

## Research Article

# Synthesis and Characterization of a New Surface-Modified Nanoparticle Using Fluoroalkanoic Acids as a Wettability Alteration Agent

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Previously, condensate banking removal in gas reservoir is mitigated using chemical treatments to alter the wettability of the near-wellbore region. However, this technique performed unsatisfactorily as it reduces the surface free energy and affects the gas relative permeability negatively. Hence, alternative surface-modified nanoparticles using fluorine-based chemicals were developed as wettability alteration agents since fluorine exhibits a high degree of liquid repellency and nanoparticles introduce high surface roughness. The newly synthesized surface-modified nanoparticles were characterized using FTIR, DLS, FESEM, and TGA. FTIR results highlight the characteristic absorption of Si-O-C group at peak 1105 and 1106  $\text{cm}^{-1}$  in both fluoroalkanoic acids, demonstrating that fluorochemical molecules have been successfully coated onto silica nanoparticles. Nanoparticle sizes measured by DLS reported higher value than FESEM due to agglomeration, and the DLS measurement was done in hydrodynamic conditions. TGA analysis reveals decomposition at temperature between 100 and 150°C, indicating that these surface-modified nanoparticles can be utilized in an environment below 100°C. Higher decomposition was perceived on PFNA-modified nanoparticles as a thicker coating of PFNA is shrouding the silica nanoparticles compared to PFOA. Implications of the results will path the way for future research direction in using fluorine-based surface-modified nanoparticles as wettability alteration agents.

## 1. Introduction

Previous research on wettability alteration only deals with chemical treatment. Fluorochemical or fluorosurfactant is the exact name of the chemical that became the interest of many researchers [1–6] due to its electronegativity and reduction of surface free energy as the fluorine atoms network formed on the surface [7]. Wettability alteration is achieved by adsorption of a surface tension-reducing agent (fluorochemicals) that reduces the surface free energy and causes the repellency of another liquid.

However, chemical treatment is not sufficient to alter the wettability solely. Almost all chemical treatment successfully increases the liquid relative permeability and decreases the

irreducible liquid saturation. Nevertheless, this treatment also reduces the gas relative permeability, which is not favourable. Reduction of gas relative permeability will impede the gas well deliverability [3].

Introducing nanoparticles in chemical treatment is not only helping wettability alteration by reducing the surface free energy alone, but also adding another wettability alteration parameter: surface roughness. Increasing surface roughness will increase the wettability alteration ability [8].

Research using nanoparticles as a wettability alteration agent in gas condensate reservoirs only started in 2013 by Mousavi et al. [9]. Several researchers [10] using different types of fluorochemicals and nanoparticles, for example, Mousavi et al. [9] and Sharifzadeh et al. [11], utilized

TABLE 1: Details of fluoroalkanoic acids used in surface functionalization method.

Chemical	CAS number	Linear formula	Molecular weight	Melting point (°C)	Boiling point (°C)
Perfluorooctanoic acid (PFOA)	335-67-1	$\text{CF}_3(\text{CF}_2)_6\text{COOH}$	414.07	55	189
Perfluorononanoic acid (PFNA)	375-95-1	$\text{CF}_3(\text{CF}_2)_7\text{COOH}$	464.08	59	218

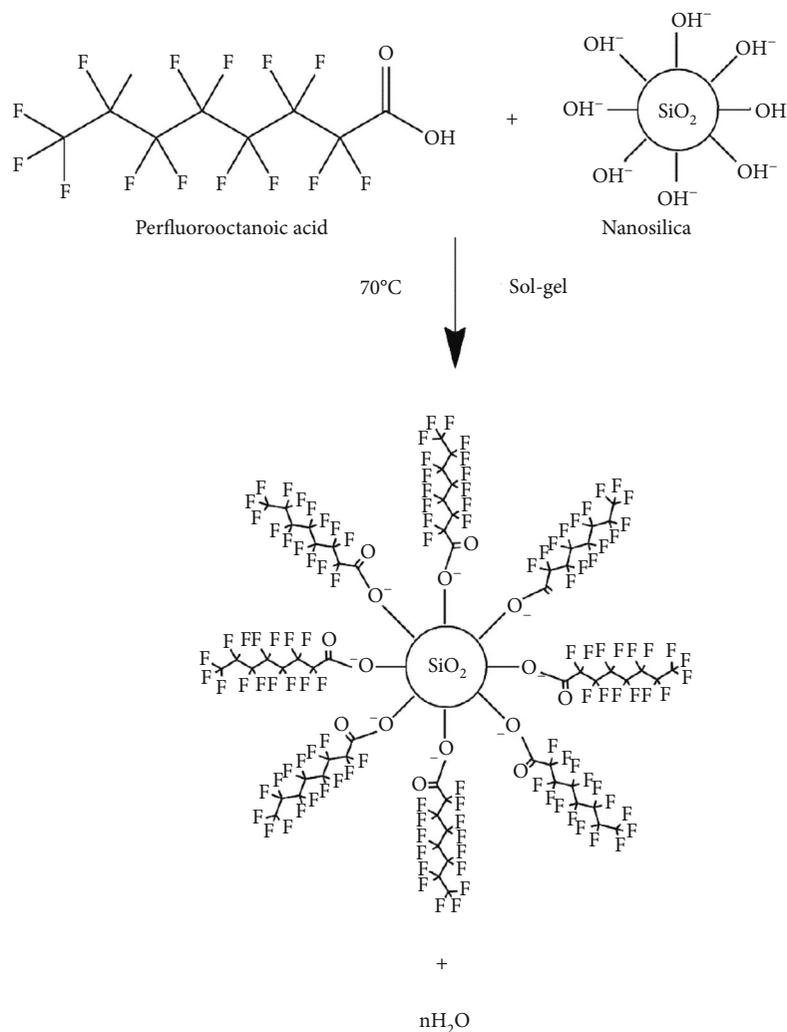


FIGURE 1: Schematic model of the silica nanoparticle formation with perfluorooctanoic acid (PFOA).

fluoroalkylsilane coated on nanosilica through sol-gel preparation. Esmaeilzadeh et al. [12], however, adopted different types of nanoparticles such as titanium oxide, silica, and carbon nanotube coated with fluoropolymer, while Sayed et al. [13] employed silica nanoparticles with fluorinated nonionic polymeric surfactant. Other studies have been applying commercial or proprietary chemicals, such as Franco-Aguirre et al. [14] with anionic commercial surfactant *Silnyl® FSJ* (SY -46% of fluoroaliphatic/hydrocarbon), Jin et al. [15] with *FG-40™* fluorosurfactant, and Aminnaji et al. [16] with *SurfacePore M™* nanofluid in fluoropolymer. To this day, there has been no research on using fluoroalkanoic acid as the coating agent for silica nanoparticles.

The motivation to use fluoroalkanoic acid as the coating agent comes from *Teflon®*. Fluoroalkanoic acid has been used to manufacture nonstick *Teflon®*-coated cookware. The non-stick surface makes the cookware convenient to use and easy to clean. It also requires little oil or butter for cooking, making it a healthy way to cook and fry food.

The emphasis of this study is on the newly developed fluorinated silica nanoparticles by using fluoroalkanoic acids as the coating agent. This newly developed fluorinated silica nanoparticles will act as a wettability alteration agent. Therefore, the objective of this study is to synthesize and characterize the new surface-modified nanoparticles using fluoroalkanoic acids as wettability alteration agents.

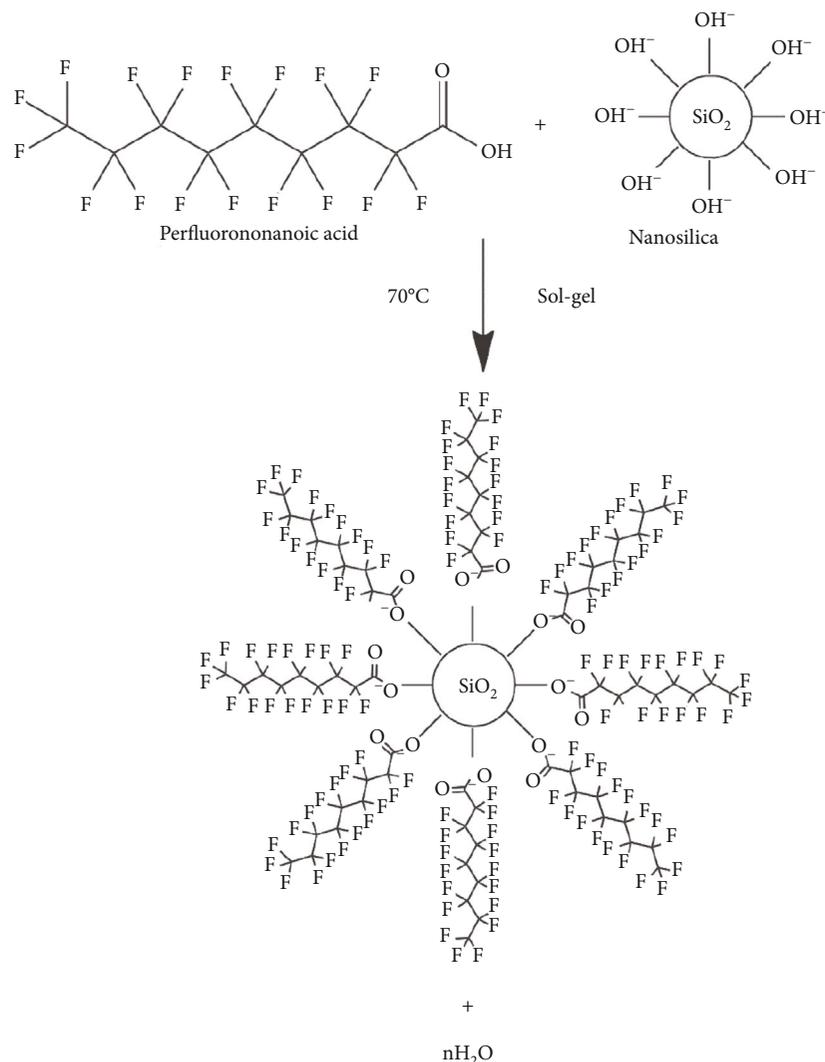


FIGURE 2: Schematic model of the silica nanoparticle formation with perfluorononanoic acid (PFNA).

## 2. Materials and Methods

Perfluorooctanoic acid (PFOA) and perfluorononanoic acid (PFNA) were purchased from Sigma-Aldrich (M) Sdn. Bhd. Details of these chemicals are presented in Table 1. Silicon dioxide (SiO<sub>2</sub>) or silica nanoparticles with hydroxyl functional group (-OH) were supplied by US Research Nanomaterials Inc. The size of nanoparticles is between 15 and 20 nm with S-type structure (spherical particles). Additionally, ethanol (CAS number: 64-17-5, 95%) was purchased from HmbG Chemicals.

The synthesis method was conducted using the procedure used in our previous study [17]. Fluorinated nanoparticles were prepared using postsynthesis functionalization method, by dispersing 0.02 g of SiO<sub>2</sub> nanoparticles in 10 mL ethanol, followed by adding 10 mL of a mixture containing PFOA (0.02 g) and ethanol (10 mL) to the solution at room temperature. Then, the solution was sonicated for 30 minutes with a temperature at 70°C, above the melting point of PFOA

and PFNA. This temperature is selected so that the fluorochemicals (PFOA and PFNA) could coat the nanoparticles easily above its melting point. The experiments above were then repeated using a different type of fluorochemicals, i.e., PFNA. Before characterization, the nanofluid—which is then coated nanoparticles suspended in ethanol—was then sent to the oven at a temperature above ethanol boiling point in order to remove any moisture residues with the nanoparticles. The end product of surface-modified nanoparticles after being dried is in a solid form (powder).

Fourier Transform Infrared Spectroscopy (FTIR) was used to determine the chemical bonds of the neutral-wetting alteration agents in this study. FTIR spectrum of the sample was obtained using a Perkin Elmer FTIR Machine, Model Spectrum One to detect the presence of fluorochemicals on the nanoparticle surface. The sample was then mixed with potassium bromide (KBr) pellets, and the FTIR device was used in transmission mode in the IR spectra range of 4000–400 cm<sup>-1</sup>.

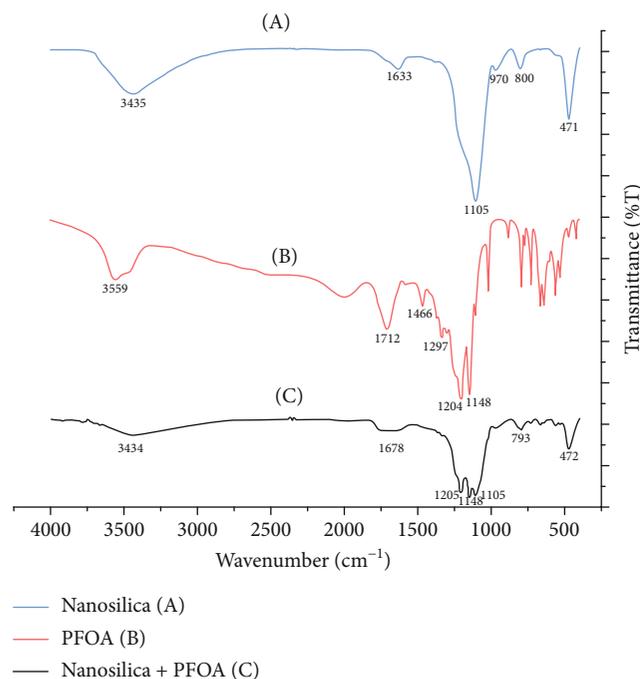


FIGURE 3: FTIR spectra of (a) unmodified nanosilica, (b) perfluorooctanoic acid (PFOA), and (c) PFOA-modified nanosilica.

The size distribution of the nanoparticles in this study was measured using Zetasizer Nano ZSP by Malvern Panalytical. Particle and molecule size is calculated using Dynamic Light Scattering (DLS). DLS tests the diffusion of particles under the Brownian motion and uses the Stokes-Einstein relationship to translate this to scale and size distribution. The Non-Invasive Back Scatter (NIBS) system is implemented to provide the maximum sensitivity at the same time as the largest size and concentration range.

Surface imaging was done using field emission scanning electron microscopy (FESEM). FESEM is an analytical technique used in material science to determine the morphological and topographical data of molecular surface structures. FESEM characterization was done using the VPFSEM model Supra55 VP by Zeiss.

Thermal stability analysis or thermal gravimetric analysis (TGA) was carried out using Simultaneous Thermal Analyzer (STA) 6000 by Perkin Elmer. This test method establishes a procedure for determining the material thermal stability and its fraction of the volatile component by monitoring the weight change that occurs as a sample is heated at a constant rate. In this study, the nanoparticles were fitted into the TGA sample pan with an initial weight of nominally between 5 and 20 mg. The thermal reaction occurred under a nitrogen atmosphere at a rate of 10°C/min from ambient temperature until 900°C. The decomposition rate will reflect the amount of coating on the nanoparticles and its stability temperatures.

### 3. Results and Discussion

The solution of fluorinated silica nanoparticles of 15–20 nm size was prepared in the laboratory. Figures 1 and 2 show

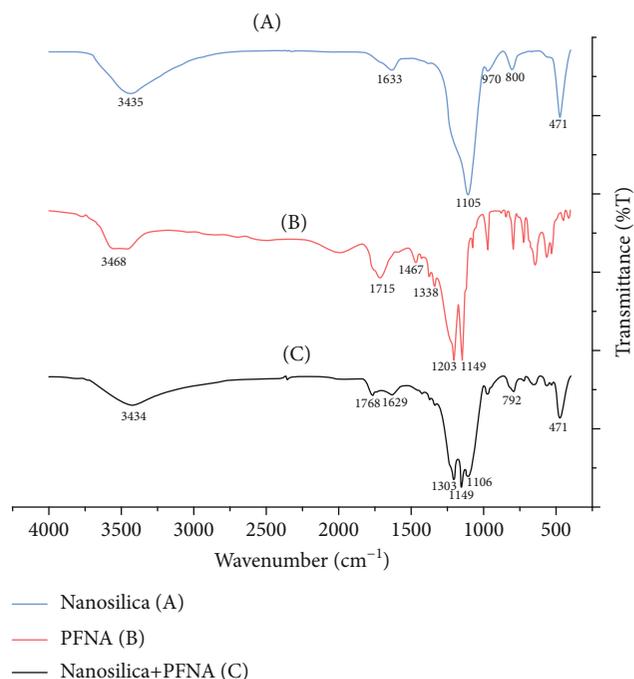


FIGURE 4: FTIR spectra of (a) unmodified nanosilica, (b) perfluorononanoic acid (PFNA), and (c) PFNA-modified nanosilica.

TABLE 2: FTIR spectra of the unmodified nanosilica.

Functional group	Peak position (cm <sup>-1</sup> )	Type of vibration
-OH	3500–3300	Broad and strong intensity absorption of -OH stretching
-OH	1633	Scissor bending vibration of O-H bond
Si-O-Si	1105	Si-O-Si asymmetric stretching vibration
Si-OH	970	Si-OH asymmetric vibration
Si-O-Si	800	Si-O-Si symmetric vibration
Si-O-Si	471	Si-O-Si bending vibration

the schematic model of the formation of functionalized silica nanoparticles in ethanol solution, with perfluorooctanoic acid (PFOA) and perfluorononanoic acid (PFNA), respectively. From the observation, the dehydration condensation can occur between the hydroxyl group (-OH) of the PFOA and PFNA molecules and the hydroxyl group (-OH) of the nanosilica at temperature 70°C. Then, the nonpolar end of the C-F bond on the molecule exposes out after the reaction. A substantial number of molecules can react with a single nanoparticle because there are so many hydroxyl groups on the nanoparticles' surface, increasing the adsorption significantly [18]. Therefore, neutral-wetting nanoparticles can be achieved using fluorochemical coating.

Figures 3 and 4 show the FTIR spectrum of unmodified nanosilica, perfluorooctanoic acid (PFOA), perfluorononanoic acid (PFNA), PFOA-modified nanoparticles, and

TABLE 3: FTIR spectra of perfluorooctanoic acid (PFOA) and perfluorononanoic acid (PFNA).

Functional group	Peak position for PFOA ( $\text{cm}^{-1}$ )	Peak position for PFNA ( $\text{cm}^{-1}$ )	Type of vibration
O-H	3559	3468	O-H stretching vibration
C=O	1712	1715	C=O stretching vibration
O-H	1466	1467	O-H bending vibration
C-O	1297	1338	C-O stretching vibration
C-F	1204, 1148	1203, 1149	C-F stretching vibration

TABLE 4: FTIR spectra of PFOA-modified nanoparticles and PFNA-modified nanoparticles.

Functional group	Peak position for PFOA-modified nanoparticles ( $\text{cm}^{-1}$ )	Peak position for PFNA-modified nanoparticles ( $\text{cm}^{-1}$ )	Type of vibration
C=O	1678	1768	C=O stretching vibration
C-F	1205, 1148	1303, 1149	C-F stretching vibration
Si-O-C	1106	1106	Si-O-C stretching vibration
Si-O-Si	793	792	Si-O-Si symmetric vibration
Si-O-Si	472	471	Si-O-Si bending vibration

PFNA-modified nanoparticles. The FTIR spectra of the unmodified nanosilica show absorption bands arising from asymmetric stretching vibration of Si-O-Si at  $1105 \text{ cm}^{-1}$  and symmetric vibration of Si-O-Si bonds at  $800 \text{ cm}^{-1}$ . Infrared analysis of the nanosilica shows a peak around  $970 \text{ cm}^{-1}$  which is attributed to Si-OH bonds, present on the unfunctionalized silica nanoparticles [19, 20]. The peak at  $471 \text{ cm}^{-1}$  is associated with Si-O-Si bonds for the rocking vibration [20–22]. Hydroxyl functional group shows an intense characteristic absorption band between  $3300 \text{ cm}^{-1}$  and  $3500 \text{ cm}^{-1}$  assigned to O-H stretching. Also, this band can be cross-checked through the  $1633 \text{ cm}^{-1}$  band due to scissor bending vibration of the O-H bond [23]. Table 2 shows the summary of IR spectra for the unmodified nanosilica.

Perfluorooctanoic acid (PFOA) and perfluorononanoic acid (PFNA) are from perfluorinated carboxylic acid family. Hence, based on the FTIR, its functional group contains C=O stretching bands at  $1712 \text{ cm}^{-1}$  and  $1715 \text{ cm}^{-1}$ ; C-O stretching bands at  $1297 \text{ cm}^{-1}$  and  $1338 \text{ cm}^{-1}$ ; O-H stretching bands at  $3559 \text{ cm}^{-1}$  and  $3468 \text{ cm}^{-1}$ ; and O-H bending vibration at  $1466 \text{ cm}^{-1}$  and  $1467 \text{ cm}^{-1}$ , for PFOA and PFNA, respectively. C-F stretching vibration is absorbed at  $1204 \text{ cm}^{-1}$  and  $1148 \text{ cm}^{-1}$  for PFOA and  $1203 \text{ cm}^{-1}$  and  $1149 \text{ cm}^{-1}$  for PFNA. Table 3 shows the summary of IR spectra for PFOA and PFNA.

In the surface-modified nanoparticles, the absorption peak of -OH group at  $3434 \text{ cm}^{-1}$  is broad with weak intensity, indicating that the monomers have reacted slowly. The peaks at  $1205 \text{ cm}^{-1}$  and  $1148 \text{ cm}^{-1}$  in PFOA-modified nanoparticles and  $1303 \text{ cm}^{-1}$  and  $1149 \text{ cm}^{-1}$  in PFNA-modified nanoparticles are the characteristic stretching peaks of the C-F bonds. The peaks at  $1678 \text{ cm}^{-1}$  and  $1768 \text{ cm}^{-1}$  in PFOA-modified nanoparticles and PFNA-modified nanoparticles, respectively, are corresponding to the characteristic of absorption C=O group. Figures 3(c) and 4(c) show that the characteristic absorption by the Si-O-C group appears at peaks  $1105 \text{ cm}^{-1}$  and  $1106 \text{ cm}^{-1}$ , revealing that fluorochemical molecules have

TABLE 5: DLS of the unmodified and surface-modified silica nanoparticles.

Sample	Size range (nm)
Unmodified nanosilica	10–15
PFOA-modified nanosilica	170–250
PFNA-modified nanosilica	210–450

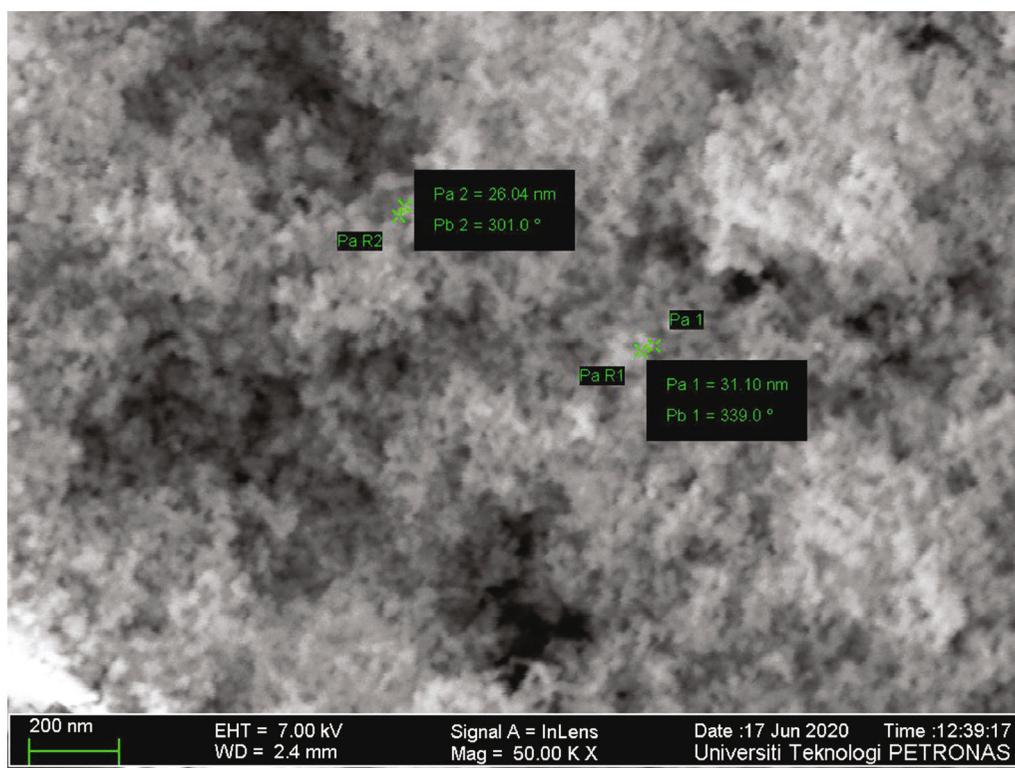
been successfully coated onto nanosilica particles by the dehydration condensation [18]. The stretching vibration absorption of Si-O-Si at  $800 \text{ cm}^{-1}$  in unmodified nanosilica is now shifted to  $793 \text{ cm}^{-1}$  in PFOA-modified nanoparticles and  $792 \text{ cm}^{-1}$  in PFNA-modified nanoparticles, while bending vibration of Si-O-Si are remained at  $471 \text{ cm}^{-1}$  and  $472 \text{ cm}^{-1}$ , respectively. Table 4 shows the summary of IR spectra for PFOA-modified nanoparticles and PFNA-modified nanoparticles.

Characterization of the functionalized particles by DLS (Table 5) showed the particles to be approximately 100–400 nm in size and that the uniformity was maintained during the functionalization of the silica nanoparticles. Meanwhile, measurement of nanoparticles using FESEM (Figure 5) showed a lower value, and the size is between 20 and 40 nm. Whilst FESEM measured the primary particle sizes of the nanoparticles, DLS measurements are often done in order to determine the true state of nanoparticles in porous media, especially when it was suspended in nanofluids. If the particles agglomerate, the values obtained from DLS will be larger than FESEM, as DLS measured the average hydrodynamic size of nanoparticles that was dispersed in a solvent.

The surface-modified nanoparticles were further characterized using the TGA method (Table 6). Although nanosilica were oven-dried under vacuum, all samples showed an initial mass loss between 27 and  $85^\circ\text{C}$ , which is assigned to the desorption of physisorbed moisture and residual ethanol



(a)



(b)

FIGURE 5: Continued.

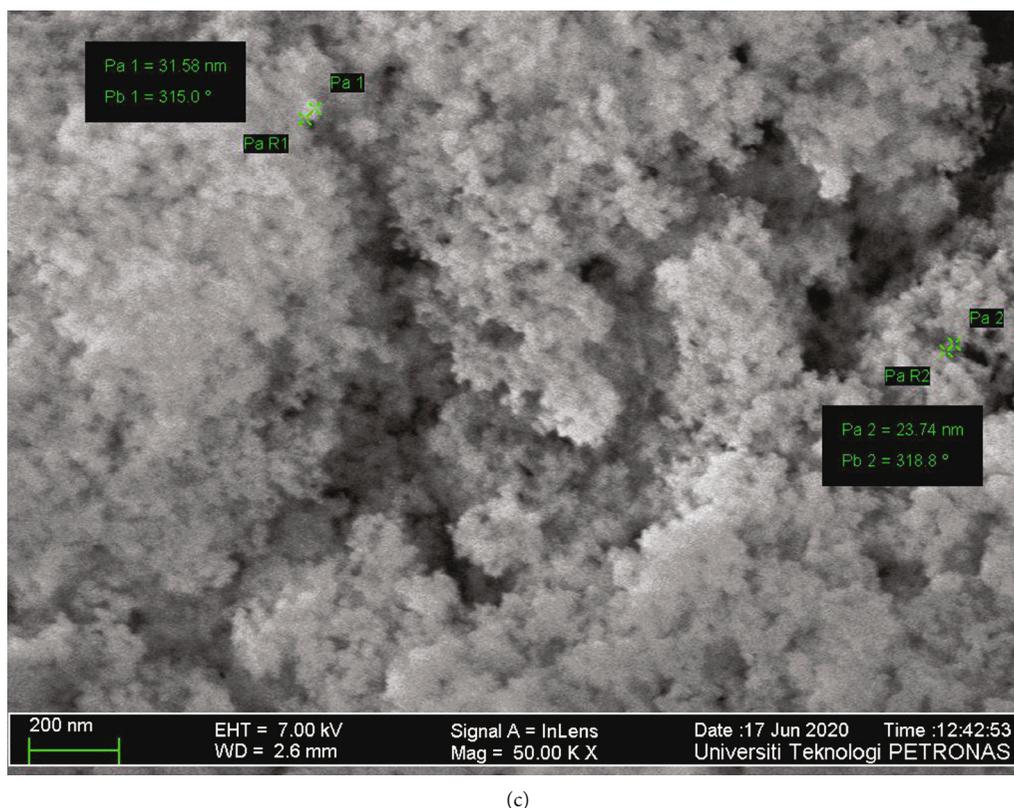


FIGURE 5: FESEM images of the (a) unmodified nanosilica, (b) PFOA-modified nanosilica, and (c) PFNA-modified nanosilica.

TABLE 6: TGA analyses of the unmodified and surface-modified nanosilica.

Sample	Temperature of 5% mass loss ( $^{\circ}\text{C}$ )	Residual at $100^{\circ}\text{C}$ (%)	Residual at $150^{\circ}\text{C}$ (%)	Residual at $900^{\circ}\text{C}$ (%)
Unmodified nanosilica	600	98.0	97.8	95.0
PFOA-modified nanosilica	65	72.4	46.0	44.6
PFNA-modified nanosilica	77	85.7	42.6	34.7

from functionalization of nanoparticles. With the surface-modified nanoparticles containing only a  $\text{SiO}_2$  network, they can be dried at elevated temperatures for an extended length of time, ensuring removal of any absorbed moisture and residual ethanol, without compromising any additional organic functionality. No significant weight reduction is observed up until temperature  $900^{\circ}\text{C}$  in unmodified nanosilica. Surface-modified silica nanoparticles, however, showed a noticeable weight reduction between the temperatures 100 and  $150^{\circ}\text{C}$ . PFOA-modified nanoparticles and PFNA-modified nanoparticles both seen 55% and 65% of decomposition, respectively. Higher decomposition was perceived on PFNA-modified nanoparticles as a thicker coating of PFNA is shrouding the silica nanoparticles compare to PFOA. Meanwhile, no decomposition is observed in unmodified silica nanoparticles, as no coating was shrouded on the unmodified silica nanoparticles.

These results are in agreement with those obtained by Mousavi et al. [9], Brassard et al. [19], and Jin et al. [18]. One interesting finding is that all the coating agents used in

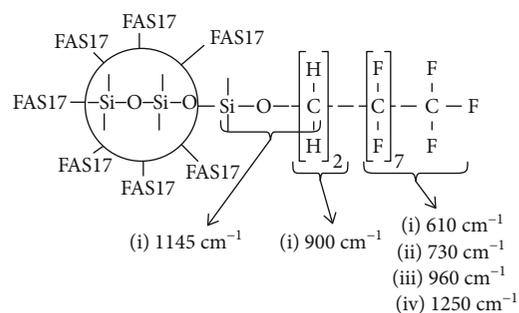


FIGURE 6: The functionalization of silica nanoparticles by FAS17 molecules is shown as well as the vibrational bonds and the corresponding wavenumbers (Brassard et al., 2011).

their study replace the  $-\text{OH}$  bond on the nanosilica. For example, Brassard et al. [19] and Mousavi et al. [9] were using fluoroalkylsilane (FAS) as the coating agent. Every H atom from each  $-\text{OH}$  bond around the silica nanoparticles is replaced by a Si atom of the FAS molecule as presented

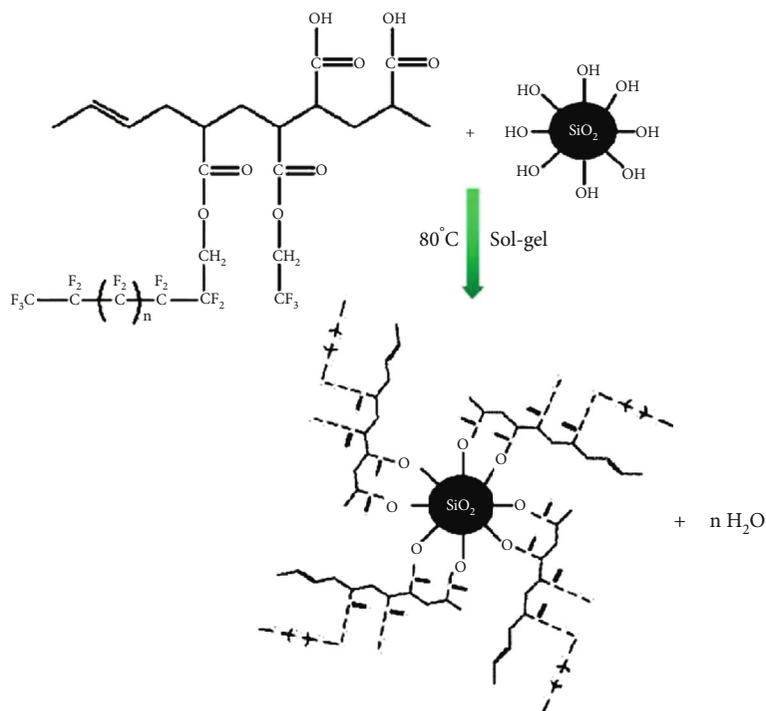


FIGURE 7: Sketch of nanosilica modification using FG40 fluorosurfactant (Jin et al., 2016).

in Figure 6. The new vibrational bonds of Si-O-C, C-H, and C-F are also shown in the same figure.

These similarities are also shared by Jin et al. [18], despite using a different type of coating agent. Although FG40 fluorosurfactant is a proprietary product, the FG40 molecule contains two carboxyl functional groups, as shown in Figure 7. The dehydration and condensation occurred between the -OH group of FG40 and the -OH group of the nanosilica at 80°C. A substantial number of FG40 molecules can react with a single nanoparticle because there are so many OH groups on the surface of the nanoparticles.

The combination of these findings provides some support for the conceptual premise that PFOA and PFNA can be used successfully as the coating agent in fluorinated-modified nanoparticles. An implication of this is the possibility that PFOA-modified nanoparticles and PFNA-modified nanoparticles are acting as wettability alteration agents in removing condensate banking. It is expected that the changes in the chain in nanosilica could improve gas productivity in the gas reservoir.

#### 4. Conclusions

In conclusion, fluorinated-modified nanoparticles can be developed using fluoroalkanoic acids such as perfluorooctanoic acid (PFOA) and perfluorononanoic acid (PFNA) as the coating agents. Fluorochemical molecule is successfully coated onto silica nanoparticles, and this is shown in FTIR results. Nanoparticle sizes measured by DLS and FESEM indicate agglomeration of nanoparticles in hydrodynamic environment. TGA analysis showed that these surface-modified nanoparticles can be utilized in an environment

below 100°C. The evidence from this study suggests that it is possible to synthesize a new surface-modified nanoparticle using fluoroalkanoic acids as a wettability alteration agent, the same coating agent used in nonstick *Teflon*<sup>®</sup>-coated cookware. Hence, the implications of these results will path the way for future research direction in utilizing surface-modified nanoparticles using fluoroalkanoic acids as a wettability alteration agent.

#### Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

#### Disclosure

Previous work of this paper has been presented in the Science Proceeding Series of the Asia International Multidisciplinary Conference in Johor, Malaysia, April 17-19, 2020.

#### Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

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