

Experimental Investigation of Silica Nanoparticle Assisted Lignosulfonate Surfactant for Chemical Enhanced Oil Recovery (EOR) Flooding

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Abstract. There has been significant interest in incorporating nanoparticles into surfactants to enhance the performance of chemical-enhanced oil recovery. This interest has arisen due to attempts to mitigate the decline in surfactant efficiency caused by various environmental factors within the reservoir. The primary objective of this study was to investigate how silica nanoparticles (SNP), combined with formulated sodium lignosulfonate (FSLs) surfactants, can improve oil recovery from reservoirs. In this paper, we conducted an experimental study to assess the impact of SNP when mixed with FSLs surfactants. The aim was to evaluate whether SNP alters the characteristics of FSLs surfactants and to determine SNP's potential to enhance oil recovery. We conducted experiments to measure compatibility, interfacial tension, and core flooding. We identified the optimal conditions for an FSLs 1% wt + SNP 0.1% wt solution for chemical flooding test based on the experimental results. The chemical flooding results showed a significantly higher recovery factor (RF) in the presence of SNP, with a recovery of 60% of the initial oil in place (IOIP), compared to only FSLs, which had an RF of 23.53% IOIP. The resulting interfacial tension (IFT) value was 10^{-4} - 10^{-3} mN/m. The solutions showed good stability in a single phase, did not precipitate, and appeared clear. Therefore, using SNP combined with FSLs surfactants demonstrates excellent potential for enhancing chemical-enhanced oil recovery methods.

Keywords: Silica Nanoparticle, Lignosulfonate, Surfactant, Chemical Flooding, EOR

INTRODUCTION

The global economy heavily relies on petroleum, including developing countries

like Indonesia. Indonesia has set a target to produce 1 million barrels per day. One approach to achieve this goal is implementing chemical-enhanced oil

recovery (CEOR) using surfactants. Various surfactants are already utilized in upstream petroleum processes, including their use in chemical EOR (Ahmadi and Sheng, 2016; Yuliansyah *et al.*, 2021). However, finding suitable chemicals in the form of surfactant or polymer for chemical EOR is not trivial. Most available surfactants are petroleum-based, often facing mismatches between water and oil well rock formations. Such surfactants can clump and disrupt production wells (Purwono *et al.*, 2023).

Conventional surfactants used in enhanced oil recovery (EOR) are petroleum-based and known to be environmentally harmful to aquatic organisms (Pinheiro and Faustino, 2017). Surfactants widely available in the market often pose environmental and human health risks. Therefore, petroleum engineers are interested in finding more environmentally friendly surfactants to implement the "green" EOR process (Deljooei *et al.*, 2021).

Several researchers have recently developed surfactants from natural materials to obtain natural surfactants that are both environmentally sustainable and cost-effective (Zhao *et al.*, 2023). Surfactants derived from renewable sources, such as plants or animals, are often referred to as polar lipids, which can be naturally extracted without chemical synthesis or can be chemically produced by combining polar elements from natural sources with non-polar components (Chowdhury *et al.*, 2022). In line with the rise to utilize natural materials for EOR surfactant, there has been growing interest in utilizing oil palm as the material source for chemical surfactant in Indonesia. Despite its source, the proposed surfactant must meet specific criteria, including broad availability, affordability, suitability as industrial waste, high recoverability, and a

non-petroleum origin (Hasokowati *et al.*, 2020; Sheng, 2015).

Chemical-enhanced oil recovery (CEOR) using surfactants or polymers is a well-established technique. There is significant interest to incorporate nanoparticles into surfactants to enhance CEOR performance. Nanoparticles offer a promising technological approach to enhance EOR effectiveness when used as injectable materials in combination with surfactants (Paryoto *et al.*, 2023). Among all nanoparticles, silica nanoparticles were often selected as an agent due to their advantages. Silica nanoparticles are relatively inexpensive and readily available resources (Zhou *et al.*, 2019). They are fine amorphous white powders with the highest production yields in large-scale industries worldwide. Because nano silica particles are cost-effective and readily surface-modified, they have become widely used materials in EOR (Tian *et al.*, 2020).

Silica nanoparticles, known for their high efficiency and outstanding characteristics of small size, large specific surface area, and highly active surfaces, can penetrate nanoscale pores within rock formations, effectively recovering residual oil (Ali *et al.*, 2021). The combination of silica nanoparticles and surfactants notably impacts interfacial tension.

One primary mechanism for combining surfactants and nanoparticles is that they significantly reduce the interfacial tension (IFT) between oil and water (Ahmadi and Sheng, 2016; Dampang *et al.*, 2024). Increasing the C-H bond density at the oil-wet interface reduces IFT due to the adsorption of surfactant molecules on the surface of silica nanoparticles (Tian *et al.*, 2020). Further, adding surfactants enhances nanoparticle solution's stability, which is a key

reason for using surfactants and nanoparticles (Almahfood and Bai, 2018).

SNP can improve the ability of surfactants to lower the IFT of oil-water. Its high surface area to volume ratio allows it to adhere to the interface between oil and water. Therefore, the contact area between oil and water increases, leading to a higher reduction in IFT, increasing the release of oil from reservoir rocks, and facilitating easier oil mobilization (Dauyltayeva *et al.*, 2023). The presence of SNPs can effectively reduce the adsorption of surfactants in rocks by occupying the surface area of the sand, reducing the contact between the surfactant and the sand surface, and facilitating the release of absorbed surfactant molecules through friction and collision with the sand surface (Wu *et al.*, 2017).

The surfactant adsorbed on SNP decreased, possibly due to the strong repulsion between the negatively charged anionic surfactant and negatively charged SNP at high pH (Rattanaudom *et al.*, 2021). The reduction in surfactant solution adsorbed onto the rock surface is probably also due to SNP, which is a sacrificial agent that was first adsorbed onto the rock surface. The smaller size and larger surface area of SNPs allow them to interact effectively with rock surfaces, reducing the amount of adsorbed surfactant and increasing oil recovery through EOR (Sze Lim *et al.*, 2023). Although several studies in the present literature reported the use of nano particles and surfactants for CEOR, to our knowledge, there is little information about the role of nano particle to enhance lignosulfonate surfactant to improve CEOR.

The work aimed was to investigate the influence of SNP addition to lignosulfonate surfactant prepared from oil palm empty fruit bunch (OPEFB). The first part of this work includes preparing surfactant from OPEFB.

Subsequently, the resulting SLS solution was mixed with cosurfactants to obtain Formulated-SLS, denoted as FSLs. The influence of SNP synthesized from sodium silicate was investigated through compatibility, IFT, and core flooding tests.

MATERIALS AND METHODS

Material Preparation

The materials used in this experiment were as follows: NaOH 100% (Merck), H₂SO₄ 96% (Merck), NaHSO₃ 99% (Merck), and Octanol 99% (Merck). PFAD soap, methanol, brine, and crude oil were obtained from Coal, Gas, and Petroleum Technology Lab UGM. The silica nanoparticles (SNP) sample was obtained from the Nanotechnology Laboratory at Universitas Diponegoro, Semarang, Indonesia. The properties of silica nanoparticles are shown in Table 1.

Table 1. Properties of Silica Nano Particle

Properties	SNP
Diameter (TEM)	6-10 nm
Diameter aggregate (PSA)	169 nm
Concentration (m/v)	12.7 %
pH	8.69
Moisture content	91%
Ash content	6.35%
Zeta potential	24.8 MV
Contaminant	NaCl (unknown)

SLS surfactant was prepared from oil palm empty fruit bunches. Initially, 90 grams of dried and finely ground empty fruit (10 mesh) were used. An additional 10 grams of NaOH was added to a 1-liter solution with a 1:1 ratio (500 ml of 96% ethanol and 500 ml of distilled water). Fine powder of OPEFB was mixed with NaOH, placed in an autoclave, stirred, and heated to 170°C for 5 hours. Subsequently, the samples were subjected to a vacuum pump to obtain the filtrate or black

liquor (BL). The BL solution was diluted four times, and H₂SO₄ (10%) solution was added to decrease the pH to 2. The resulting BL sample was held for at least 8 hours or overnight. After allowing the BL to settle, the BL solution was filtered using a vacuum pump and filter paper to extract the lignin paste. The lignin paste was subjected to an oven with a vacuum system at 70°C. The resulting lignin paste was mashed to obtain the desired lignin powder.

Sulfonation was conducted by mixing 10 grams of lignin powder with 15.6 grams of NaHSO₃ and 500 ml of distilled water in a reflux system at 97°C, stirring rate at 300 rpm for 4 hours. Subsequently, the solution was distilled to reach 100°C, forming a solid product. Next, about 300 ml of methanol was added to the solid product and stirred for 30 minutes. The mixture was filtered using a vacuum pump to collect the paste and transferred to another distillation at 70°C. The solid paste was then placed in an oven at 70°C and refined to transform it into SLS powder.

Table 2. Specification of Brine

Parameter Analysis	Brine
TDS (ppm)	13,006
Salinity (ppm)	8,110
pH	8.452
Na ⁺ (ppm)	3,162
K ⁺ (ppm)	24.14
Ca ²⁺ (ppm)	83.75
Mg ²⁺ (ppm)	165
Cl ⁻ (ppm)	4,931
CO ₃ ²⁻ (ppm)	234
SO ₄ ²⁻ (ppm)	2.63
Total Organic Carbo (TOC) (ppm)	674.69

The formulation of SLS surfactant with octanol and PFAD soap followed the same procedure as described by Azis *et al.* (Azis *et*

al., 2021). The SNP solution was then diluted with a specific amount of brine to form the silica nano solution. The resulting diluted silica nano solution was sonicated for 10-20 minutes. Subsequently, the SLS surfactant solution was blended with the silica nano solution and the brine to reach a solution volume of 100 ml.

Here, FLS solution with 1% wt concentration was mixed with SNP concentrations of 0.05, 0.1, and 0.2% wt, respectively. The resulting mixture was then stirred again for 5-20 minutes, and this solution was referred to as the nanofluid solution. The brine specification is available in Table 2.

The oil and brine used were obtained from an oil field in Sumatra. These solutions will be compared with FLS 1% and FLS 1%+SNP 0.1%. Table 3 provides detailed information about the materials' properties.

Table 3. Properties of Materials

Description	FLS 1%	FLS %+ SNP 0.1%
Oil density, gr/cm ³	0.80	0.80
Brine density, gr/cm ³	0.985	0.985
Surfactant IFT, mN/m	2.69x10 ⁻³	2.35x10 ⁻⁴
Gas permeability, md	200-230	200-230
Gas porosity, %	18-21	18-21
Brine permeability, md	11.44	11.90
Brine porosity, %	19.21	18.374
PV brine, cm ³	3.89	3.68
PV gas, cm ³	3.65	3.54

Methods

Compatibility Test

A compatibility test was conducted to evaluate to what extent the chemical solution interacts with the reservoir fluid and rock formation without causing adverse reactions. Compatibility tests were performed by mixing a chemical solution with brine. In the ideal

case, the solution would dissolve completely without any deposits or lumps that could hinder the injection process into the rock.

The compatibility test method was conducted: FLS 1 wt % with various SNP contents (0%; 0.05%; 0.1%; 0.2% wt) solutions of 15 ml each were put into test tubes and then shaken to obtain a homogeneous solution. Then, each tube was tightly closed and placed in a blue MOV12A gravity oven at 60–70°C. Next, the solution was left for 3 days and monitored regularly.

IFT Measurements

The IFT test was conducted using a spinning drop interfacial tensiometer of TX-500D, manufactured by CNG USA Co. For this test, solutions of FLS 1% wt, mixed with various SNP content (0; 0.05%; 0.1%; 0.2% wt), were prepared for this test. These solutions were subjected to an interfacial tension (IFT) test, each trial lasting for 30 minutes and repeated twice.

Core Flooding Test

Core flooding experiments were conducted on two core samples of the same rock type, Berea stone, to evaluate nanofluid solutions' capability to enhance oil recovery accurately. Two core plugs were prepared for this purpose, and their properties are listed in Table 4.

Table 4. Properties Core Plugs

Description	core1	core2
Length, cm	4.13	4.0
Diameter, cm	2.50	2.50
Area, cm ²	4.91	4.91
V bulk, cm ³	20.28	19.66
Viscosity liquid, cp	1	1
Slope Q/Δ P, cm ³ s ⁻¹ /atm	0.014	0.015
Permeability, mD	11.44	11.90

The core flooding setup is shown in Fig. 1. A metallic core holder was used to keep the core with the aid of N₂ to pressurize the core holder. The experimental conditions included a confining pressure of 200 Psig, a temperature of 60°C, and a constant injection rate of 0.3 cm³/min. Three separate metallic tanks store oil, brine, and surfactants. A core plug from Berea sandstone was also thoroughly saturated with brine until no more air bubbles were visible. Subsequently, the core was saturated with oil for at least 24 hours.

The core flood test was initiated with a water flood test by injecting the core with brine, and the volume of displaced oil was recorded from the core. Subsequently, chemical flood injection was conducted, and similarly, the amount of oil recovered was monitored. In this work, core flood tests were conducted with and without SNP. In the first set, FLS solution of 1 wt.% was used to improve the oil recovery. Subsequently, a mixture of FLS solution of 1 wt.% and SNP concentration of 0.1 wt.% was used to improve the oil recovery.

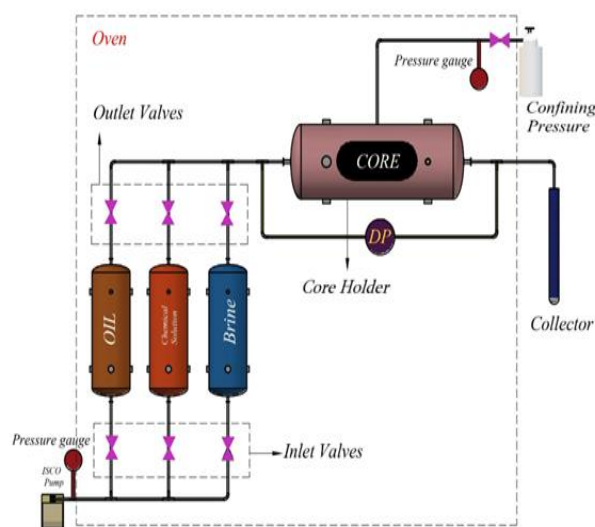


Fig. 1: Core Flooding Setup Schematic

RESULTS AND DISCUSSION

Compatibility Test

Good solubility of the surfactant and SNP in the formation of water/salt is very important because it can produce a mixture of surfactant and SNP with even distribution so that surfactant performance remains consistent. Conversely, if the solubility is not optimal, the mixture of surfactant and SNP in the brine becomes uneven and can negatively impact its performance when used in reservoirs. A good solution will remain stable even when it fluctuates at 60°C. Solutions that pass the compatibility test can withstand heat without damage.

Figure 2 shows the compatibility test result. All solutions showed good homogeneity, forming a single phase, appearing clear, and not precipitating. Thus, it can be concluded that all solutions were compatible with the brine.



Fig. 2: Compatibility of FSLs 1% and FSLs 1% with SNP (%)

IFT Measurements

The plot illustrating the interfacial tension of the resulting FSLs solution with various concentrations of SNP is depicted in Fig. 3. All measurements were obtained after 30 minutes at least 2 times and averaged. The

measurements were conducted to confirm the replicability of the results. As time progressed, the IFT value decreased until it reached a certain equilibrium point, consistent with findings from previous studies (Daghbandan *et al.*, 2022; Kuang *et al.*, 2018; Zhao *et al.*, 2021).

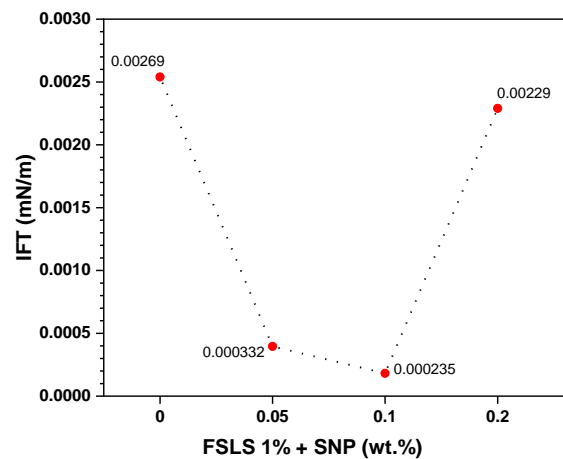


Fig. 3: IFT vs Concentration of Nano Fluid

To assess the impact of SNP, we also measured the IFT between FSLs and crude oil both in the absence and presence of SNP. IFT measurement of FSLs 1% wt without SNP reveals an initial IFT value of 2.69×10^{-3} mN/m. Following the addition of SNP in the system, an SNP concentration of 0.05 wt% gave a further decrease in IFT to 3.32×10^{-4} mN/m. Increasing the SNP to 0.1% wt gave an IFT value of 2.35×10^{-4} mN/m. However, SNP concentration of 0.2% wt gave a little increase of IFT to 2.29×10^{-3} mN/m. In the research conducted by Tian *et al.* (2020) by adding SNP to a mixture of surfactants petroleum sulfonate (PS) and silane (KH570), the resulting IFT was 5.42×10^{-3} mN/m, which is still higher than the IFT presented in Fig. 3.

The addition of silica nanoparticles can affect the arrangement of surfactant monomers at the oil-water interface, impacting the equilibrium of the interface tension. The interfacial tension can be

affected by the number of surface-active molecules at the oil-water interface, and increasing IFT values indicate a reduction in the number of active molecules at the oil-water interface (Jiang *et al.*, 2016).

Furthermore, silica nanoparticles have a large surface area. When added to the surfactant, silica nanoparticles increase the interfacial area between the oil and water phases within the reservoir, allowing the surfactant to work more effectively in lowering IFT. Generally, IFT decreases as nanofluids are adsorbed at the interface between the fluid phases (Ali *et al.*, 2019). Our results also indicated that the most suitable SNP concentration is 0.1 wt.%. It gave the lowest IFT and was a stable solution.

The increase in interfacial tension after the addition of 0.2 wt.% SNP was also potentially attributed to forming a Surfactant Coated Particle (SCP) (Jiang *et al.*, 2016). SCP arises due to silica nanoparticles possessing surfaces with Van der Waals attraction. The Van der Waals attraction on the surfaces of silica nanoparticles leads to the adherence of surfactant molecules, thereby creating a coating layer around the nanoparticles.

Core Flooding Test

Figures 4 and 5 showed the results of the core flood test in the absence (black line) and in the presence (red line) of 0.1 wt % SNP. Without SNP (black line), the results of the water flood test gave a recovery factor of 5.88% IOIP after about 5.78 PV injection. Further injection (chemical injection) was conducted using FLS solution for about 15.02 PV. After chemical injection, it gave an incremental recovery of 18.75% residual oil in place (ROIP) for a total RF of 23.53%.

The core flooding results using FLS 1%+SNP 0.1% at the water flood injection of around 7.33 PV provide a recovery factor of

15% IOIP. Subsequent injection of 17.11 PV resulted in a dramatic increase of oil recovery to 52.94% ROIP when flooded with the FLS 1%+SNP 0.1% solution, resulting in a total RF of 60%. Overall, it is clear that the FLS 1%+SNP 0.1% solution (45% IOIP) demonstrates superior recovery performance compared to the FLS 1% solution alone (17.65% IOIP). The results here can be compared to those of Pillai *et al.* (2019) using Jatropha Surfactant 8000 ppm + nanoparticles 1% + brine 2%, which reported an additional recovery of 24.6%. Further, Paryoto *et al.* also reported that by using commercial surfactant + Fe₃O₄ nanoparticles obtained a recovery factor of 5.09% OOIP or 17.80% ROIP (Paryoto *et al.*, 2023).

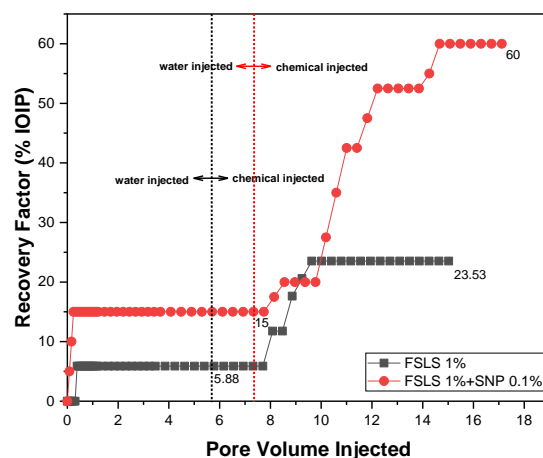


Fig. 4: Recovery Factor vs PV Injected

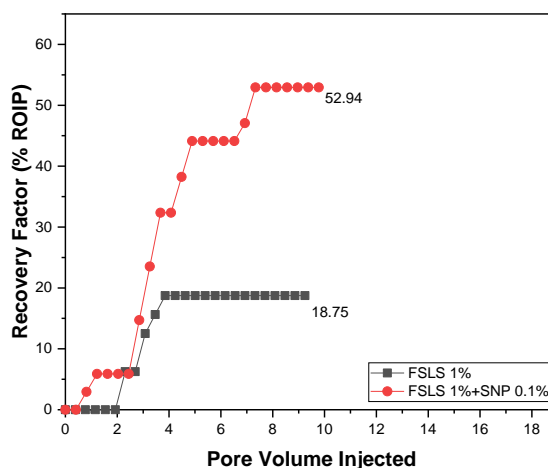


Fig. 5: Incremental Oil Recovery (% ROIP)

The presence of SNP in the nanofluid solution causes a decrease in oil/water interfacial tension (as shown in Fig. 3). The SNP used in the nanofluid solution is a hydrophilic silica nanoparticle, which exhibits a good affinity to water. The FSLs, an anionic surfactant, competes with the negatively charged SNP (pH 8.69) for adsorption on the rock surface, thereby reducing the surfactant adsorbed on the rock. The decrease in surfactant adsorbed on SNP may be due to the strong repulsion between the negatively charged anionic surfactant and negatively charged SNP at high pH levels (Rattanaudom *et al.*, 2021).

Nanoparticles play a crucial role as sacrificial agents, serving as a preferential adsorption agent to interact with rock surfaces compared to surfactants. The advantage of nanoparticles lies in their smaller size and larger surface area, which can reduce the amount of surfactant adsorbed on the rock surface because the rock surface is already occupied by nanoparticles (Sze Lim *et al.*, 2023).

CONCLUSIONS

The influence of SNP on the sodium lignosulfonate surfactant prepared from oil palm empty fruit bunches (OPEFB) has been investigated in the present work. The results from the compatibility test showed that SNP concentrations from 0 to 0.2 wt.% gave good compatibility with the surfactant solution. The IFT test showed that the addition of SNP up to 0.1 wt.% gave ultralow IFT of 2.35×10^{-4} mN/m. Furthermore, the core flooding test showed that the combination of FSLs 1wt%+0.1 wt.% SNP dramatically increased from 15% to 60% (IOIP).

From this work, it appears that the role of SNP was two folds: first it helped to reduce

IFT of surfactant and secondly it acted as a sacrificial agent serving as a preferential adsorption agent to interact with rock surfaces compared to surfactants. Therefore, using SNP combined with FSLs surfactants demonstrates excellent potential for enhancing chemical-enhanced oil recovery methods.

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