studies did not assess the impact of particle size or conduct displacement tests under typical reservoir conditions. Additionally, Mohammadi et al., 2017 examined the potential of TiO_2, ZrO_2, and SiO_2 nanoparticles dissolved in organic solvents to stabilize asphaltene particles in crude oil, functioning as precipitation inhibitors and dispersants. They noted that TiO_2 nanoparticles can exhibit opposite behavior under basic conditions. The dynamic light scattering (DLS) technique was used to elucidate the mechanism of asphaltene precipitation in the presence of nanoparticles. However, there was no evidence from studies conducted under reservoir conditions.

Cortés et al. (2012) synthesized NiO nanocrystals supported on silica nanoparticles. The researchers observed that, at a consistent temperature, the adsorption of asphaltenes into the hybrid nanoparticles increased with NiO content. They attributed the strong asphaltene-nanoparticle affinity to the nanoparticles' pronounced hydrophilicity and the effective dispersion of NiO on silica nanoparticles. In particular, they found that irrespective of asphaltene concentration, adsorption on the hybrid nanoparticles' surface decreased with rising temperature, suggesting an exothermic nature of the process. Additionally, (Kazemzadeh et al. 2015) evaluated SiO_2, NiO, and Fe_3O_4 nanoparticles for asphaltene precipitation inhibition. They observed that systems containing n-heptane without nanoparticles facilitated asphaltene aggregation. Conversely, in the presence of nanoparticles, an increase in n-heptane content augmented the adsorbed quantity of asphaltenes on nanoparticles in the following order: SiO_2 > NiO > Fe_3O_4. Furthermore, micromodels were used to assess crude oil recovery in the presence of nanoparticles. Micromodels proved instrumental in comprehending Enhanced Oil Recovery (EOR) processes at the pore scale and in observing fluid behavior within porous media.

APPLICATION OF MAGNETITE IN THE ASPHALTENE INHIBITION

Magnetite (Fe_3O_4) is an iron oxide mineral composed of 56 atoms including 32 oxygen, 16 Fe^{+3} and 8 Fe^{+2} and can be denoted as $(Fe^{+3})_8^{Td}$ $[Fe^{+3} \ Fe^{+2}]_8^{Oh} \ O_{32}$. The oxygen atoms form a face-centered cubic (FCC) lattice, and the iron atoms are located in 32 octahedral and 64 tetrahedral sites (Gomez-Robayo., 2019).

(Al-Jarabi et., 2007) investigated the influence of Fe_3O_4 nanoparticles on the removal of asphaltenes from crude oil. Nanoparticles with sizes ranging between 20 and 30 nm were assessed in n-heptane/

toluene systems. The assessment revealed the removal of approximately 80% of asphaltenes, with higher adsorption observed as nanoparticle concentration in the system increased. Moreover, it was demonstrated that around 95% of asphaltenes could be removed after 1.5 hours using magnetite nanoparticles.

(Kazemzadeh et al., 2015) evaluated various mass fractions of Fe_3O_4, ranging from 0.05 to 1.0%, and found that the highest concentration of magnetite examined exhibited the most effective inhibition of asphaltenes. Additionally, it was determined that asphaltene characteristics, particularly in terms of structure, profoundly impact the performance of nanoparticles in terms of stabilization.

(Setoodeh, et al.,2018) assessed magnetite nanoparticles and magnetite functionalized with various materials, including GO. They were able to ascertain that magnetite nanoparticles possess asphaltene adsorption capacity, which was significantly enhanced when magnetite was functionalized with GO.

GRAPHENE OXIDE (GO)

Structurally, in its pristine form, graphene is a crystalline material made up of sp2-hybridized carbon atoms that are densely packed in an hexagonal lattice similar to a honeycomb (Terrones et al., 2010). In a more simple manner, it can be defined as the two-dimensional allotrope of carbon, or an isolated layer of many that are found stacked in a graphite crystal (Gomez Robayo, 2019).

Initially, graphene was conceptualized as a theoretical model to elucidate the genesis of diverse carbon allotropes. The prevailing notion held that these sheets couldn't be isolated without succumbing to atomic vibrations that prevail at room temperature. However, in 2004, Andre Geim and Konstantin Novoselov defied this notion by ingeniously using adhesive tape to exfoliate graphite into remarkably high-quality graphene sheets. Their groundbreaking discovery was documented in the journal Science (Novoselov, et al., 2004), which merited them the 2010 Nobel Prize in Physics, recognizing their pioneering efforts in procuring, identifying, and characterizing this exceptional material.
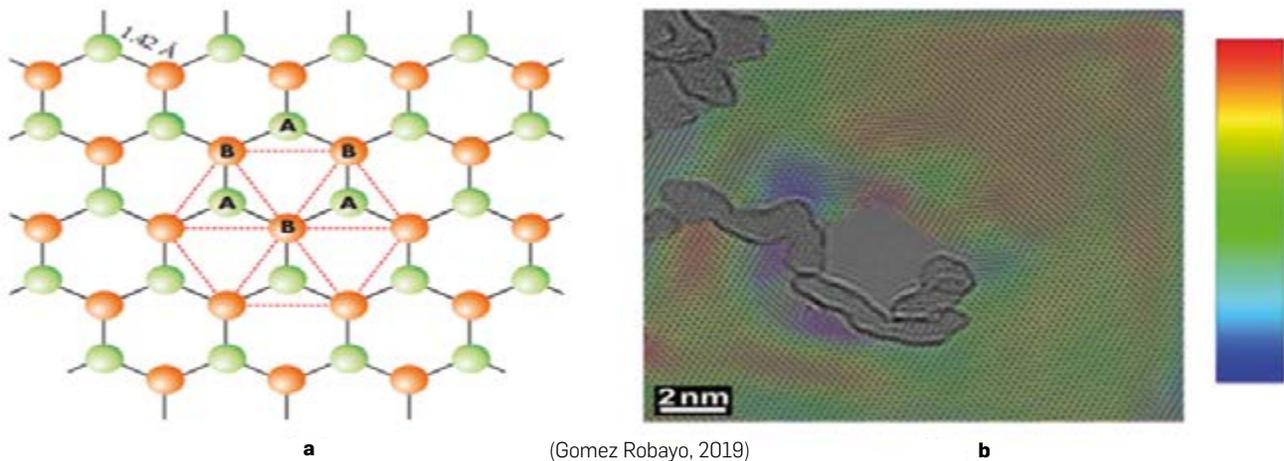

a   (Gomez Robayo, 2019)   b

Figure 2. a) Model of the crystalline structure of graphene, b) HR-TEM image of a graphene sheet, where the color scale is associated with the distance observed in the C=C bonds
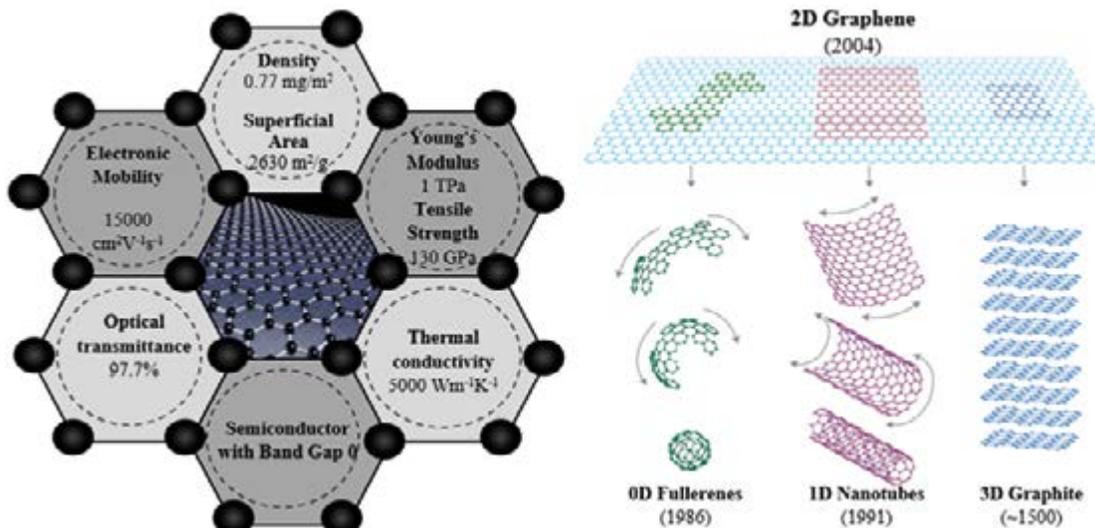

Note. This figure represents the data collected in the experimental phase.

Figure 3. Properties of pristine graphene and its modeling as a structural block of other carbon allotropes

At present, it has been established that graphene attains stability due to its vibrations being harmoniously absorbed within ripples exhibiting ~1 nm amplitudes across its lattice. This distinct behavior arises from a vibrational instability inherent in its crystalline structure (Martínez-Guerra et al 2009).

NANOFLUID

A nanofluid constitutes a liquid medium that encompasses suspended particles at the nanometer scale. Commonly used nanoparticles encompass metal oxides, carbides, or carbon nanomaterials, while the carrier fluids comprise water or hydrocarbons like ethylene glycol, diesel, or crude oil. In the context of this project, a diesel-based fluid was used as the foundation, enriched with the intention of enhancing nanoparticle stability. This enhancement was achieved through a targeted viscosification process, facilitated by specialized commercial additives used in Hydraulic Fracturing operations with base fluids. These fluids are indispensable when it is necessary to fracture formations sensitive to water.

OLIENSIS TEST

Crude oil has the potential to undergo instability and precipitate asphaltenes under specific conditions, primarily influenced by pressure but also involving temperature and concentration changes A thorough characterization of the crude is imperative to assess its stability, which serves as the basis for implementing necessary measures to avert asphaltene precipitation during the productive phase of oil well operations.

Numerous experimental techniques exist to identify the onset of asphaltene precipitation in a crude sample, such as gravimetric methods, acoustic resonance, and near-infrared light scattering. However, for pragmatic purposes in production settings, the smear test method, commonly known as the Oliensis test, is frequently used. This method swiftly and effectively indicates whether a given crude oil exhibits a highly stable, unstable, or moderately stable precipitation tendency.

In this study, the Oliensis test was conducted in laboratory conditions as a preliminary method to evaluate the performance of nanofluids containing magnetite and graphene oxide (GO) nanoparticles for inhibiting asphaltene precipitation. While the test is qualitative and less suitable for detailed quantitative comparison, it is still valuable for observing general trends in asphaltene stability and aggregation. To address this limitation, core flooding tests were used as the primary quantitative method. These tests provide a more rigorous evaluation of the nanofluids' performance under reservoir conditions, aligned with the study's scope of assessing real-world applicability. The combination of the Oliensis test and core flooding experiments ensures a comprehensive evaluation framework, with the former offering practical insights and the latter delivering robust, quantitative measurements under reservoir-representative conditions.

The Oliensis technique implies observing the marks formed by a droplet of the sample (crude + toluene + (heptane or pentane)) on a filter paper (Figure 4). As the droplet permeates the paper, it diffuses radially. If asphaltenes are evenly dispersed, the stain retains a uniform hue, accompanied by a darker central halo upon drying. In contrast, if asphaltenes have flocculated or aggregated into larger entities, their diffusion through the paper is sluggish, yielding a central aureole with varying degrees of clarity, contingent on the

case. When further diluted with an alkane (n-pentane, n-hexane, or n-heptane) after flocculation, the aureole can evolve from a faint trace to a small, dark circle, indicating non-instantaneous precipitation.

The flocculation point hinges on temperature, pressure, and the asphaltenes' nature, as well as the stability of the micelle. Greater non-polarity in the medium, exemplified by heightened aliphatic content, enhances resistance to detachment from the nonpolar milieu, thereby intensifying association to an extent that could be infinite. This, in turn, fosters the formation of flocculates and consequent precipitation. (D1370, 2015).
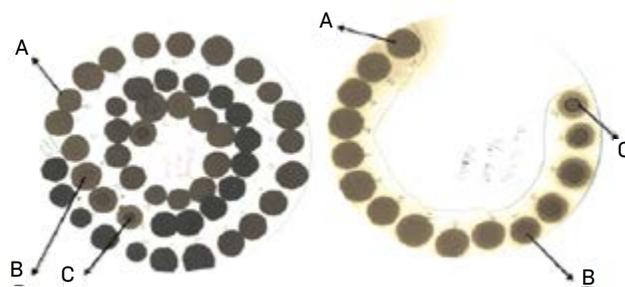

Figure 4. Spot test or Oliensis test

Spot test or Oliensis test- A. Dispersed asphaltenes B. Asphaltenes beginning to flocculate C. Flocculated and precipitated asphaltenes (ASTM D1370-00) (Asomaning, S. (2003))

COMPATIBILITY TEST

These evaluations are conducted under reservoir temperature conditions to assess the potential for emulsion or precipitate formation during the interaction of the treatment with formation fluids. Mixtures consist of the treatment, crude oil, and formation water in ratios of 20:80, 50:50, and 80:20 (treatment to oil and formation water). These mixtures are placed in hermetically sealed glass flasks, manually shaken for one minute, and then heated to reservoir temperature for two hours.

After the heating period, the flasks are removed from the oven, and detailed observations are made regarding the mixture's appearance and interface. This includes evaluating phase separation, the presence of precipitates, viscosity changes, and potential emulsion formation. The methodology adheres to the API standard (API RP-42, 1990).

For immiscible phases, compatibility is determined based on the following criteria:

- Phase separation exceeds 80%.
- The aqueous phase remains free of crude oil.
- No emulsions are present or prone to form.
- The interface remains well-defined without the appearance of new phases or solid layers.

For miscible fluids, compatibility is confirmed if their mixing does not result in color changes, increased viscosity, or precipitate formation.

COREFLOODING TEST

The core flooding test on plug samples (core flooding) is carried out with the objective of simulating, under dynamic and reservoir conditions (pressure and temperature), the natural wettability and the response of the rock to the different displacements that are carried out, performed with injection and stimulation fluids to generate, remove, and inhibit asphaltene damage. In this study, a plug (native rock sample) from a Colombian field was selected to perform the evaluation.

1. Sample Saturation: Saturate the sample with brine until 99% of the pore volume is reached. Brine Injection:
2. Inject brine at 1 ml/min until stability is achieved over 10 pore volumes, with a maximum pressure differential variation of 5%.
3. Crude Injection: Inject crude oil at 1 cc/min until stability is reached over 10 pore volumes, with a maximum variation of 5%.
4. Waterflooding 1: Inject 100 pore volumes of brine and measure the effective water permeability at rates of 1 ml/min and 1.5 ml/min to rule out Threshold Pressure phenomena.
5. Asphaltene Damage Generation: Inject asphaltene-rich crude oil that has been pre-treated with n-pentane to reach the flocculation point and precipitate asphaltenes through the porous medium.
6. Brine Injection Post-Damage: Inject brine to determine the effective water permeability after asphaltene damage.
7. Crude Injection Post-Damage: Inject crude oil to determine the effective oil permeability.
8. Second Waterflooding: Inject brine and conduct waterflooding to determine the effective water permeability.
9. Removal Agent Injection: Inject the removal agent treatment, using a carrier fluid without nanoparticles.
10. Waterflooding 3: Inject brine and determine the effective water permeability at two rates.
11. Crude Injection: Inject crude oil to determine the effective oil permeability.
12. Asphaltene Inhibition Treatment: Inject an asphaltene inhibition treatment using a nanofluid with nanoparticles at a concentration of 200 ppm.
13. Second Asphaltene Damage Attempt: Inject asphaltene-rich crude oil to attempt a second asphaltene damage scenario.
14. Waterflooding 4: Inject brine and determine the effective water permeability at two rates.
15. Crude Injection: Inject crude oil to determine the effective oil permeability.
16. Waterflooding 5: Inject brine and determine the effective water permeability at two rates.

4. EXPERIMENTAL DEVELOPMENT

PREPARATION AND STABILIZATION OF NANOFLUID

Nanofluid preparation constitutes a pivotal stage in nanofluid assessments, with two predominant methodologies used in its formulation. The first is the one-step approach, entailing the direct generation of nanoparticles within the carrier medium. For instance, nanoparticles are formed through the evaporation and condensation of nanomaterials directly within a liquid phase. Alkhavan et al., 2015, utilized chemical vapor deposition to synthesize nanofluids containing multi-walled carbon nanotubes in deionized water.

The second technique adopted for this study involves the prior synthesis of nanoparticles followed by their dispersion in the base fluid. This methodology is known as the two-step method (Kaggwa & Carson, 2019). The principal challenge associated with this method is achieving the stability of nanomaterials within the carrier medium. The incorporation of surfactants has been a widely employed strategy to attain stabilization, aimed at diminishing the interaction forces between nanoparticles and the carrier medium. Additionally, the solution needs to be homogenized using ultrasound and high-speed mixers (Yu, W., & Xie, H. 2012). Industrially, the two-step approach has gained prominence due to its reduced labor intensity and enhanced cost-effectiveness (Haddad, et al 2014). The stabilization of metallic nanoparticles presents even greater challenges, primarily due to their high surface area-to-mass ratio and magnetic attraction properties (Setoodeh, et al., 2018).

In this study, the initial effort to mitigate nanoparticle instability involved the addition of surfactants. Moreover, augmenting the viscosity of the carrier medium was essential to confer stability upon the nanoparticles within the fluid matrix. To this end, commercially available agents commonly employed in oil-based hydraulic fracturing fluids were used. Considering that the nanofluid in this study is intended for application in wells prone to organic flake precipitation, particularly asphaltenes, it is imperative for the nanofluid to exhibit substantial stability. This stability is vital to ensure prolonged suspension within the carrier medium, allowing effective transportation from the surface to the reservoir for subsequent injection. The table below presents the final formulation of the nanofluid after achieving the nanoparticles stabilization.

Table 1. Nanofluid formulation

Additive	Function	Concentration	Units
Diesel	Organic Base	55.90	%
Xylene	Organic solvent	38.00	%
Mutual solvent green line	Mutual solvent	4.00	%
Surfactant green line	Anionic surfactant	2.00	%
Viscosifying agent 1	anionic phosphate ester	0.08	%
Viscosifying agent 2	Anionic phosphate ester optimized	0.04	%
GO@Fe$_3$O$_4$ 2:1	Nanoparticles	200	ppm

Note: These data were collected from the study

The process undertaken to develop the carrier fluid for this project is outlined below. Initially, the following formulation was blended: 223.6 mL of Diesel, 152 mL of Xylene, 16 mL of mutual solvent, and 8 mL of non-ionic surfactant. This mixture was introduced into a 500 mL beaker and stirred consistently at 400 RPM. The mutual solvent and surfactant used in this assessment are characterized as green line, signifying they are new generation additives with high biodegradability, as per the OECD 301 test. These additives have little impact on both humans and the environment when compared to traditionally used red line products, such as diethylene glycol monobutyl ether. This concoction was labeled "mixture 1" accordingly.

Subsequently, a series of solutions was prepared, each containing 50, 100, 200, 300, and 500 ppm concentrations, composed of "mixture 1" and GO@ Fe$_3$O$_4$ nanoparticles in a 2:1 ratio. These samples underwent sonication for 50 minutes using a 240 W power sonic bath. The same procedure was repeated with a 1:1 ratio of

GO@Fe$_3$O$_4$ nanoparticles. All of these mixtures were subjected to stability assessment using visual inspection over a 12-hour period. However, despite these efforts, persistent instability of nanomaterials within the carrier fluid was observed.

In response, a second set of mixtures was prepared, using the two prior nanofluids and subjecting them to sonication with an ultrasonic probe operating at a frequency of 40%. Ultimately, it became evident that the nanofluids still remained unstable, thus requiring reformulation. Images illustrating the observed nanofluid stability in this section are provided in figures 5 to 8.
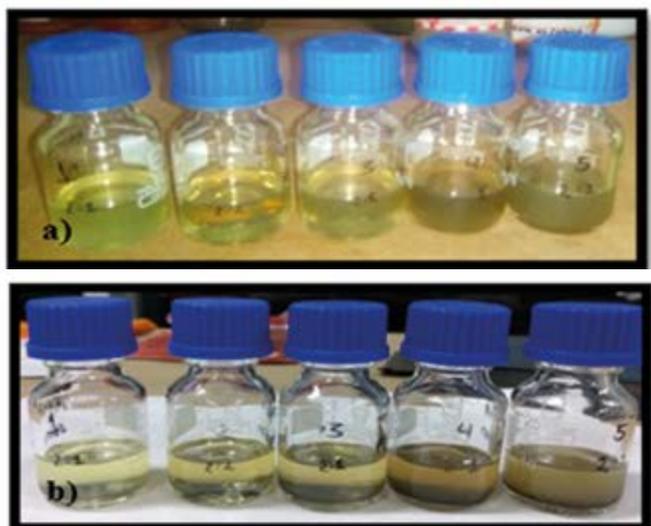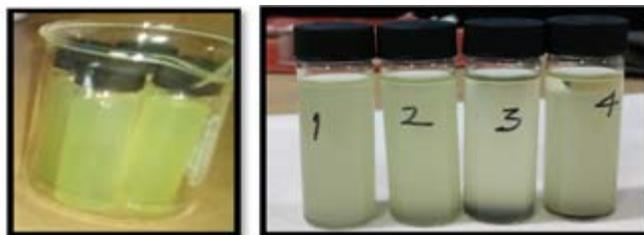

Note. This figure represents the data collected in the experimental phase. Precipitated nanoparticles are observed at the bottom of the container.

Figure 5. Stability tests performed using ultrasonic bath (GO@Fe$_3$O$_4$ 2:1) a) t=0s, b) t=12h

Note. This figure represents the data collected in the experimental phase. Precipitated nanoparticles are observed at the bottom of the container.

Figure 6. Stability tests performed using ultrasonic bath (GO@Fe$_3$O$_4$ 1:1) a) t=0s, b) t=12h

Note. This figure represents the data collected in the experimental phase. Precipitated nanoparticles are observed at the bottom of the container.
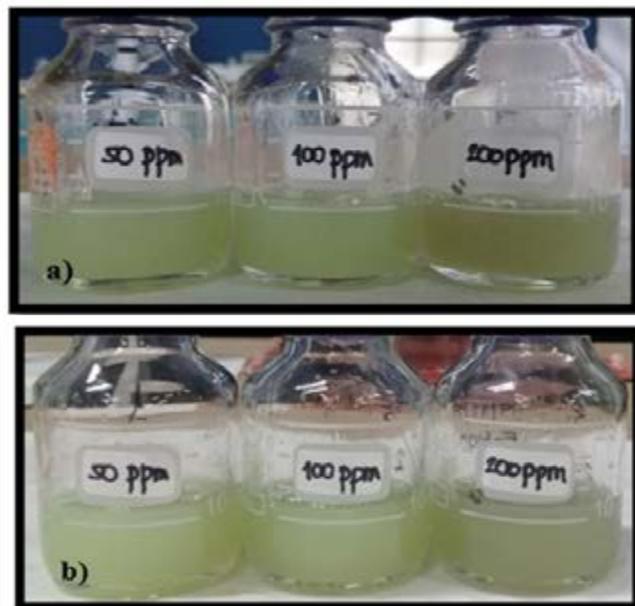
Figure 7. Stability tests performed using ultrasonic probe (GO@Fe$_3$O$_4$ 2:1) a) t=0s, b) t=12h

Note. This figure represents the data collected in the experimental phase. Precipitated nanoparticles are observed at the bottom of the container.

Figure 8. Stability tests performed using ultrasonic probe (GO@Fe$_3$O$_4$ 1:1) a) t=0 s, b) t=12h

It was also significant that the results showed that GO@Fe$_3$O$_4$ 2:1 nanocomposite and mixed with ultrasonic probe presented slightly better stability than GO@Fe$_3$O$_4$ 1:1, therefore, it was decided to continue the evaluations with the former. After observing poor instability in the nanofluid in spite of using an ultrasonic probe for mixing, it was necessary to reformulate the carried fluid obtaining the final formulation shown in table 1. The mixing procedure is presented below. 139.75 mL of diesel was placed in a 500 ml beaker and stirred at room temperature at a speed of 400 rpm using a triple-blade propeller mixer (d/D: 0.5), then 95 mL of xylene was added, subsequently 10 mL of mutual solvent, next 5 mL of non-ionic surfactant was added, then 0.2 mL of viscosifying agent 1 (anionic phosphate ester) and 0.1 mL of viscosifying agent 2 (anionic phosphate ester optimized) was added, it was continued mixing for 5 minutes until obtain a viscous fluid, finally GO@Fe$_3$O$_4$ 2:1 nanocomposites were added in concentrations of 50, 100 and 200 ppm, to verify steadiness of the nanofluid; subsequently, a stability test was performed, finding that the nanofluid was stable after 12 hours. The images of the visual observation of stability are presented below.

Note. This figure represents the data collected in the experimental phase. Precipitated nanoparticles are not observed at the bottom of the container.

Figure 9. Stability tests to nanofluid with three different GO@Fe$_3$O$_4$ concentration 50,100 and 200ppm a) t=0 s, b) t=12h

ASPHALTENE STABILITY TEST

Subsequently, obtaining the stability of the nanoparticles within the transport agent, Oliensis tests were implemented to assess the performance of the nanofluid in stabilizing asphaltenes under laboratory conditions. First, the stability of the crude (blank) was evaluated, then the stability of the crude plus transport agent, finally the nanofluid was evaluated with concentrations of 50, 100 and 200ppm of GO@Fe$_3$O$_4$ 2:1 nanocomposite.

To conduct the Oliensis test, five solutions were prepared: transport agent, blank, nanofluid within 50, 100 and 200 ppm of GO@Fe$_3$O$_4$. The mixing procedure is presented below: Transport agent, which is the nanofluid without nanoparticles; the required quantities of every additive for 400ml of treatment are presented below. Diesel 139.75 mL; Xylene 95mL; mutual solvent 10mL; non-ionic surfactant 5mL; 0.2 mL viscosifying agent 1; 0.1 mL viscosifying agent 2 were added to a 500 mL beaker under constant stirring at 400 RPM.

Blank, to obtain this solution, 5 g of Colombian oil was mixed with 5 g of toluene in a beaker under magnetic stirring beaker under constant stirring at 400 RPM.

Nanofluid within 200ppm of GO@Fe$_3$O$_4$, to obtain this solution, 0.05 g of GO@Fe$_3$O$_4$ was mixed with 250 mL and in a beaker under magnetic stirring beaker under constant stirring at 400 RPM; then the nanofluid was sonicated using an ultrasonic probe. To prepare the other two solutions (50 and 100ppm of GO@Fe$_3$O$_4$), it was necessary to dilute from this nanofluid.

First, the blank was evaluated following the procedure below.
- A drop of blank was taken using a syringe and deposited in the center of a 20 to 45 µm cellulose filter paper.
- Then, 4 mL of analytical n-pentane was added to blank, and after 1 min a drop was taken and deposited onto the filter paper. This step was repeated twice. placing the drop forming a spiral.
- Then, 1 mL of analytical n-pentane was added to the blank, and after 1 min a drop was taken and deposited onto the filter paper. This step was repeated until the appearance of a halo.
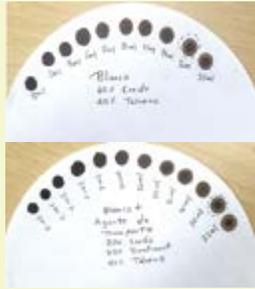- The test was labeled blank, 50% oil and 50 toluene.

Second, the nanofluid oil was assessed once again.
- The 5g nanofluid was mixed with 5g of oil, and added to a 50 mL beaker under magnetic stirring.
- A drop of this solution was taken using a syringe and deposited in the center of a 20 to 45 µm cellulose filter paper.
- Then, 4 mL of analytical n-pentane was added to the solution, and after 1 min, a drop was taken and deposited onto the filter paper. This step was repeated twice, placing the drop forming a spiral.
- Then, 1 mL of analytical n-pentane was added to the solution, and after 1 min, a drop was taken and deposited onto the filter paper. This step was repeated until a halo appeared.

Third, the last procedure was repeated for the nanofluid within 50, 100 and 200ppm of GO@Fe$_3$O$_4$.

The results of these evaluations are shown in the table below.

Table 2. Oliensis Test Results

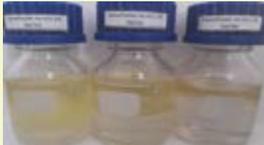| Photographic record of blank and transport agent	Concentration
	For the blank, the precipitation of the asphaltenes occurred when 16 mL of n-pentane was added. For the transport agent, the precipitation of the asphaltenes occurred when 20 mL of n-pentane was added.
Photographic record of nanofluid within 50 and 100ppm of GO@Fe3O4 2:1	Observation
	For the nanofluid within 50ppm, the precipitation of the asphaltenes occurred when 22 mL of n-pentane was added. For the nanofluid within 100ppm, the precipitation of the asphaltenes occurred when 24 mL of n-pentane was added.
Photographic record of nanofluid within 200ppm of GO@Fe3O4 2:1	Observation
	For the nanofluid within 200ppm, the precipitation of the asphaltenes occurred when 26 mL of n-pentane was added

Note: These data were collected from the study

The results observed through the Oliensis test show that as the concentration of nanocomposites increases, the stability of the asphaltenes improves. For the blank it was necessary to add 16mL of n-pentane to destabilize the asphaltenes, while for the nanofluid within 200ppm concentration of nanocomposites (GO@Fe$_3$O$_4$ 2:1) it was necessary to add 26mL of n-pentane. Although the Oliensis results present this crude with a low probability of a tendency to precipitate asphaltenes, the field evidence shows that this problem exists, so it was decided to use the crude to present this system as an alternative treatment to solve it.

COMPATIBILITY TEST

Then, a compatibility test was implemented to determine the absence of any noise associated to incompatibility between the nanofluid and the well fluids; the evaluations were performed among nanofluid with 200ppm concentration of nanocomposites (GO@Fe$_3$O$_4$ 2:1) and both with synthetic brine and crude oil in proportions of 20/80, 50/50 and 80/20. The following table shows the observed results.

Table 3. Compatibility Test

Compatibility test Nanofluid vs Crude	Observation
	The presence of a single phase is observed, without manifestation of emulsions or precipitates.
Compatibility test Nanofluid vs synthetic brine	Observation
	The presence of two phases is observed, 100% break between phases, no emulsions or precipitates are evident; clear, and defined interface.

Note: These data were collected from the study

COREFLOODING TEST

The core flooding is considered the main test evaluation for this study, since the articles and the evaluations that were reviewed, showed that the magnetite nanoparticles have the potential to inhibit the asphaltenes precipitation, and, once the stability problems of the nanoparticles within the nanofluid had been overcome, the evaluation at reservoir conditions was developed.

Table 4. The composition of the synthetic brine used for the evaluation.

Additive	Function	Concentration	Units
KCl	Cay inhibitor	1	%

Note: These data were collected from the study

The viscosity of the synthetic brine was estimated using correlations for aqueous solutions, which allow estimating the viscosity of a brine at a given temperature and pressure. The viscosity value for the 1% KCl brine @ 180°F and 1000 psi system pressure is 0.350 cP.

DEAD OIL FROM COLOMBIAN WELL

Crude oil was obtained from a Colombian well with the following characteristics: 47% BS&W, 20o API, SARA analysis (Saturates=38.76%, Aromatics=24.01%, Resins=27.38% and Asphaltenes=9.85%) with colloidal instability index (CII = 0.94), (CII> 0.9 unstable asphaltenes); the crude oil was dehydrated until reaching BS&W = 1%, filtered, and injected into the sample to bring it to irreducible water saturation (Swirr). The rheological behavior of the crude oil was observed at the test temperature of 180°F; whose viscosity value was 267 cP.

NATIVE PLUG

For the evaluation of the asphaltene precipitation inhibitor treatment, at the core flooding level, a sample of native rock from the Colombian field formation was used. Table 6 shows the petrophysical properties of the sample used.

PROTOCOL ASPHALTENE INHIBITION EVALUATION IN COREFLOODING

The following figure shows step by step the evaluation conducted to determine the nanofluid performance in asphaltenes inhibition.

CONSIDERATIONS FOR FIELD DEPLOYMENT OF NANOTECHNOLOGY-BASED TREATMENTS

Field deployment of nanoparticle-based nanotechnology, such as that described in this paper, requires careful consideration of well conditions, economic feasibility, and the specific goals of the intervention. While this paper primarily focuses on laboratory and reservoir-condition evaluations, potential field application methods include bullheading the nanofluids into the formation, or deploying them via coiled tubing, depending on the well's configuration and operational constraints. It is recognized that nanotechnology-based treatments can be more expensive to apply compared to traditional methods. However, the potential economic return is closely linked to the durability and long-term effectiveness of these nanofluids. In previous applications performed in specific fields operated by ECOPETROL, it has been observed that the extended lifespan of such nanofluids can lead to substantial economic benefits, justifying the higher initial investment. Therefore, any implementation of nanotechnology must include a comprehensive economic analysis, incorporating factors such as well productivity, treatment longevity, and operational costs. Such analysis is essential to determine the feasibility and optimization of deploying these advanced nanofluids in field settings. Future studies and applications should aim to bridge the gap between laboratory results and field operations by integrating both technical and economic evaluations.

Table 5. SARA test & CII Colloidal instable index

Crude	% Saturates	% Aromatics	% Resins	% Asphaltenes	Viscosity (cP)	Molecular Weight
Crude	38.76	24.01	27.38	9.85	267/ 180 °C	350 g/mol
Colloidal instability Index (CII)			0,94 if CII>0,9 Asphaltenes are unstable.			

Note: These data were collected from the study

Table 6. Native plug petrophysical properties

Overburden (psi)	Length (cm)	Diameter (cm)	Porous Volume (mL)	Porosity %	Klinkenberg Permeability (mD)	Air permeability (mD)
2300	6.797	3.745	15.871	21.4	2740	2777

Note: These data were collected from the study
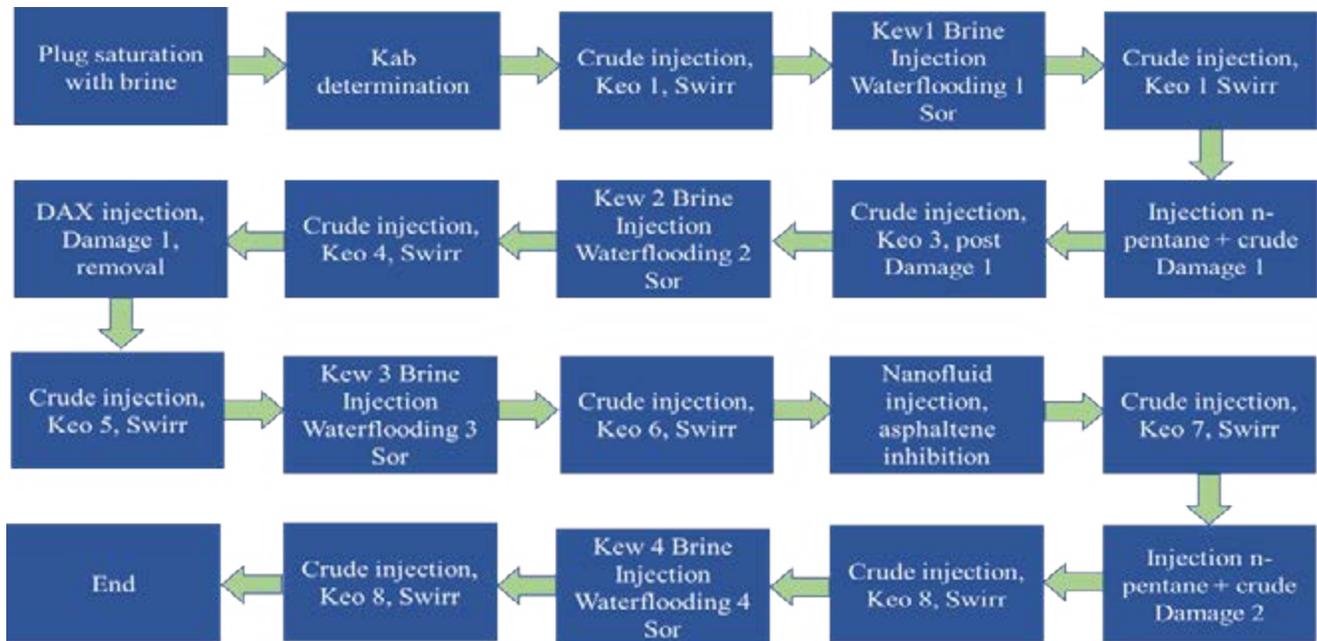
Note. This figure represents the data collected in the experimental phase

Figure 10. Core flooding evaluation protocol

5. RESULTS ANALYSIS

COREFLOODING RESULTS

The following two tables provide the results of the evaluation of the inhibitor in the native core plug at reservoir conditions.

Table 7. The composition of the synthetic brine used for the evaluation.

Parameter	Water Permeability (mD)	Oil Permeability (mD)
Kw	1779,93	
Keo1(Base Line)		1461,9
Kew1(Waterflooding 1)	165	
Keo2		1380
Keo3(Post damage 1)		553.7
Kew2 (Waterflooding 2)	455,02 ↑ 176%	
Keo4 (before remotion)		458,7 ↓ 66.8%
Keo5 (Post remotion)		1125
Kew3 (Waterflooding 3)	225.7	
Keo6 (Post remotion)		896 ↑ 95.3%
Keo7 (Post Nanofluid)		909
Keo8 (Post damage 2)		1022
Kew4 (Waterflooding4)	254.1 ↓ 44.2%	
Keo9 (final result)		989 ↑ 115.6%

Note: These data were collected from the study

It is evident that after damage induction 1 by asphaltene precipitation, the effective permeability to oil decreased significantly, dropping from 1461.9 mD to 458.7 mD. Conversely, the effective permeability to water increased from 165 mD to 455.02 mD, which is primarily associated to the increased oil-wet tendencies of the rock caused by asphaltene precipitation. After applying the removal treatment (carrying fluid without nanoparticles), the effective oil permeability partially recovered to 896 mD, while the water permeability decreased to 254.1 mD, reflecting improved fluid flow conditions. Upon subsequent injection of the nanofluid treatment, and a second damage induction, the effective oil permeability further improved to 989 mD, demonstrating the nanofluid's capacity to mitigate asphaltene precipitation effects. These results highlight the dual effectiveness of the carrying agent in damage removal, and the nanofluid's role in inhibiting subsequent organic precipitation.

Table 8. Summary of Relative Permeabilities Results

Parameter	Baseline (Dimensionless)	Post-Damage (Dimensionless)	Post-Removal (Dimensionless)	Post-Inhibition (Dimensionless)
Swirr	0,1935	0,1494	0,2817	0,3636
Sor	0,2520	0,4726	0,2646	0,1134
Krw end point	0,0928	0,2556	0,1268	0,1428
Kro end point	0,8213	0,3111	0,6320	0,5742
Recovery Factor	0,5545	0,3780	0,4537	0,5230

Note: These data were collected from the study

From the relative permeability and fluid saturation data, the rock's initial recovery factor was 55.45%. After the first damage event caused by asphaltene precipitation, the recovery factor decreased, reflecting diminished oil displacement efficiency. Upon application of the removal treatment, the recovery factor improved to 45.37%. Following the injection of the asphaltene inhibitor nanofluid, the recovery factor approached the baseline value, reaching 52.3%,
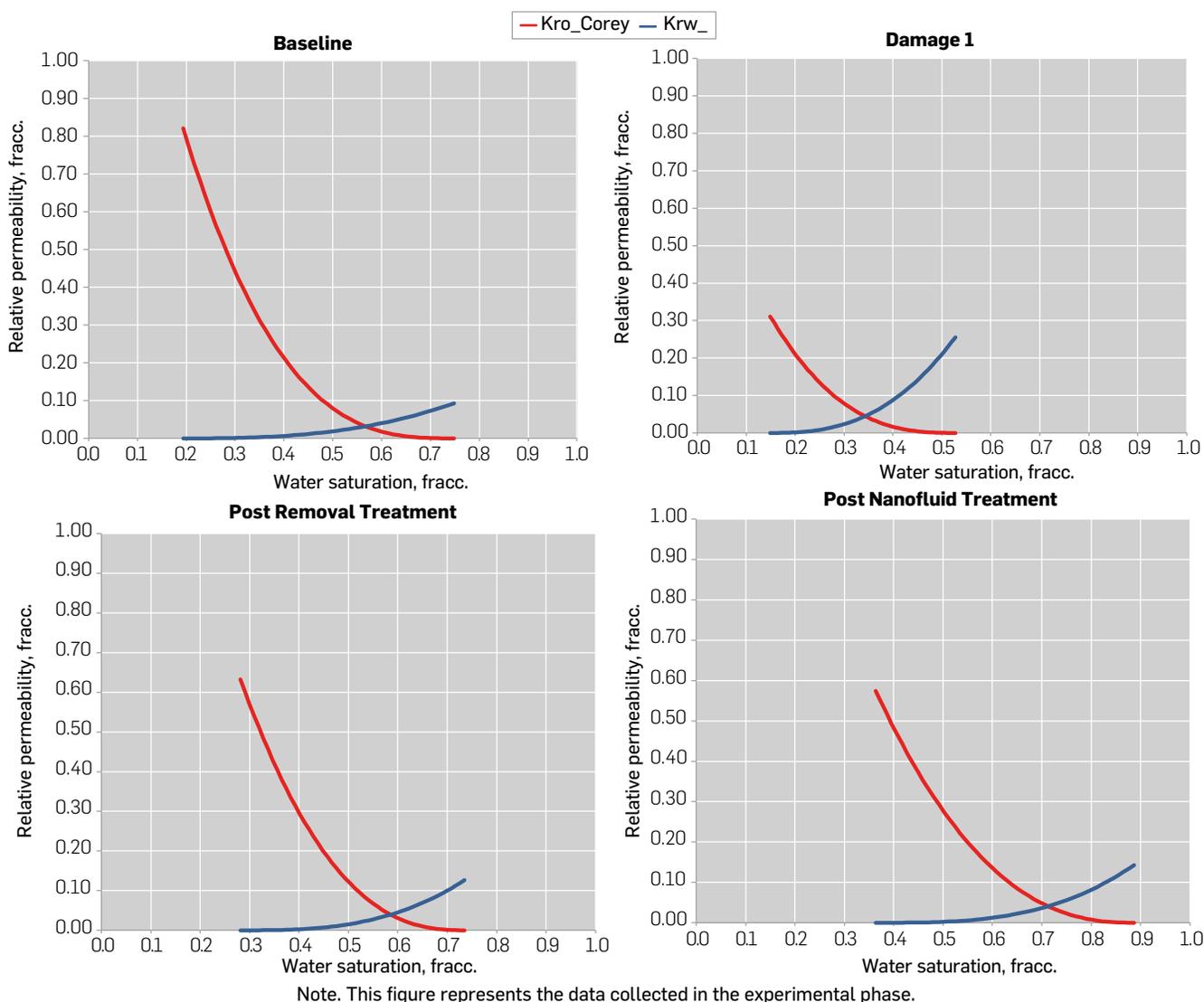
Figure 11. Summary of Relative Permeability Results

suggesting successful mitigation of asphaltene damage and restoration of rock properties.

Relative permeability curves further validate this progression; after the first damage event, the curve shifted left, demonstrating increased oil-wet behavior due to asphaltene deposition. The removal treatment shifted the curve rightward, indicating a restoration of water-wet tendencies. The final injection of the nanofluid caused a pronounced rightward shift, suggesting enhanced water-wet behavior due to the adsorption of nanoparticles, which altered the rock's surface properties.

CONCLUSIONS

1. The nanofluid with magnetite nanoparticles and functionalized with graphene oxide (GO@Fe_3O_4 2:1) developed and evaluated showed good performance as asphaltene inhibitor.
2. It was evidenced that the transport agent treatment performed well in removing organic formation damage, as it was able to recover the effective permeability to oil by 84.57% after the generation of damage caused by asphaltene precipitation.
3. The nanofluid is able to turn the wettability of the rock to wet water even after the generating formation damage for precipitation of asphaltenes.
4. Graphene oxide and magnetite nanocomposites were synthesized; these were suspended and stabilized in a Diesel-based transport medium (Carrier), using green line surfactants and commercial viscosifiers such as anionic phosphate ester, and additives used in hydraulic fracturing technologies.
5. Implementing nanofluid on a field scale through pilots at reservoir conditions similar to those evaluated in this study, with asphaltene precipitation problems, to establish the effectiveness of the nanofluid at this scale.

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NOMENCLATURE

GO   *Graphene Oxide.*

SARA   *Saturate, Aromatic, Resin and Asphaltene is an analysis method that divides crude oil components according to their polarizability and polarity.*

CII   *Colloidal instable index.*

mD   *The darcy (or darcy unit) and millidarcy (mD) are units of permeability.*

Kew   *Water Permeability.*

Keo   *Oil Permeability.*

Krw   *Water relative permeability.*

Kro   *Oil relative Permeability.*

RF   *Recovery Factor.*

Sor   *Residual oil saturation.*

Swirr   *Irreducible water saturation.*