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## Silica nanoparticles as a high-performance filtrate reducer for foam fluid in porous media

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### ABSTRACT

During the fracturing operations for oils and gases, not only the oil and gas reservoirs, but also the nearby civil aquifers are often polluted by the invasion of fracturing fluid filtrates. In this study, we investigated the potential of silica nanoparticles as a high-performance filtrate reducer for a foam fluid in a porous media. First, the three factors affecting filtration reduction using nanoparticles, i.e., surface rheology, foam slipping, and foam stability, were described. Then, the foam filtration through a porous media in the core was measured using a dynamic fluid-loss device, and the effects of foam quality, pressure drop, and core permeability on the performance of the filtrate reducer were evaluated. The difficulty of bubbles flowing from a throat to a pore in a porous media was described by resistance gradient coefficient  $C_f$ , which is a combination of surface tension and viscoelastic modulus and increases by adding nanoparticles. Nanoparticles improve the roughness of the SiO<sub>2</sub>/sodium dodecyl benzene sulfonate foam film surface, thus increasing the slipping resistance  $F_{slip}$  when foams flow on the wall of a throat in a porous media. For the foams in a porous media, the diffusion of bubbles decreased in the presence of nanoparticles, and the growth rate of gas bubble size also decreased, thus increasing the foam resistance to gas channeling. The results of core filtration tests indicate that the fluid-loss-control properties increased with foam quality ranging from 0 to 85%, and the negative effects of pressure drop and permeability increase to foam filtration were weakened by adding SiO<sub>2</sub> nanoparticles. Thus, silica nanoparticles can be used as a high-performance filtrate reducer for a foam fluid in a porous media.

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### Introduction

The hydraulic fracturing of shale gas reservoirs has created a huge demand for high-performance fracturing fluids [1–5]. However, the main concern is the damage caused by the fluid filtrate invasion into formation, and not only the reservoirs but also nearby civil aquifers has been polluted [6–9]. Foam fluids are now well established as a viable alternative of traditional fracturing fluids because the liquid content of a foam fluid is small, thus reducing the damage to sensitive formations [10]. Benefited from a special two-phase microstructure, a foam fluid also has a smaller total fluid-loss volume than a traditional fracturing fluid [11,12]. Moreover, a high-energy gas has an excellent flowback capability that removes the fluid rapidly to minimize further damage to the formation [13,14]. However, some factors lead to uncontrollable filtration of foam and restrict its extensive use. First, the bubble

films of an aqueous foam are soft and could easily deform to match the size of the microchannels in a rock matrix, thus causing the bubble invasion of formation. Moreover, the soft bubble films can disintegrate to smaller bubbles by the complex pore-throat structure of a formation. Foams with smaller-size bubbles are more easily leaked. Second, the bubble surface of surfactant-stabilized foams is smooth [15], and the bubbles tend to slip on the wall under a low-pressure driving force. This is a disadvantage for the foam-plugging capability in the formation. Third, foam drainage, rupture of liquid films, and interbubble gas diffusion cause foam instability. Although crosslinked polymers such as guar, guar derivatives, and synthetic polymer have been used to stabilize foam, these polymers form insoluble residues in the formation, and the insoluble materials plug the pore throats, causing impaired leak-off and formation damage.

Thus, the enhancement of film strength, bubble surface roughness, and foam stability in a clean manner, especially in oil and gas reservoirs, are the key factors to decrease the foam filtration and extensive use of a foam fluid. During the past decade, there has been increasing interest in particle-stabilized foams

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(or Pickering foams) using inorganic nanoparticles such as silica because of their well-defined shape, availability in different sizes and narrow size distributions, and the chemical tenability of their surfaces [16–18]. Those nanoparticles formed dense particle films similar to a “colloidal armor” at the bubble surfaces, thus dramatically changing the physical properties of foam [19–21]. Many studies showed that nanoparticle-stabilized foams are stable for weeks or months even under extremely harsh conditions. This is because of the ability of surface layers formed by suitable selected particles to slow down or even prevent the foam rupture and gas diffusion (Ostwald ripening) between the bubbles. When the particles are sufficiently highly packed on the liquid–gas interface, the bubble surface becomes solid-like, and the elastic surface modulus  $E'$  increases [22,23]. The particle-stabilized bubbles do not deform. Moreover, the surface morphological structure of bubbles changed in the presence of nanoparticle adsorption. The compression of a particle layer, increasing its surface concentration, and further compression cause the liquid–gas surface to undulate, providing a rippled surface texture.

The use of particle-stabilized foams has gained attention because of their potential applications in the fields of food industry, mineral flotation, cosmetics, ceramics, and polymers [24–28]. Recently, nanoparticle-stabilized foams have been used in the oil and gas industry. Espinosa and Caldelas have developed a procedure for generating nanoparticle-stabilized supercritical CO<sub>2</sub> foams *in situ* in porous media and they reported that the flow resistance of foams increases with the addition of nanoparticles [29]. Aminzadeh-goharrizi et al. have investigated the effect of nanoparticles on the alteration of flow during the injection of CO<sub>2</sub> to porous media, and they indicated that nanoparticles result in the increase of sweep efficiency and decrease of gravity override, beneficial to flooding oil [30]. Yu et al. have reported the use of nanoparticle-stabilized foams as a high-performance agent for oil displacement. After the flooding of nanoparticle-stabilized foams, the permeability of Berea sandstone core did not change, indicating that plugging by nanoparticles does not occur, and permeability damage occurs [31]. Singh and Mohanty have investigated the synergistic effect between nanoparticles and surfactants on the performance of foam in porous media for oil recovery. Synergy between nanoparticles and surfactant could potentially be exploited for minimizing the usage of surfactants and maximizing the propagation distance of foam for subsurface applications [32]. Significant studies have been reported on the excellent performance of nanoparticle-stabilized foams in porous media. Nevertheless, these studies were mainly based on core flooding, with majority of the applications being confined to enhanced oil recovery (EOR). There are few reports about the properties and applications of nanoparticles as a filtrate reducer for a foam fluid in porous media.

In this study, the effects of silica nanoparticles on the mechanism of foam filtration were analyzed from three aspects: viscoelasticity of liquid–gas interface, bubble surface morphological structure, and foam stability in porous media. Moreover, the factors affecting foam filtration, i.e., foam quality, shear velocity, and matrix permeability were investigated.

## Experimental

### Materials

The SiO<sub>2</sub> nanoparticles (HDK, H15, purity >99.8 wt%) used in this study were purchased from Wacker Chemical Co., Ltd., Germany. The nanoparticles appeared as a white powder and almost spherical with an average diameter of ~14 nm. To increase the hydrophobicity, the nanoparticles were modified by coating with dimethyl siloxane by covalent bonds with a silanol group

density of ~1.0/nm<sup>2</sup>. The specific surface area of the SiO<sub>2</sub> nanoparticles was ~200 m<sup>2</sup>/g. These nanoparticles were modified by the manufacturer for increasing hydrophobicity via covalent-bond formation with dimethylsiloxane, and the density of the silanol groups on the nanoparticle surface was ~1.0/nm<sup>2</sup>. The specific surface area of the SiO<sub>2</sub> nanoparticles was ~200 m<sup>2</sup>/g, and its water contact angle was approximately 80°. The weight loss was less than 0.6 wt% after the particles were dried for 2 h at 105 °C. The surfactant used in this study was sodium dodecyl benzene sulfonate (SDBS, anionic surfactant, purity >99.0 wt%) obtained from Sigma (USA). Ethanol (Sinopharm Chemical Reagent Co., Ltd., China) was used as a co-solvent to solubilize the partially hydrophobic nanoparticles. Nitrogen with a purity of 99.9 wt% was used as the internal phase of foam and purchased from Tianyuan Inc. (China). Deionized water was double distilled from potassium permanganate to remove the traces of organic compounds. All the glasswares were cleaned in a surfactant-free cleaning agent to avoid any contamination. The surfactant-free cleaning agent is a mixture of 12 wt% of potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>) and 67 wt% sulfuric acid (H<sub>2</sub>SO<sub>4</sub>). The experiments were conducted at room temperature (25 °C) unless specified otherwise.

### Methods

#### Preparation and characterization of dispersions

The SiO<sub>2</sub>/SDBS dispersions were prepared by mixing the nanoparticles with a concentration from 0.0 wt% to 1.0 wt% and SDBS with a concentration from  $2 \times 10^{-3}$  wt% to 0.5 wt%. All the mixtures were stirred for 10 h and then ultrasonicated for 30 min. When the solutions appeared slightly hazy, they were used for the experiments.

The surface tension and interfacial dilational viscoelasticity of the dispersions were measured using a bubble/drop profile analysis tensiometer (Tracker-H, Teclis, France). This technique has been successfully used to evaluate gas–liquid interfaces and surface rheology [29–31]. The same setup was used to determine the SiO<sub>2</sub>/SDBS dispersions. To achieve a steady surface tension, droplets of the dispersions were stand for ~40 min, and then periodical oscillations were triggered on the droplets. Bubble area sinusoidal oscillations with a frequency of 0.1 Hz were used in the measurement, and the relative amplitude  $\delta S/S_0$  was 15%. A circulator bath was used to maintain the temperature of the measuring cell at 25 °C.

#### Micromodel experiments

A glass-etched model was designed to monitor the microstructure of the foam during filtration. As shown in Fig. 1, a two-dimensional network of pores and throats was etched in the micromodel using a photochemical method. To ensure the reliability of the etched porous media to the formation microstructure, the structure of pores and throats obtained from a core offered by Shengli Oilfield was patterned and used in the network etching. The dimension of the network was 80 mm × 80 mm × 6 mm, and the depth and width of the pores and throats were approximately 10 μm and 15–50 μm, respectively. To simulate the fluid filtration, two slots were designed in the model. The first slot faced the network inlet and worked as the foam filtration start side. The second slot faced the network outlet and worked as the foam leak-off outside. The width and depth of the slot were approximately 300 μm and 50 μm, respectively. The two slots were designed to ensure a uniform fluid filtration into the porous media and avoid the effect of local filtration.

Foams were generated by mixing gas and liquid in a foam generator. As shown in Fig. 1, the flow rate and quality of foam was controlled using a gas flowmeter and an ISCO pump by adjusting the flow rate of the gas and liquid. The foam was injected into the

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