



Polymer Integrated Silica Nanofluid Synthesis: Unveiling new frontiers of Performance Enhancement in EOR

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Abstract

Various enhanced oil recovery (EOR) methods are widely used in today's oil fields. These EOR methods facilitates in retrieving the additional oil more prominently than primary and secondary methods. However, proficient knowledge about efficient fluid displacement, distribution and analysis of porosity and permeability of the reservoir is essential to conduct these EOR methods in the field. Various researches on nanoparticles for EOR are being conducted and in this research, we dive into the polymer integrated silica nanofluid and its potential.

The research is based on synthesizing silica nanofluids (SiNF) instead of purchasing commercially. Sol-Gel method was used to synthesize SiNF for this experiment. After synthesis the paper employs Dynamic Light Scattering (DLS) to measure zeta potential and particle size - crucial for nanofluid stability. Once stability is achieved, the study evaluates the nanofluid's effectiveness in EOR through a progressive series of tests measuring wettability, surface tension, and interfacial tension (IFT). Detailed analysis of the collected data underscores the efficiency of this innovative approach, marking it as a highly promising advancement in the field. Subsequent Scanning Electron Microscopy (SEM) tests further corroborate the potential of the nano particles. Further the Rheology analysis helps us understand the change in viscosity and viscoelastic properties which gives the potential for improve oil recovery by altering the flow characteristics. Overall, the wealth of data amassed promises to provide invaluable insights that could revolutionize EOR practices.

Keywords: Dynamic Light Scattering, Contact Angle Goniometer, Nanofluids, Surface Tension, Interfacial Tension, Scanning Electron Microscope

Introduction

Nanofluids are suspension of colloidal solution whose size range is < 100 nanometer (nm). Nowadays silica nanofluids are of great importance in the field applications for EOR processes. Silica is mostly preferred due to its lower cost and easy application in surface modification. The use of nano fluids is highly appreciated these days due to its various properties. Nano particles are ideal for high pressure and temperature zones such as the reservoirs. Researchers have also found various use of silica nano particles in EOR processes [1-2]. This paper also studies the use of silica nanofluid synthesized with polymer PAM. Mostly it uses the SOL-GEL method to synthesize the SiNF instead of purchasing Silica Nano particles commercially [3-4-5].

The research conducted emphasizes in improving Oil recovery from the pore spaces that primary and secondary recovery methods are unable to produce [6-7]. For that, a stable nanofluid is prepared using a magnetic stirrer and few chemicals such as Ethanol, TEOS (Tetraethyl orthosilicate) which is the precursor and NH_3 (Ammonia) which is the catalyst. The synthesis is done with a time dilation of two mins for every two drops of TEOS and 6-8 drops of ethanol in PAM solution. Ammonia is added at last in drop-wise manner. This whole process may require ten to twelve hours. After synthesizing the nanofluid DLS test are conducted for checking the stability. If sample is stable then we proceed for further tests but if it is not stable then we repeat the procedure again with different approach in the drop-wise technique [8].

This paper dives into the potential of Silica nano fluids that would be used for recovery of the immobile Oil trapped in the pore spaces after primary and secondary processes. It tries to understand the surface tension, interfacial tension and wettability test through Contact angle Goniometer, and many more. Further SEM analysis gives the visual data showing the actual shape and size of the nano particles [9].

The motivation behind conducting this research lies at the challenges and transformative opportunities within the industry oil production. The contemporary oil industry is characterized by an increasing need to boost oil recovery rates and implement sustainable, efficient practices. Conventional methods have their limitations, often leaving significant quantities of oil unrecovered, leading to the use of Enhanced Oil Recovery (EOR) techniques. In this context, the promise of nanofluids as a game-changing solution has captured the attention of researchers and industry professionals alike [10].

Nanofluids represent a novel approach with the potential to redefine oil production practices. These specially engineered fluids, with nanoscale solid particles dispersed in liquid, offer the possibility of significantly enhancing EOR methods. Among the various nanofluids, silica-based nanofluids have shown promise, but the integration of polymers introduces a new dimension to this technology [11]. This integration opens the door to innovative, high-performance materials that can potentially revolutionize EOR strategies, contributing to the sustainability and efficiency of the oil production industry.

The motivation for this study stems from the belief that polymer-integrated silica nanofluids have the potential to address the industry's pressing challenges. These challenges include the efficient extraction of residual oil, the development of sustainable practices, and the need for materials that can perform under the extreme conditions found in EOR reservoirs. To harness this potential, a detailed investigation is required, focusing on the synthesis, stability, and performance of these nanofluids. This research is driven by the goal of providing invaluable insights that can lead to a transformation in EOR practices, ultimately benefiting the oil industry and contributing to a more sustainable energy future [12-13].

The primary objectives of this research are as follows:

- a) **Synthesis of Polymer-Integrated Silica Nanofluids:** Develop an innovative and robust synthesis method for polymer-integrated silica nanofluids (stable) using the sol-gel technique. This objective is critical to ensure the successful integration of polymers into silica nanofluids, setting the foundation for subsequent assessments [14-15].
- b) **Stability Assessment:** Investigate and establish the stability of the polymer-integrated silica nanofluids. This involves the measurement of zeta potential and particle size using Dynamic Light Scattering (DLS), crucial for ensuring that the nanofluids can perform effectively under challenging EOR reservoir conditions [16].
- c) **Performance Evaluation:** Evaluate the performance of the polymer-integrated silica nanofluids in the context of EOR applications. This includes assessing parameters such as wettability, surface tension, and interfacial tension (IFT) to gauge the nanofluids' effectiveness in improving oil recovery rates [17-18].
- d) **Scanning Electron Microscopy (SEM) Analysis:** Conduct SEM tests to visually inspect the nano-sized particles in the polymer-integrated silica nanofluids, providing further confirmation of their potential for application in EOR [14-19].
- e) **Rheology Analysis:** Rheology experiment was conducted using a rotational rheometer. Rheology analysis helps us to understand the change in viscoelastic properties which gives the potential for improve oil recovery [20].
- f) **Data Analysis and Insights:** Analyse the data collected from the various tests comprehensively. The goal is to identify any consistent, favourable attributes of the nanofluids that could pave the way for their integration into the oil industry, potentially revolutionizing EOR practices [21].

By achieving these objectives, this research aims to unlock new frontiers in performance enhancement and applications within the oil production industry, ultimately contributing to increased oil recovery rates and more sustainable practices in this vital sector.

Materials and Methods

Materials required for synthesis of SiNF:

Magnetic Stirrer, Polyacrylamide (PAM), Tetraethyl orthosilicate (TEOS), NH_3 (Ammonia), Ethanol and droppers are used for the synthesis of SiNF.

Synthesis Process:

The synthesis of nanofluid in our study involves a single-step process utilizing specific chemicals, which are meticulously introduced in a particular order and quantity. We employed the sol-gel method for this synthesis, carrying out three distinct variations while keeping the precursor for SiO_2 constant as TEOS. The solvent used is polymer PAM (Polyacrylamide). The concentration of PAM and the chemical Ethanol is 4:1, TEOS is in ratio of 0.15 with Ethanol and catalyst used is accordingly changed. For 100 ml PAM 25 ml Ethanol and 4 ml TEOS is used and NH_3 1.5 ml and similarly for 80 ml PAM 20 ml Ethanol and 3 ml TEOS is used and NH_3 1 ml.

The synthesis is done with a time dilation of two mins for every two drops of TEOS and 6-8 drops of ethanol in PAM solution. Ammonia is added at last in drop-wise manner. This whole process may require ten to twelve hours. After synthesizing the nanofluid DLS test are conducted for checking the stability. If sample is stable then we proceed for further tests but if it is not stable then we repeat the procedure again with different approach in the drop-wise technique.

We utilized Ammonia as the catalyst, serving the crucial role of initiating hydrolysis and condensation reactions. To achieve the desired basic nature of the nanofluid, we deliberately selected a basic catalyst. This synthesis process is a continuous one, extending over duration of 10 to 12 hours. The addition of chemicals is methodically executed at regular intervals, introducing equal quantities using the drop-wise technique.

The equipment at the core of this process is the magnetic stirrer, a sophisticated apparatus that harnesses a rotating magnetic field to induce rapid spinning of a stir bar immersed within the liquid. This action effectively stirs the contents. The rotating field is generated either through a rotating magnet or a set of stationary electromagnets strategically positioned beneath the liquid vessel. In this device we set the temperature and frequency of the rotation (rpm) per requirement [22].

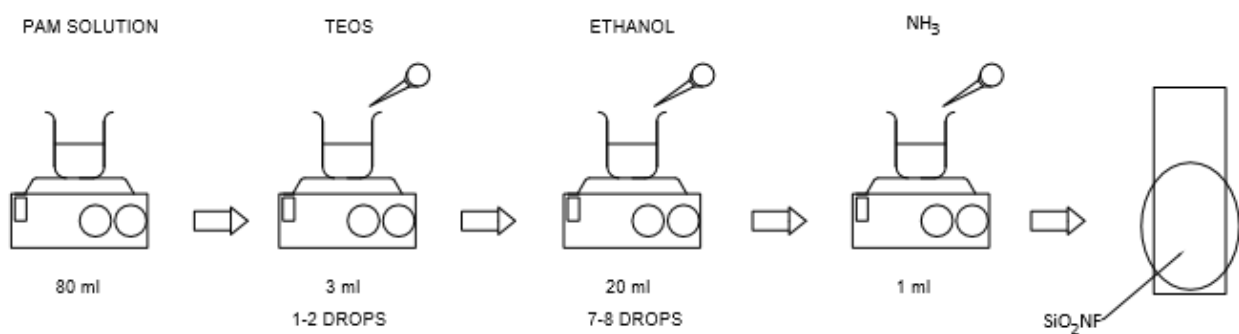


Figure 1. Schematic representation of single-step SiO_2 nanofluid preparation

Particle Size Analysis:

Utilizing a Particle Size Analyzer to measure nanoparticle size and zeta potential is essential in characterizing nanoparticles and their suspensions. It provides valuable insights into the behaviour and stability of nanoparticles. Determining size distribution is critical for assessing nanoparticle performance in various applications. Zeta potential measurements reveal surface charge and stability, aiding in predicting particle interactions, dispersion behaviour, and agglomeration tendencies.

The equipment finds numerous applications in the Oil and Gas sector, particularly in the analysis of emulsions, colloidal systems, and fluid properties. Emulsions are prevalent and their stability is crucial for efficient operations. Measuring droplet size and distribution in emulsions helps optimize formulation and understand stability mechanisms. Zeta potential measurements assess interactions in complex fluid systems like crude oil, water, and additives. This knowledge enhances oil recovery processes, optimizes drilling fluids, and improves oilfield chemical performance.

Particle size significantly influences the physical, chemical, and biological properties of nano particles. Zeta potential represents the electric potential difference between a nanoparticle's surface and the surrounding solution. It serves as an indicator of nano particle surface charge and offers insights into nano particle stability and colloidal behaviour [23].

Wettability Experiment:

Wettability is the property that characterises a liquid's ability to spread or adhere to a solid surface. This experiment can help in selecting and optimizing EOR techniques based on the specific wettability characteristics of the reservoir rock. Understanding whether a reservoir is naturally water-wet or oil-wet helps in choosing appropriate EOR chemicals that might be used to alter the wetting properties to enhance oil recovery. The experiment aims to understand the interaction between a liquid and a solid surface through the measurement of the contact angle at the liquid-solid interface [24].

The basic setup is Sample Preparation, Liquid Selection, Droplet Placement, Image Capture, Contact Angle Measurement and Data Analysis. The Goniometer calculates the contact angle between a liquid and a surface using the Young-Laplace equation [25].

Preparation of Reservoir Surface Condition Pellet using Crude Oil and Sandstone powder:

C3 crude oil was thoroughly mixed with powdered sandstone in a beaker, and the mixture was left to rest for a week. Afterward, the mixture was spread out to create a flat surface for further analysis. To measure the contact angle, a syringe was prepared by fitting it with a spring and filling it with the prepared sample. The syringe was then placed in the apparatus holder. The camera cover was removed, the light switched on, and the camera activated through the ACAM software. The experiment setup involved selecting the number of images to be captured and the interval between them. Once set, the surface was positioned under the needle tip, ensuring the baseline was perfectly aligned on the computer screen. The pin was rotated to release a droplet onto the surface, and measurement was initiated by pressing the 'M' button. After completing the measurement, the data files were saved. In case reanalysis was needed, the boundaries were adjusted, and the process was repeated, followed by saving the newly measured files.

Surface Tension Experiment:

Surface tension is a property of liquids that describes the force at the liquid's interface with its surroundings. It arises from cohesive forces among liquid molecules, causing those at the surface to be more attracted to neighbouring molecules in the bulk of the liquid. The experiment was conducted using a contact angle goniometer. Using a contact angle goniometer to study surface tension involves a slightly different approach than measuring wettability directly. While the primary function of a contact angle goniometer is to measure the angle formed by a liquid at the contact point with a solid, it can also be adapted to indirectly calculate surface tension using specific techniques such as the sessile drop method, pendant drop method, or the drop shape analysis.

In this experiment we use the pendant drop method with a contact angle goniometer to measure surface tension. Equipment and materials needed for the measurement are *contact angle goniometer*: equipped with a high-resolution camera and software capable of analysing drop shape, *the synthesized silica nano fluid*: to be examined, *needle or capillary tube*: for forming droplets of the liquid, *syringe pump*: to control the droplet size precisely, *temperature-controlled environment*: since surface tension is temperature-dependent. Afterwards, the procedure below to measure the surface tension is followed.

To set up the goniometer, the camera and lighting system were mounted to ensure clear visibility of the droplet, with a uniform background providing sufficient contrast for accurate image capture. The liquid sample,

intended for surface tension measurement, was filled into a syringe, to which a needle or capillary tube was attached. The needle was positioned vertically above the camera's field of view, and a droplet was slowly extruded from the syringe pump. Care was taken to ensure the droplet remained hanging at the tip without falling, with its size adjusted for optimal shape analysis, as larger droplets generally facilitate more accurate curvature visualization and measurement. A clear image of the pendant droplet was captured using the goniometer's camera, ensuring that the droplet's silhouette was well-defined. The image was then imported into the goniometer's software for analysis. The software applied a fitting technique based on the Young-Laplace equation, which models the droplet's shape by balancing gravitational and surface tension forces, enabling the calculation of surface tension from the droplet's curvature and dimensions. To ensure data reliability, the measurement was repeated several times, with consistency in droplet formation and measurement being critical for accurate and reproducible results.

Ensure the experiment is conducted under controlled environmental conditions as both temperature and humidity can affect surface tension. Compile the surface tension values obtained across multiple trials. This method of using a contact angle goniometer to measure surface tension is indirect but effective, particularly in laboratory settings where direct measurements might be difficult or require more specialized equipment. The precision of the instrument and the software's ability to accurately model and analyse the droplet shape are crucial in ensuring the reliability of the measurements.

Interfacial Tension (IFT) Experiment:

IFT represents the force between different phases at gas-liquid, gas-solid, liquid-liquid, or liquid-solid interfaces [26]. It is a measure of the force per unit length existing at the interface between two immiscible fluids, such as oil and water. This parameter is crucial because it affects how easily oil droplets can move through a reservoir's pore structure [27-28]. By measuring IFT, researchers can optimize the composition surfactants or other chemicals in injection fluids. The goal is to achieve the lowest possible IFT, enhancing the ability of these fluids to displace oil effectively. It helps in understanding the phase behaviour of fluid mixtures under reservoir conditions. This is critical for designing EOR strategies that involves miscible or near-miscible displacement process [29-30].

The IFT experiment conducted between Silica Nanofluid (SiNF) and crude oil using the Spinning Drop Tensiometer. The result was generated through the software using the device. At first, we need to find the density of Crude Oil and the sample we are measuring i.e. SiNF. We use a Pycnometer to measure the density of CO and the SiNF. Then we simply put the measured values of density in the software for measuring the IFT.

The following procedure was performed to measure the density of the crude oil and SiNF sample which is very important for IFT measurement. The pycnometer was first cleaned and dried thoroughly to ensure accurate measurements. Its empty weight was determined using an analytical balance and recorded as "M1." The pycnometer was then filled with distilled water, and its combined weight was measured and noted as "M2." Afterward, the water was emptied, and the pycnometer was carefully dried. The dry pycnometer was then filled with the crude oil sample, ensuring the sample was at the same temperature as the distilled water. The weight of the pycnometer with the crude oil sample was measured and recorded as "M3."

The density of the crude oil sample can be determined using the following formula:

$$\text{Density of Crude Oil } (\rho) = (M3 - M1) / V_{\text{pycnometer}} + \text{Density of air}$$

$$\text{We have, Volume of pycnometer } (V_{\text{pycnometer}}) \text{ in ml} = (M2 - M1) / D_{\text{water}} - D_{\text{air}}$$

$$[\text{We have, Density of air } (D_{\text{air}}) = 0.001293 \text{ gm/mol}]$$

$$[\text{Density of Distilled Water } (D_{\text{water}}) = 0.99704 \text{ gm/mol}]$$

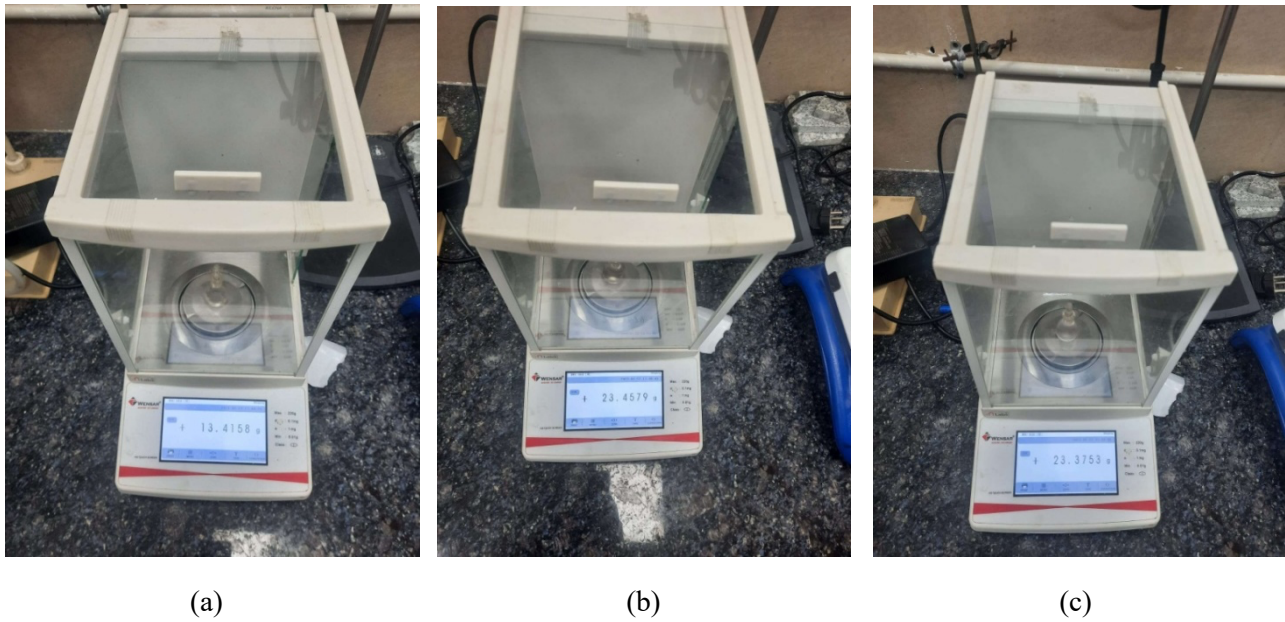


Figure 2. (a) Mass of pycnometer (M_1) = 13.4158g; (b) Mass of pycnometer filled with distilled water (M_2) = 23.4579 g; (c) Mass of pycnometer filled with Si NF Sample (M_3) = 23.3753g

$$V_{\text{pycnometer}} = 23.4579 - 13.4185 / 0.99704 - 0.001293$$

$$= 10.08499 \text{ ml}$$

$$\text{Density of Si NF} = \text{Mass of sample (M3)} - \text{Mass of air (M1)} / V_{\text{pyc.}} + D_{\text{air}}$$

$$= 23.3753 - 13.4185 / 10.0849 + 0.001293$$

$$= 0.988585 \text{ gm/ml}$$

$$= 988.585 \text{ kg}^3$$

Density of Crude Oil = 0.875467 gm/ml (it was known)

Results and Discussion

DLS and Zeta Potential Results:

Table 1. Particle size and zeta potential

Days of Observation	Sample	Day 1	Day 7
Particle Size (nm)	Non-Diluted	1.465	2.853
	Diluted	0.7129	0.6579
Zeta Potential (eV)	Non-Diluted	-39.9	-43.4
	Diluted	-50.1	-40.6

The zeta potential of the nanoparticles was measured to be approx. -40 mV, indicating a high degree of surface charge which suggests excellent colloidal stability under physiological conditions. Repeated measurements across two different batches showed less variability in both size and zeta potential, confirming the reproducibility of the nanoparticle synthesis process. The DLS analysis confirmed that the synthesized nanoparticles possess a narrow size distribution and high zeta potential, characteristics that are favourable for stable suspensions and effective for use in EOR.

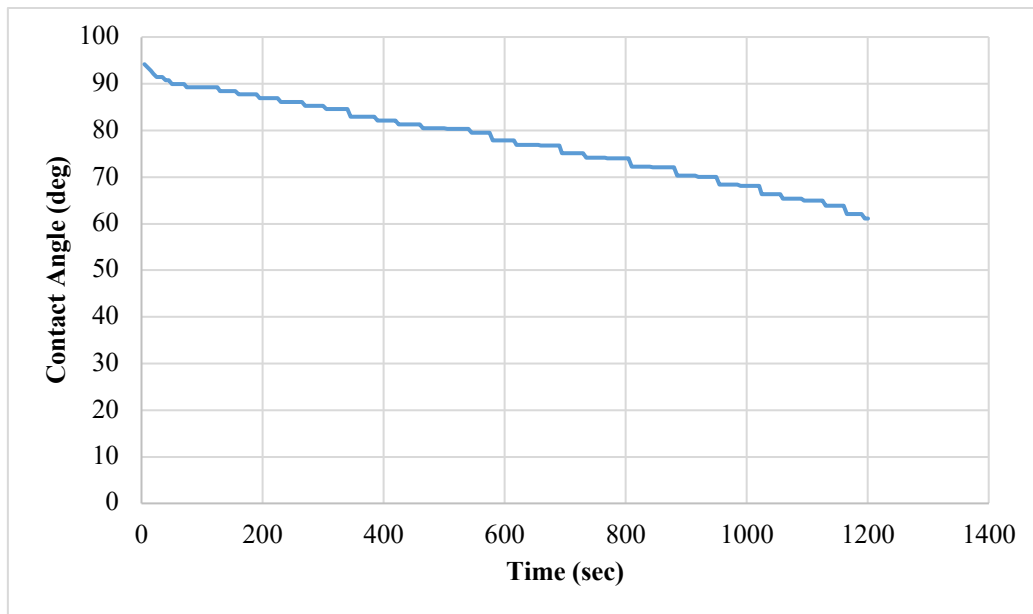
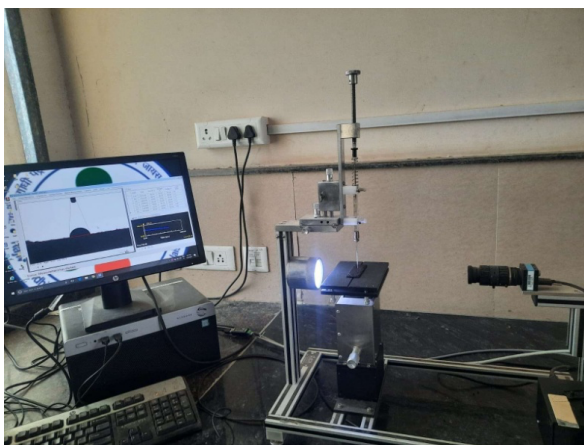
Graph of Contact Angle v/s Time:

Figure 3. Contact angle variation with time of SiO₂ nanofluids for Sandstone rock and Crude Oil sample pellets

Contact angle measurements are critical in assessing the wettability of reservoir rocks, which directly influences the efficiency of waterflooding processes in enhanced oil recovery (EOR). These measurements help in understanding the interactions between the reservoir rock surfaces and the injection fluids.

Contact angles were measured using a high-precision goniometer on polished sandstone samples. The experiment took 20 minutes time with a total of 240 images (1 image per 5 second) at a temperature of 30° C.



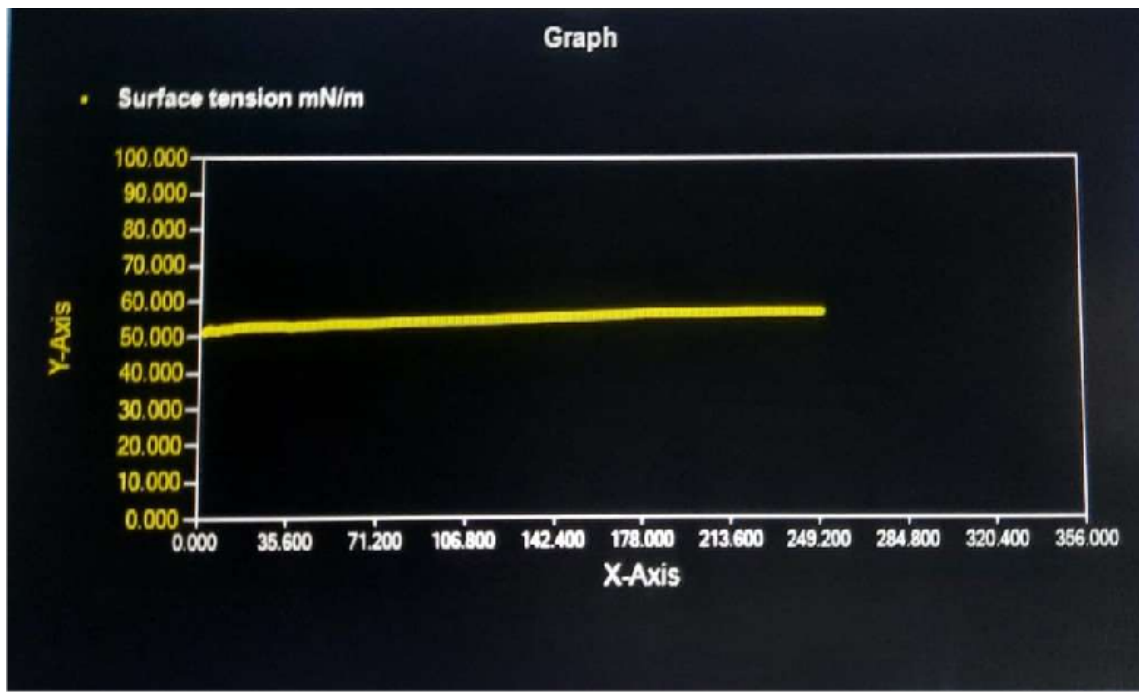
(a)



(b)

Figure 4. (a) Contact-angle Goniometer; (b) Crude oil sandstone pellet

For the sandstone crude oil samples, the average contact angle with SiNF was 77.6505°, suggesting that the rock has neither a strong preference for oil nor for water. This condition can result in mixed wettability scenarios where both waterflooding and oil recovery efficiencies could be optimally balanced or compromised, depending on other reservoir conditions and EOR strategies employed. This intermediate state could offer opportunities for targeted interventions to alter surface properties to maximize recovery or improve the efficiency of waterflooding and other EOR methods.

Surface Tension result:**Figure 5.** Graph of Surface Tension obtained from Goniometer

The result obtained after the experiment is 56.57mN/m. The reduced surface tension of 56.57 mN/m suggests enhanced capability of the surfactant solution to infiltrate pore spaces and displace trapped oil, potentially increasing the efficiency of waterflooding operations. The measured surface tension of 56.57 mN/m for the sample solution underscores its potential to improve oil recovery by enhancing fluid penetration and oil displacement in porous media. These results support the formulation's suitability for SiNF flooding applications in EOR processes.

IFT results:**Table 2.** Results obtained from spinning drop Tensiometer

Results	Value	St. Dev
$\bar{\sigma}$ [mN/m]	4.142457	0.160527
f_{rot} [rpm]	5999.5	0.4
T [°C]	29.2	0
$T_{Heating}$ [°C]	29.4	0
V [μL]	0.234	0.001

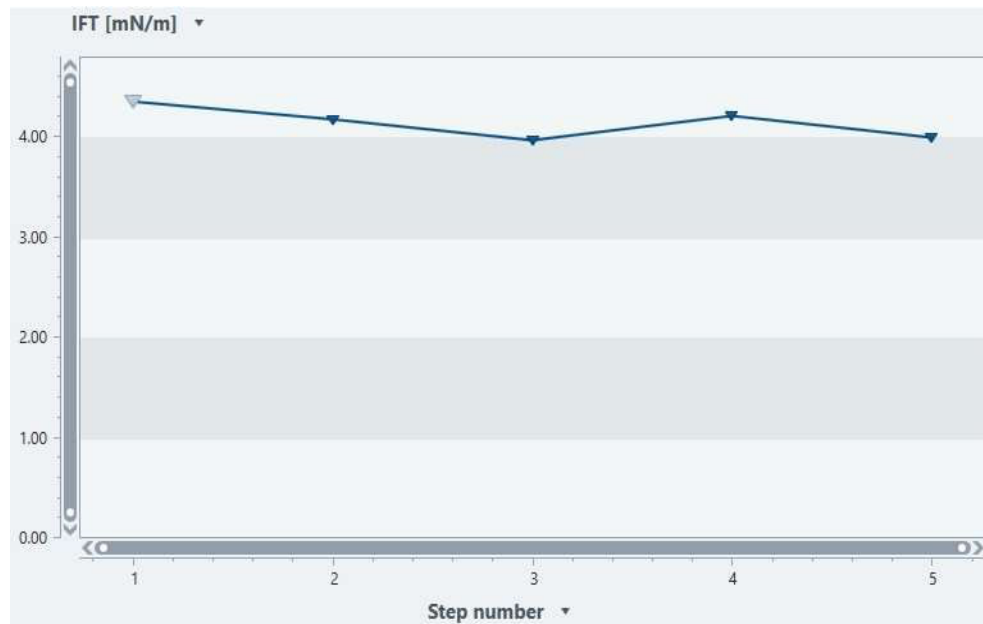


Figure 6. Graph of IFT VS Step number

From the above Graph we can conclude that the IFT value ranges between 3.9 to 4.3. The interfacial tension between the sample solution and the reservoir crude oil was measured to range from 3.9 to 4.3 mN/m, indicating a highly effective reduction in interfacial tension compared to the baseline measurements of crude oil and water, which typically exceed 20 mN/m. The low IFT values achieved suggest significant potential for enhancing capillary number, thereby promoting more efficient displacement of trapped oil. This reduction in IFT enhances the ability of the injection fluids to overcome capillary forces retaining oil within the reservoir rock pores, crucial for improving sweep efficiency.

SEM Analysis:

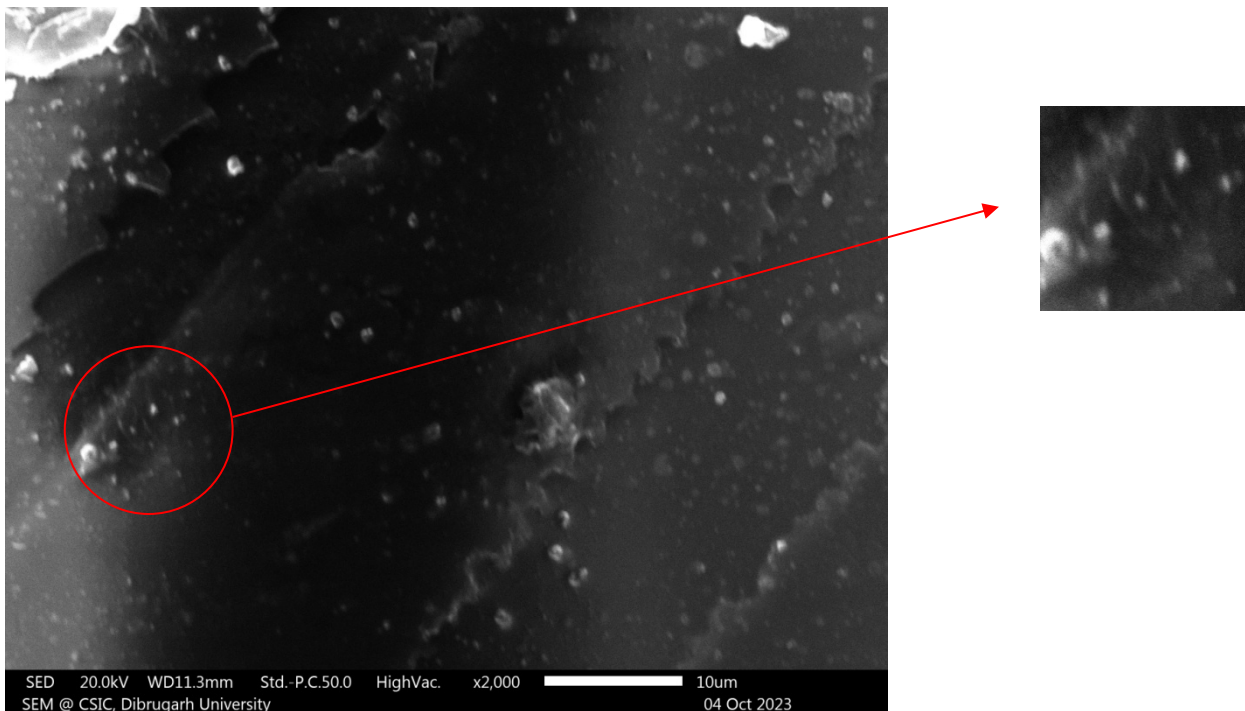


Figure 7. Image Captured from SEM

The above image depicts the size of the nano particle through SEM image. It confirms that the size of the nano particles is under 100 nm. SEM analysis confirmed the suitable morphology and size distribution of silica

nanoparticles, reinforcing their potential as effective agents in EOR applications. Images were taken at various magnifications ($\times 2,000$ and $\times 2,500$) to comprehensively analyse the particle size distribution and morphology. Although the images could have been taken at a larger magnification (like $\times 50,000$ and $\times 55,000$) for clear view which would depict its spherical shape and small size of nanoparticles.

Rheology Analysis:

In recent years, research has focused on understanding how the addition of silica nanoparticles in injection fluids can enhance oil displacement by influencing factors such as viscosity, shear thinning behaviour, and viscoelasticity. However, the detailed mechanisms by which these nanoparticles impact the rheological properties of the fluids remain a subject of ongoing investigation. This study aims to systematically investigate the rheological behaviour of silica nanoparticle dispersions under room temperature. By understanding these interactions, we can better assess the potential of silica nanoparticles to improve EOR efficiency.

Rheological measurements were conducted using a rotational rheometer. The viscosity of the nanoparticle dispersions was measured as a function of shear rate (ranging from 0.1 to 100 s^{-1}) at room temperature. Additionally, oscillatory tests were performed to assess the viscoelastic properties, with frequency sweeps conducted from 0.1 to 100 rad/s at a constant strain of 1% .

The viscosity measurements revealed that the addition of silica nanoparticles significantly alters the rheological behaviour. As shown in Figure 8&9, the viscosity of the nanoparticle dispersions increased with nanoparticle concentration, indicating a thickening effect. This effect was more pronounced at lower shear rates, suggesting the presence of shear-thinning behaviour, which is beneficial for EOR applications where fluid flow through porous media is critical.

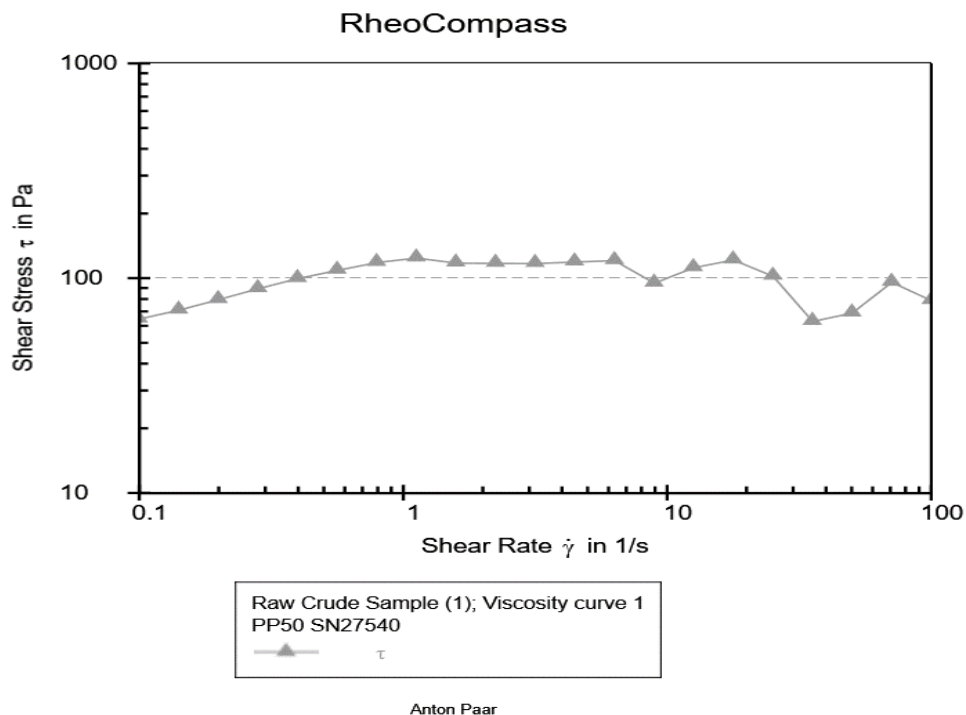


Figure 8. Graph of shear stress v/s shear rate

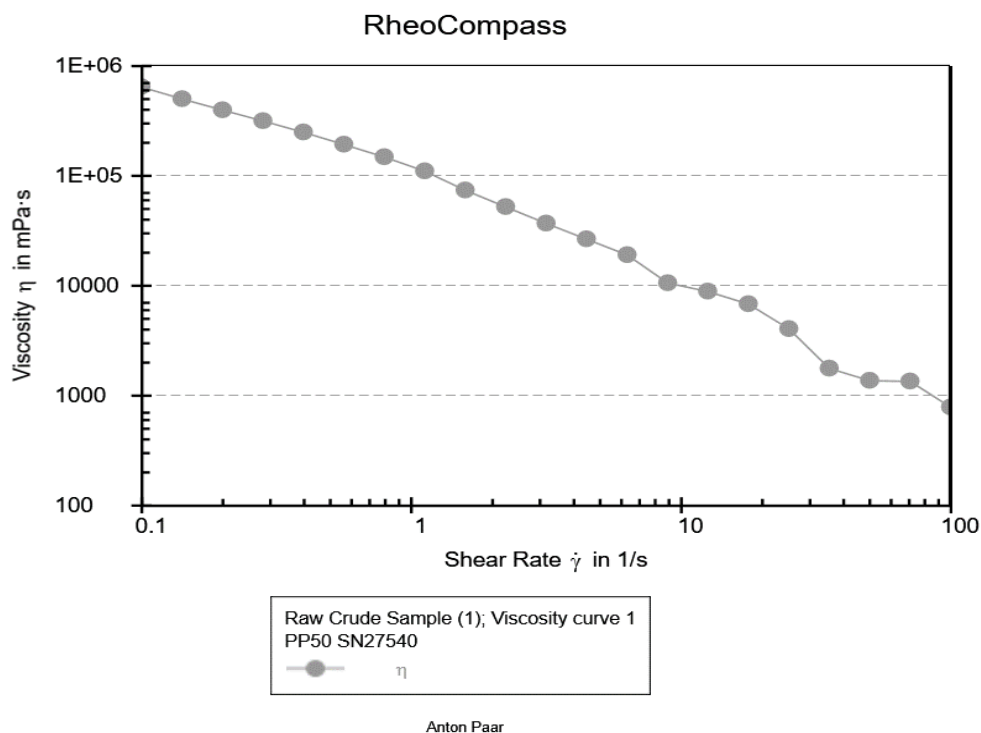


Figure 9. Graph of viscosity v/s shear rate

The rheological analysis of the raw crude sample reveals a clear shear-thinning behavior, as evidenced by the steady decrease in viscosity with increasing shear rate. The shear stress versus shear rate profile indicates a near-constant plateau at lower shear rates, confirming non-Newtonian characteristics typical of heavy crude oils. This behavior highlights the fluid's potential for viscosity modification when integrated with polymer-silica nanofluids. Such nanofluid systems are expected to enhance flow dynamics, reduce resistance under shear, and improve oil displacement efficiency during Enhanced Oil Recovery (EOR) operations. The findings establish a crucial baseline for comparing the rheological improvements achieved through polymer-integrated silica nanofluid formulations, thereby unveiling new possibilities for performance enhancement in EOR applications.

Conclusion

In this study, a novel Silica nanofluid was synthesized with polymer PAM using the SOL-GEL method. After synthesis the fluid was tested for stability through DLS test and zeta potential measurements. Maintaining stability of nanofluids is one of the most important tasks. Once stability is achieved, the fluid was tested for other parameters such as Surface Tension, IFT, Wettability, etc. The results for the mentioned test were very encouraging. It shows the Surface Tension is 56.57mN/m which means a stable compound. From the Graph of IFT, we can conclude that the IFT value ranges between 3.9 – 43. The amounts of data gathered suggest the nanofluid to be highly efficient for changing the properties for EOR practices. Further SEM analysis shows the image of the synthesized silica nanofluid. Although the image doesn't clearly show the nanoparticles but if 50,000 x images were available it could identify the images clearly. The enhanced viscosity and viscoelastic properties observed in the silica nanoparticle dispersions suggest their potential to improve oil recovery by altering the flow characteristics of the injection fluid. The shear-thinning behaviour, in particular, is advantageous for reducing the mobility ratio between the injected fluid and the oil, leading to more efficient displacement and improved recovery. Thus, the results verify the effectiveness of the polymer synthesized silica nanoparticles in EOR field applications.

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