



# Effect of surface modification of silica nanoparticles by silane coupling agent on decontamination foam stability

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## ABSTRACT

The effect of surface modification of silica nanoparticles by Dimethyldichlorosilane (DMDCS) on decontamination foam stability was investigated by the measurement of decaying foam volume with time using a Foamsan. The hydrophobicity of silica nanoparticles modified by DMDCS was characterized by active ratio via a floating test and contact angle analysis. Contact angle measurement has shown that silica nanoparticles surface become more hydrophobic as DMDCS concentration increases. Foam stability test in unmodified silica particles-surfactant mixtures revealed that silica nanoparticles-surfactant stabilized foams are much more stable than surfactant-stabilized foams and certain level of surfactant concentration is required for the synergy between silica nanoparticle and surfactant. In foam stability test with modified silica particle-surfactant mixtures, it was found that silica nanoparticles with the proper level of hydrophobicity shows the best performance in foam stability and this result was supported by optical and fluorescence microscope images.

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## 1. Introduction

Decontamination is a major activity to reduce potential releases and uptakes of radioactive materials, while nuclear facilities are being repaired, or under decommissioning (Riess et al., 2009). Compared to conventional aqueous decontamination, decontamination foam is advantageous in the cleaning of metallic walls, overhead surface, and elements of facilities due to the fact that it can overcome the problems caused by the complexity of the equipment and the location of radioactive contaminants (Nunez and Kaminski, 2007). In addition, the foam generates relatively low volume of waste and substantially decreases the amount of chemical decontamination agents used and the amount of secondary waste volume occurred during decontamination process, thus great attention has been on decontamination foam (Causse et al., 2009). Although there are some advantages in decontamination foam, decontamination efficiency may be dropped as a result of the decreasing time of chemical decontamination agents to the contaminated area if foams are unstable. Therefore, it is the key issue for successful decontamination process to stabilize the decontamination foam.

Surfactant is a foaming agent that facilitates formation of foam and it is an essential material for decontamination foam. Since decontamination process is usually conducted in severe condition caused by the strong acidity of chemical decontamination agents, surfactants that can function well in strong acidic condition are needed for decontamination process. Among numerous kinds of surfactants, fluorosurfactants or fluorinated surfactants are great candidates to be suitable for this condition. Fluorinated surfactants, are synthetic organofluorine chemical compounds that contain a perfluoroalkyl group and known for its superiority to nonfluorinated surfactants in terms of wetting action and both thermal and chemical stabilities in harsh environments (Srivastava et al., 2005; Pabon and Corpart, 2002; Baudequin et al., 2011; Blin et al., 2006).

It is well known that small solid particles are used as antifoam agents in surfactant-stabilized foams. Hydrophobic particles added to an aqueous surfactant solution initially move to the film surfaces and then bridge both surfaces of thinning film caused by drainage and finally the film rupture occurs (Garrett, 1979; Aveyard et al., 1994). However, particles do not always destabilize foams. Fairly hydrophilic particles are present in the aqueous phase of the foam films and enhance foam stability by slowing down drainage (Kruglyakov and Taube, 1972; Pugh, 1996). The rate of decreasing drainage can be explained by stratification process. The particles in the films tend to have an orderly arrangement,

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resulting in stepwise thinning of the film. The driving force for the particle to exhibit stepwise thinning is the chemical potential gradient of particle at the film periphery, where the particle leaves the interface and a vacancy is formed in its place (Kralchevski et al., 1990). The stratification behavior of silica nanoparticles was found to increase film stability. The tendency toward orderly arrangements of particles was reported to decrease significantly as the polydispersity of the nanoparticles increased (Sethumadhavan et al., 2001; Sethumadhavan et al., 2004). In order to improve foam stability even more, particles should adsorb onto and remain around bubbles in water. For the aqueous system with silica particles, only particles of the correct wettability were found to stabilize the foam. Partially wetted particles were shown to accumulate at bubble surfaces, providing a barrier preventing rupture and coalescence (Ip et al., 1999). Particles adsorbed on bubble surfaces form a dense layer which prevents coalescence and halts completely disproportionation (Subramaniam et al., 2006; Abkarian et al., 2007). Network formation between adsorbed and non-adsorbed particles can lead to additional stabilization reducing, in this case, drainage of water between bubbles (Cervantes Martinez et al., 2008).

Silica nanoparticles have been widely used in many fields and can also play a role in stabilizing foams. Strong hydrophilic property of silica nanoparticles caused by the hydroxyl group ( $-\text{OH}$ ) on the surface can be adjusted through surface modification for the best performance as a foam stabilizer. The surface of silica nanoparticle was modified with cationic surfactant cetyltrimethylammonium bromide (CTAB), and optimal conditions such as temperature and the amount of CTAB (Ma et al., 2010). Fumed silica nanoparticles were coated to different extents of hydrophobicity with silane coupling agent and the use of these small particles in stabilizing aqueous foams in air-water-particle systems in the absence of any other surface-active substance was demonstrated (Binks and Horozov, 2005).

Although extensive studies have been performed on surface modification of silica nanoparticles by either surfactant or silane coupling agents, little has been reported on the correlation between foam stability and the hydrophobicity of surface modified silica nanoparticles in particle-surfactant system. In the present study, the effect of surface modification of silica nanoparticles by DMDCS on foam stability was systematically investigated. Silica nanoparticles have been prepared with various levels of hydrophobicity with DMDCS and their hydrophobicity was characterized by measuring active ratio via floating test and contact angle with a sessile drop method. Foam stability test in unmodified silica particles-surfactant system was first conducted to confirm the synergy between silica nanoparticles and surfactant and then, the stability of foams in the system of surfactant-modified silica particles with different levels of hydrophobicity by DMDCS was evaluated by the use of a Foamscan instrument and the result was supported by optical and fluorescence microscope images.

## 2. Experimental

### 2.1. Materials

Silica nanoparticles with the primary particle size of 12 nm and BET surface area of 175–225  $\text{m}^2/\text{g}$  were purchased from Sigma-Aldrich Co. Water used for sample preparation was ultrapure, which have been double dis-tilled and passed through a Nanopure (Sybron-Brinkman Inc.) ion exchange system. Nitric acid solution (1.0 M  $\text{HNO}_3$ ) with a purity of greater than 98.0% was purchased from Sigma-Aldrich Co. and used for pH adjustment of aqueous solutions. Two kinds of cationic surfactants such as Cetyltrimethylammonium bromide (CTAB) and Dodecyltrimethylammonium

bromide (DTAB) were purchased from Sigma-Aldrich Co. Zonyl TBS is an anionic sulfonate fluorinated surfactant, whose structure is given by  $\text{R}_f\text{CH}_2\text{CH}_2\text{SO}_3\text{Y}$  where  $\text{R}_f$  is  $\text{F}(\text{CF}_2\text{CF}_2)_{1-9}$  and Y is hydrogen or ammonium. It was purchased from DuPont Company and used without any further purification. Dimetyldichlorosilane (DMDCS) and toluene with a purity greater than 99.0% were purchased from Sigma-Aldrich Co. and used for surface modification of silica nanoparticles. Rhodamine B (Sigma-Aldrich Co.) was used to stain silica nanoparticles for high resolution fluorescence microscope observation.

### 2.2. Methods

#### 2.2.1. Preparation of aqueous dispersions of silica nanoparticles

Silica nanoparticles was weighed into a glass bottle of dimensions 6.7 cm (h) by 4.6 cm (d) followed by adding pure water or aqueous surfactant solution of different concentration. The mixture was then dispersed using an ultrasonic processor (Vibracell, SONICS) operated at an output of 50 W for 2 min.

#### 2.2.2. Zeta potential of aqueous dispersions of silica nanoparticles

The change in the surface charge of nanoparticles with pH is important to determine the range of pH where surfactant adsorption should occur. 1.0 wt% of silica nanoparticles were dispersed in distilled water with the pH being adjusted by adding  $\text{HNO}_3$ . The dispersions were left overnight at 25°C in a temperature incubator (DF-95B, Duri Science) and the zeta potential was measured using an electrophoretic light scattering spectrometer (ELS-8000, OTSUKA) at 25 °C.

#### 2.2.3. Surface modification of silica nanoparticles

DMDCS with different levels of concentration were added to a mixture of 2.5 g of silica powder and 150 mL of toluene and the suspension was stirred for 12 h at room temperature. Then, the solid was collected by centrifugation (3000 rpm, 10 min), washed twice with ethanol (50  $\text{cm}^3$ ), and dried at 383 °C for 3 h. The samples were prepared by grinding the silica powders using a mortar.

#### 2.2.4. Characterization of surface modified silica nanoparticles

In order to investigate the modification of the silica nanoparticles surface, the contact angle and active ratio were measured. The powdery samples were placed on the slide glass and pressed into a thin layer under pressure and then a single droplet of water was dropped onto it. The contact angle against water on a horizontal surface of a layer was obtained with a drop shape analysis system (Phoenix-1, SEO). The floating test was performed to measure an active ratio, where the active ratio is represented by the ratio of the floated product to the overall weight of the sample after mixing in water and stirring vigorously (Wang et al., 2007; Mihajlovic et al., 2009; Zhang et al., 2008). 100 mg of silica sample modified by DMDCS was placed into 100 mL of water, followed by stirring vigorously for 1 min, and silica nanoparticles floated to the top of water-air interface were collected. The collected particles were dried at 100 °C for 24 h and the weight of the collected sample was measured.

#### 2.2.5. Characterization of foam properties

The foam properties (foamability and foam stability) were characterized using a foam test apparatus (Teclis/IT Concept, France). 3 mL of particle-surfactant solutions were added to a base unit and the foam was generated by blowing nitrogen gas through a porous glass filter at the bottom of a glass tube. When the foam volume reached to 160  $\text{cm}^3$ , gas supply was stopped and the foam volume change was monitored by using a CCD camera. Foamability was measured from the time for foam to increase from 50  $\text{cm}^3$  to 150  $\text{cm}^3$  with gas injection at a constant flow rate and foam

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