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## Modifying Investigation of Nano-silica Additive for Orientating Applications on Enhanced Oil Recovery

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### Abstract

Nano additives can be attributed to the nanomaterials' unique properties including high surface to volume ratio, wettability control, interfacial tension reduction, viscosity behavior improvement, and oil sweeping profile adjustment which result in reducing water cut, increasing sweep efficiency and improving oil recovery. This paper aims to modify silica nanoparticles and to investigate polymer-coated silica nanoparticles (PSiNPs) for enhanced oil recovery. A copolymer containing nano-SiO<sub>2</sub> was synthesized by free radical polymerization using epoxy resin E44 and nano-SiO<sub>2</sub> functional monomer as raw materials. The nano-SiO<sub>2</sub> copolymer containing PSiNPs was characterized by scanning electron microscope (SEM), transmission electron microscope (TEM), Fourier transform infrared (FT-IR) spectroscopy, thermogravimetric analysis (TGA) and differential thermal analysis (DTA). The solution properties, such as rheological property, viscoelasticity, mobility control ability, hydrophilic/hydrophobic ability and oil displacement efficiency of the copolymer, were investigated using a dynamic contact angle (DCA) analysis. Crude oil from the Diamond field (Cuu Long basin) was used for flooding tests. The concentrated solutions of PSiNPs were diluted upto 0.5 wt. % in synthetic seawater. Experimental results show that PSiNPs can improve water flood oil recovery efficiency. In tertiary recovery mode, the incremental oil recoveries varied from 2.1 % to 6.24 % of original oil in place (OOIP) achieved by reference seawater flood. These results reveal a potential way to use silica-based nanofluid for enhanced oil recovery purposes.

**Keywords:** Nano additives; enhanced oil recovery; oil sweeping profile.

### 1. Introduction

#### Nomenclature

DCA	dynamic contact angle
DTA	differential thermal analysis
E <sub>D</sub>	displacement efficiency
FT-IR	Fourier transform infrared
NF	nanofluid
PSiNPs	polymer-coated silica nanoparticles
SEM	scanning electron microscope
SSW	synthetic seawater
SWF	seawater flooding
TEM	transmission electron microscope
TGA	thermogravimetric analysis

In Vietnam, the production rates of existing oil fields are quickly declining and the frequency of new explorations has become scarce. Therefore, the significance of enhanced oil recovery (EOR) techniques is highly understood by oil companies. In this regard, nanoparticles (NPs) have been researched as additives to improve

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water flooding oil recovery (Alberto et al., 2019; Alberto and Ole, 2021; Anirbid et al., 2022; Bin et al., 2021; Cheraghian et al., 2016; Goshtasp et al., 2020; Hoang, 2018; Le, 2012; Marwan and Nageh, 2019; Nguyen, 2013; Xiaofei et al., 2017; Zhongbin et al., 2013). The small size of the nanoparticles and large surface area-to-volume ratio increase the particle mobility and surface activity, particularly at elevated temperature, which contribute to alter the fluid–rock interface properties. This makes the NPs appropriate and potential for enhanced oil production. Thus far, silica NPs are the most studied nano-materials for EOR applications (Alberto et al., 2019), as they are the most abundant compounds on Earth and are of natural occurrence in sandstone formations. More importantly, silica NPs can be easily produced and surface modified to target specific applications. Previous studies have reported promising oil recovery results due to silica NPs injection (Hoang, 2018; Nguyen, 2013; Le, 2012; Alberto et al., 2019; Alberto and Ole, 2021; Zhongbin et al., 2013) under a variety of laboratory conditions. The flooding experiments varied from the use of silica NPs with different size, composition, surface modification, etc., and the NPs suspended in aqueous solutions with diverse ionic strength or in non-aqueous solutions. The concentration of silica NPs in the suspension was also varied. The results proved that nano-SiO<sub>2</sub> additive exerts a good effect on EOR because this additive takes the role in improving rheological properties of solutions at elevated temperature and decreasing oil – water interfacial tension (IFT) under emulsion or bulk foam environments. The dispersion of nanosilica particles is formed by cut force and IFT reduction and the stability of emulsions/foam in porous media is balanced by adsorption of nanoparticles on contact surfaces. Despite the potential of the nanoparticles, there is a lack of experimental repeatability. The application of NPs, at laboratory scale, have shown an incremental recovery of oil ranging from 5 to 15 % of the OOIP, the highest reach is 32 % of the OOIP. The most frequent range has been 5 % of the OOIP (Alberto and Ole, 2021). Obviously, oil recovery by NPs is a complex phenomenon, partly because the reservoirs are unique and have different characteristics. On the other hand, the variability of experimental approaches in assessing the efficiency of nanoparticle recovery ends up with variable results and variable interpretation of the causes of oil displacement. Almost studies applied nano technology on EOR were carried out along with using polymers or surfactants (Cheraghian et al., 2016; Goshtasp et al., 2020; Alberto et al., 2019; Bin et al., 2021) to produce highly homogeneous system and increasingly combined effect. The EOR application of inorganic NPs is also hampered by poor dispersibility or stability in the injection fluid. Nanoparticles have an immense surface free energy triggered by their ultra-small size and large surface area, which increases the tendency of the NPs to aggregate/agglomerate in solution in an attempt to gain the low energy state (Alberto et al., 2019). To overcome the stability issues, the surface of the nanoparticles can be modified by attaching or coating macro-molecules such as polymers on NPs surface to decrease the surface free energy and to increase the dispersibility. On EOR applications, nanoparticles have impressive advantage to prevent decomposition of polymer under porous media. Nanoparticles with polymer chains coating on the particle surface are often referred to as polymer-coated NPs or , and they can offer significant advantages such as improved solubility and stability, greater stabilization of emulsions, and easier transport through porous media over bare nanoparticles. There are still few experimental studies concerning EOR applications of polymer-coated silica nanoparticles (PSiNPs) and the oil recovery mechanisms of the NPs need to be well understood. To some extent, this paper can meet these purposes.

## 2. Experimental materials and methods

### 2.1. Equipment, materials and chemicals

Nano silica from SigmaAldrich containing 90 - 95 % of particles with size distribution in the range of 20 - 30 nm was used in the work. These silica nanoparticles have microporous area of 27 - 28 m<sup>2</sup>/g, and BET specific surface area of 118 - 120 m<sup>2</sup>/g. Epoxy resin E-44 (CAS 61788-97-4) with molecular weight of 400 - 450 g/mol was used for modifying nano silica. The epoxy resin E-44 has a purity of 90 %, melting point of 12 - 20 °C, and viscosity of 6 - 10 mPa.s at 25 °C. Hydrophilic-hydrophobic KYPAM polymer with molecular weight ranging from 13 x 10<sup>6</sup> to 21 x 10<sup>6</sup> g/mol was used as polymer for flooding. Other additional chemicals such as toluene, imidazole and acetone and equipment were used as needed. All chemicals were used as received without any further purification. Water was deionized by passing through an ion exchange column and doubly distilled.

### 2.2. Experimental

Firstly, 20 g of nanosilica were dispersed in 300 mL of toluene containing 3 g of imidazole as catalyst for reaction. Slowly and gradually dropping 10 g of epoxy resin in round-bottom flask reaction system with pipe cooler. The reaction mixture was stirred at about 120 to 150 rpm and at 110 °C for 2 hours. After reaction completely removal of excess imidazole. Then, the amount of precipitate was separated by centrifugal method. The precipitation product was dried at 110 °C for 10 hours with temperature increment of 5 °C/minute and finally pulverized.

Modified nanosilica (nano-SiO<sub>2</sub>) particles referred as polymer-coated silica nanoparticles (PSiNPs) were

formed by reacting between -OH groups of nanosilica and epoxy groups of epoxy resin. Based on this reaction, the silica surface was organized and the originally hydrophilic silica surface became hydrophobic. There are two forms of modified nanosilica produced by this process (form 1 and form 2) as shown in figure 1:

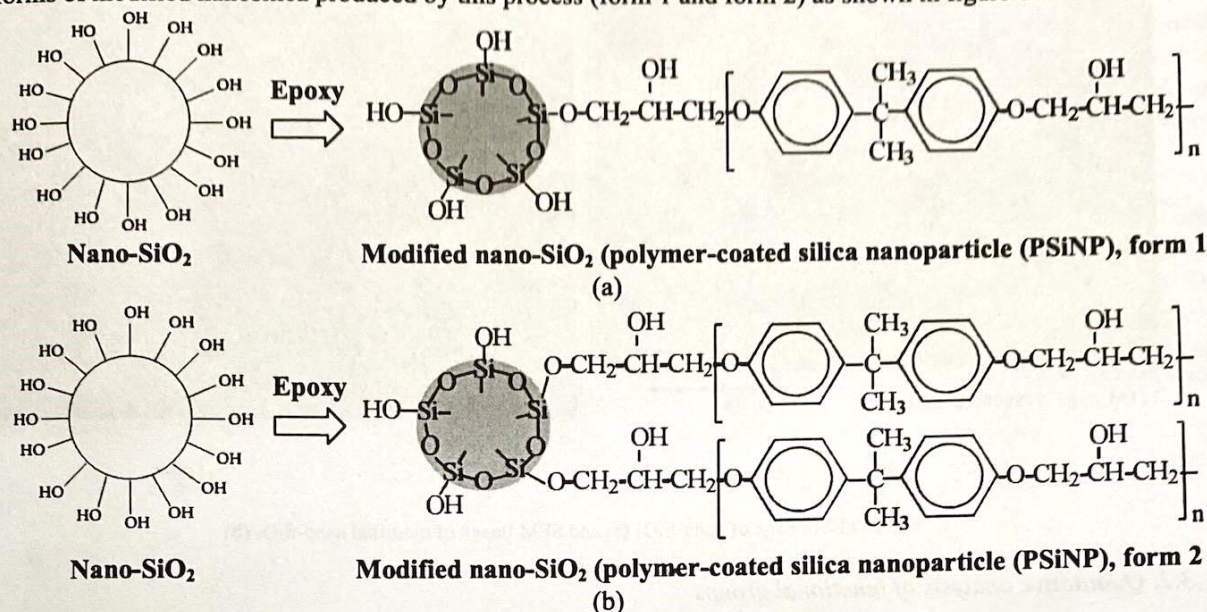


Fig. 1. Structures of two forms of modified nanosilica: (a) form 1, (b) form 2

### 2.3. Characterization methods

The nano-SiO<sub>2</sub> and modified nano-SiO<sub>2</sub> copolymer were characterized by scanning electron microscope (SEM) or transmission electron microscope (TEM) for defining surface state and microstructures. The methods of Fourier transform infrared (FT-IR) spectroscopy, thermogravimetric analysis (TGA) and differential thermal analysis (DTA) were used for qualitative analysis of nano-SiO<sub>2</sub> before and after modification. The hydrophobicity and other solution properties of the nano-SiO<sub>2</sub> and modified nano-SiO<sub>2</sub> copolymer were also investigated using the Wilhelmy dynamic contact angle (DCA) analysis.

## 3. Results and discussion

### 3.1. Surface state and particle size measurement

TEM and SEM images of samples (figure 2) depict the surface state, particle size and typical chains of polymer nanoparticles due to significant interaction among nanosized particles. The TEM image of nano-SiO<sub>2</sub> particles before modification (figure 2a) shows that silica nanoparticles are relatively spherical shape and uniform size. Due to surface structure characterization, silica nanoparticles are trendly aggregated to form particle assemble. The SEM image of modified nano-SiO<sub>2</sub> particles (figure 2b) indicates that the size of nano-SiO<sub>2</sub> particles after modification is only marginally changed (about 40 ÷ 60 nm). On the surface, modified nano particles are also trendly aggregated to form larger particle assemble at the edge of the system. The reason is that the surface of nanoparticles was organized by bulky epoxy molecules and combined with each other.

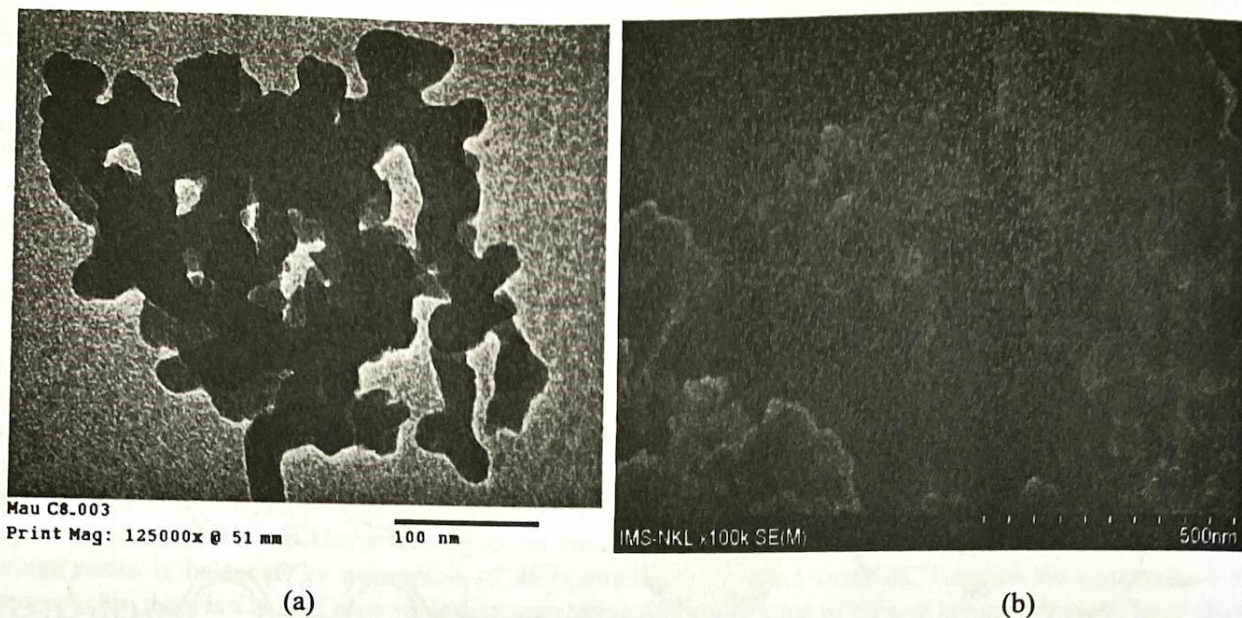


Fig. 2. TEM image of nano-SiO<sub>2</sub> (a) and SEM image of modified nano-SiO<sub>2</sub> (b)

### 3.2. Qualitative analysis of functional groups

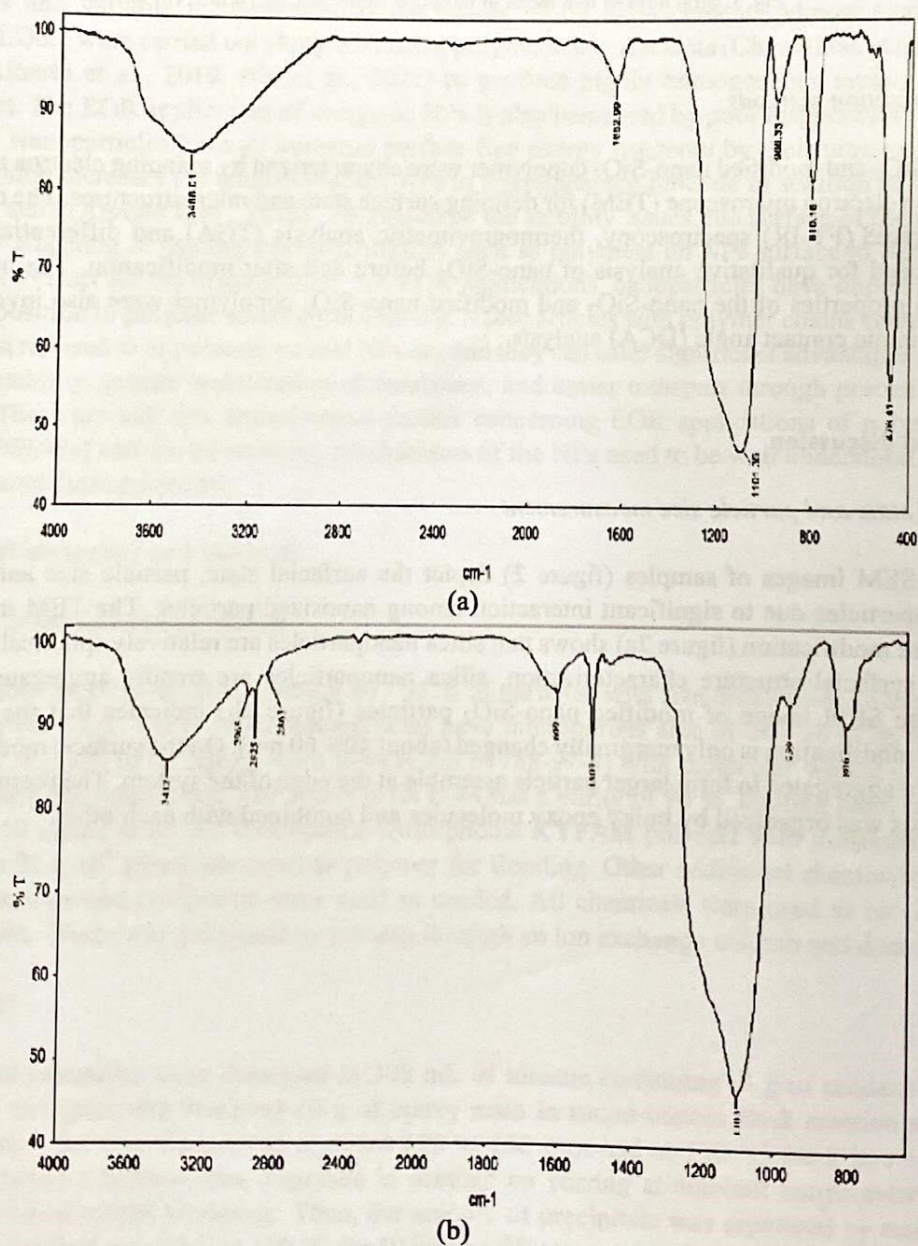


Fig. 3. FT-IR spectra of nano-SiO<sub>2</sub> (a) and modified nano-SiO<sub>2</sub> (b)

FT-IR spectra of nano-SiO<sub>2</sub> and modified nano-SiO<sub>2</sub> are shown in figure 3 respectively. In FT-IR spectrum of nano-SiO<sub>2</sub> (figure 3a), there is present of the peak at wavenumber 3468 cm<sup>-1</sup> characterized for valence oscillation of free -OH groups. These free -OH groups serve for physical adsorption of water on the silica surface. The peak at wavenumber of 1101 cm<sup>-1</sup> is for asymmetrical oscillation of Si-O-Si groups. The peak at wavenumber of 966 cm<sup>-1</sup> is for oscillation of Si-O in silanol groups. The peak at wavenumber of 810 cm<sup>-1</sup> is for symmetrical valance oscillation of Si-O-Si in silica network. And the peak at wavenumber of 1652 cm<sup>-1</sup> is characterized for oscillation of H<sub>2</sub>O adsorbed on silica surface (Nguyen et al., 2016).

After modified by epoxy resin (figure 3b), there are the adsorption peaks at wavenumbers of 2863 cm<sup>-1</sup> and 2965 cm<sup>-1</sup> characterized for valence oscillation of -CH<sub>2</sub>- groups in carbon chain of epoxy. The peak of -OH groups also appears at wavenumber of 3412 cm<sup>-1</sup> demonstrating that -OH groups are still kept on the silica surface. The intensity of this peak in spectrum of the modified nano-SiO<sub>2</sub>, however, is smaller than that of nano-SiO<sub>2</sub> without modification. The adsorption peak at wavenumber of 1103 cm<sup>-1</sup> of the modified nano-SiO<sub>2</sub> also is for asymmetrical oscillation of Si-O-Si groups. And the oscillation of aromatic ring appears at wavenumbers of 1509 cm<sup>-1</sup> and 806 cm<sup>-1</sup> relating to double bond C=C of the aromatic core.

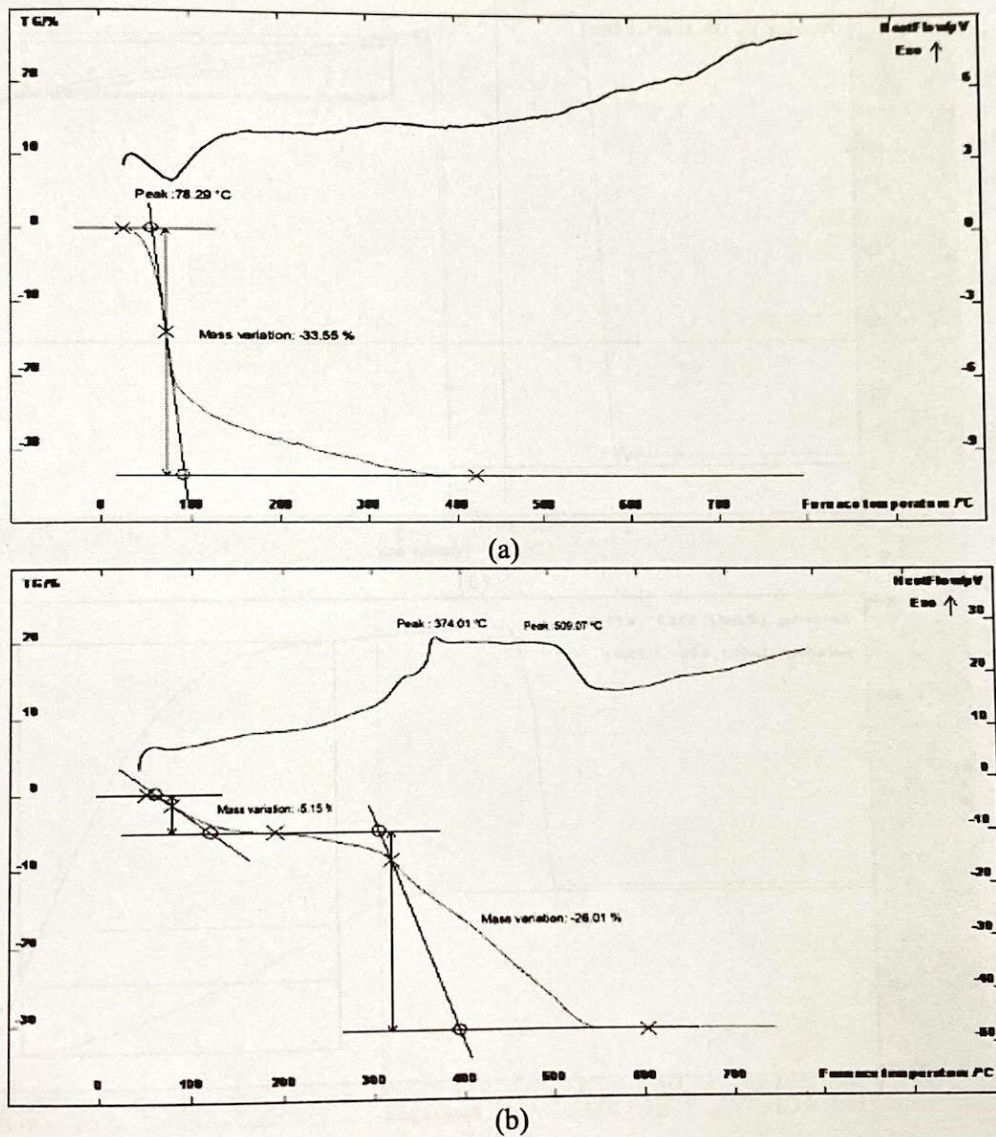


Fig. 4. TGA/DTA graphs of nano-SiO<sub>2</sub> (a) and modified nano-SiO<sub>2</sub> (b)

Qualitative analysis of functional groups was also carried out by thermogravimetric analysis (TGA) and differential thermal analysis (DTA). TGA/DTA signal graphs of nano-SiO<sub>2</sub> and modified nano-SiO<sub>2</sub> are shown in figure 4 respectively. TGA/DTA curves of nano-SiO<sub>2</sub> (figure 4a) show that estimated weight loss of about 33.55 % occurs at 70 - 150 °C which probably is due to the loss of H<sub>2</sub>O molecule. On DTA curve, there is only a peak at 78 °C which refers to endothermic effect for thermal degradation. At higher temperature of 78 °C, the mass of silica is trivially changed. The reason for this peak at 78 °C is that silica nanoparticles can be able to adsorb largely water on their surface. In the temperature range from 70 °C to 150 °C, this amount of water was evaporated and separated out of material surface. At elevated temperature, the weight loss still continued but slowly happened. This process is actually dehydration of -OH groups on silica surface. The dehydration reaction is as follows: ≡ Si-OH + HO-Si ≡ → ≡ Si-O-Si ≡ + H<sub>2</sub>O.

For TGA/DTA graph of modified nano-SiO<sub>2</sub> (figure 4b), TGA/DTA curves also show that the weight loss happened gradually but suddenly. The TGA curve indicates that modified nano-SiO<sub>2</sub> is thermally decomposed in two steps. The first step occurs at 80 - 150 °C as a small result of 5.1 % estimated weight loss which may be due to the loss of water molecules physically adsorbed on silica surface. The second step occurs at 200 - 550 °C with about 26.1 % weight loss which may be attributed to the loss of organically functional groups on material surface. The weight losses appeared in DTA as endothermic and exothermic peaks which refer to several chemical processes occur as a result of thermal degradation. On DTA curve, there are actually broad peaks from 374 °C to 509 °C. In this temperature range, the peak at 374 °C refers to exothermic effect for initially structural decomposition of aromatic ring of epoxy resin. At temperature of about 500 °C, the weight loss may be due to the loss of structural decomposition of glycol groups. At higher temperature of 550 °C, the peak refers to endothermic effect for reconstructing bonds in nano-silica. Thermal analysis data results are in close agreement with that there is the presence of epoxy groups on silica nanoparticle surface. These epoxy groups relatively strongly linked on silica nanoparticles and only weakened at elevated temperature of over 300 °C.

3.3. Hydrophobicity by dynamic contact angle (DCA) analysis

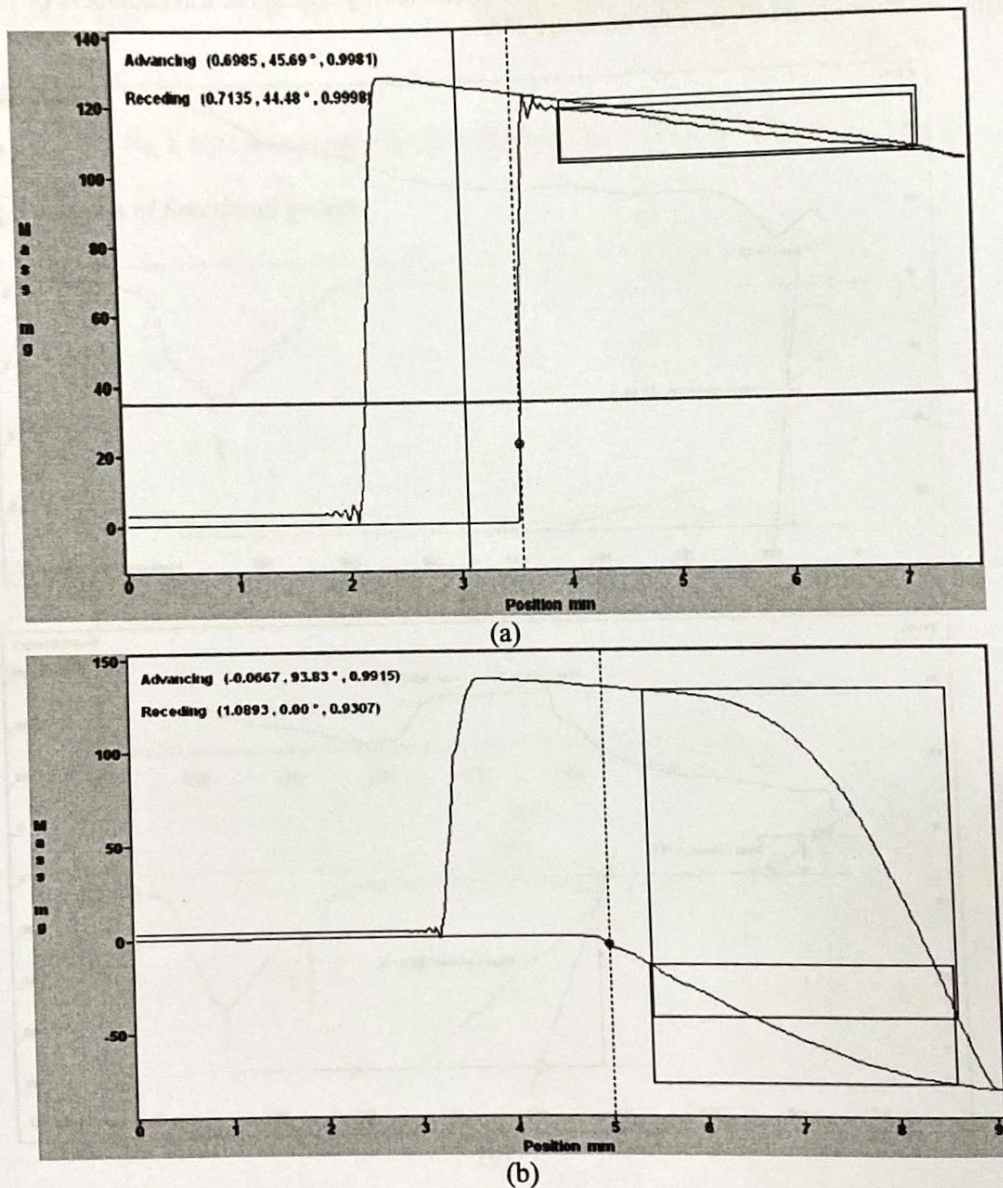


Fig. 5. Dynamic contact angle (DCA) measurements of nano-SiO<sub>2</sub> (a) and modified nano-SiO<sub>2</sub> (b)

The hydrophobicity and other solution properties of nano-silica before and after modification were assessed by using Wilhelmy method dynamic contact angle (DCA) measurement. The nano-silica sample was spread all across the rectangular glass with dimensions of 10 x 12 x 0.5 mm. The sample speed of soaking and dragging is 150 μm/s at the depth of 4 mm. Figure 5 shows the dynamic contact angle measurement results of nano-silica and modified nano-silica with epoxy, respectively. The wetting dynamic contact angle of nano-silica (figure 5a) was 45.69 ° and increased significantly to 93.83 ° after modification with epoxy resin (figure 5b). This angle is determined from the graph showing the relationship between the measured mass and the depth of the sample dipped into the liquid solution. The increase in wetting dynamic contact angle of nano-silica indicates that the change from the hydrophilic property into the hydrophobic (i.e. oilphilic) one. As already known, the silica

nanoparticles possess the -OH groups on the silica surface. Because of polar nature and hydrophilicity of these -OH groups, silica nanoparticles are hydrophilic. After modifying with epoxy resin, the rings in epoxy groups were opened followed by the combination of the -OH groups of silica. This reaction caused the appearance of nonpolar hydrocarbon groups on the surface, which then strongly decrease the hydrophilicity of silica (Nguyen et al., 2016).

The change on the surface of modified nano-silica can also be expressed by delayed characteristic ring of DCA measurement. According to figure 5a, for nano-silica, right after the ring touching water surface, the mass of silica increased sharply then decreased slowly. It shows a high affinity of nano-silica to water. Conversely, on figure 5b, for nano-silica modified with epoxy, after touching water surface, the mass of silica increased insignificantly then reduced dramatically below zero. It reveals a weak interaction between the modified silica and water. Therefore, the modification has changed the characteristic of material surface, reduced hydrophilicity of material. On the basis of this important property, modified nano-silica particles can be well dispersed in nonpolar environment such as oil medium.

### 3.4. Applicability of modified nano-silica on enhanced oil recovery

**Porous medium and preparation procedure:** Black crude oil with viscosity of 4,5 cP from the Diamond field (Cuu Long basin) was used for flooding tests. The synthetic seawater (SSW) was prepared by adding the main salt component found in sea water. The total amount of dissolved salt was 3 wt. % of NaCl. The specific surface area of modified nano-silica particles (also referred to polymer-coated silica nanoparticles (PSiNPs)) used in these experiments is 119 m<sup>2</sup>/g. Hydrophilic-hydrophobic KYPAM polymer with molecular weight from 13 x 10<sup>6</sup> to 21 x 10<sup>6</sup> g/mol was used as polymer for flooding. The solutions of PSiNPs referred to nanofluids were prepared at a concentration of 0.01 wt. %, 0.05 wt. %, 0.1 wt. %, and 0.5 wt. % in SSW, respectively. The prepared nanofluid (NF) solution was stirred for approximately 10 min using a magnetic stirrer to ensure a homogeneous solution before use. To avoid the aggregation of nanoparticles from solutions, a ultrasonic transducer (400 W and 0.5 Hz) was also used to keep the prepared solutions homogeneous and stable. The properties of the prepared solutions of SSW and NFs are reported in Table 1. Two sandstone cores were used for core flooding experiments. The cores were firstly cleaned and then dried before saturation of the synthetic seawater until core mass is constant.

Table 1. The properties of the prepared solutions of SSW and NFs

Sample	% (weight of PSiNPs)	Relative density	Viscosity (cP)	E <sub>D</sub>
SSW	-	1	-	1.00 (equivalent)
NF 1	0.01	1.019	1.009	2.10
NF 2	0.05	1.019	1.067	4.36
NF 3	0.1	1.020	1.160	6.23
NF 4	0.5	1.022	1.347	6.24

**Rock core flooding:** The core was loaded in the core-holder and oriented horizontally before injection of black crude oil. Prior to the flooding experiments, the cores were carried out by injection of volumes of crude oil until saturation. This was to eliminate air bubbles and/or re-establish oil connectivity within the cores. Water flooding was conducted prior to nanofluid flooding. Both tests were conducted at the same flow-rate of 0.5 mL/min. Synthetic seawater flooding (SWF) was firstly performed at 0.5 mL/min until no oil production was occurring for. At this stage, residual oil saturation at the end of water flooding (S<sub>or1</sub>) (Nguyen, 2013; Alberto et al., 2019) was defined. Afterwards, the injection was switched to nanofluid flooding of different nano-silica concentrations at 0.5 mL/min to measure if additional oil could be recovered. Residual oil saturation at the end of nanofluid flooding (S<sub>or2</sub>) was also defined. The oil displacement efficiency (E<sub>D</sub>) due to nanofluid was evaluated using the following equation:

$$E_D = \left[ 1 - \frac{S_{or2}}{S_{or1}} \right] * 100 \quad (1)$$

The oil displacement efficiency (E<sub>D</sub>) by nanofluid flooding ranged from 2.1 % to 6.24 % of original oil in place (OOIP) as given in Table 1. The results show that nanofluid flooding of the solution of PSiNPs at a concentration of 0.1 wt. %, combining with hydrophilic-hydrophobic KYPAM polymer, can produce the oil displacement efficiency (E<sub>D</sub>) of 6.23 %, which increased 5.23 % comparing to seawater flooding.

## 4. Conclusion

In this work, modified nano-silica particles (also referred to polymer-coated silica nanoparticles (PSiNPs)) were successfully synthesized using epoxy resin. The structural characterizations of silica particles before and after modification were studied using TEM/SEM, FTIR, TG/DTA, and DCA analysis. The results indicated that the change of material surface from hydrophilic to hydrophobic. The modified nano-silica particles were also used for rock core flooding to test enhanced oil recovery of nano material synthesized. The results show that nanofluid

flooding of the solution of PSiNPs at a concentration of 0.1 wt. %, combining with hydrophilic-hydrophobic KYPAM polymer, can produce the oil displacement efficiency ( $E_D$ ) of 6.23 %, which increased 5.23 % comparing to seawater flooding.

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