

Effect of Cetyltrimethyl Ammonium Bromide on Foam Stability and SiO₂ Separation for Decontamination Foam Application

거품제염을 위한 실리카 나노입자와 CTAB (Cetyltrimethyl Ammonium Bromide)의 거품안정성 및 분리특성 평가

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As part of planning for waste minimization, decontamination foam has been considered as a potential application for the cleaning of radioactive contaminant. In this study, we synthesized silica particles to improve foam stability by varying synthesis parameters. Cetyltrimethylammonium bromide (CTAB) was found to influence the stability of the decontamination foam. The reason was that higher interaction between SiO₂ nanoparticles and surfactant at the air-water interface in aqueous solution is beneficial for foam stability. CTAB can also be used as an additive for the aggregation of silica nanoparticles. In the separation of SiO₂ nanoparticles, CTAB plays a critical role in the nanoparticles flocculation because of the charge neutralization and hydrophobic effects of its hydrocarbon tails.

Keywords: Decontamination foam, Foam stability, SiO₂, Cetyltrimethylammonium bromide, Silica separation

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원자력 시설 내 방사능을 포함하는 물질의 제염을 위한 방법 중 거품제염은 2차폐기기물의 양을 저감시킬 수 있는 장점을 지니고 있다. 본 연구에서는 거품안정성을 증가시킬 수 있는 실리카 나노입자를 여러가지 조건을 달리하여 합성하였다. Cetyltrimethylammonium bromide(CTAB)는 거품제염제의 거품안정성에 많은 영향을 나타내었다. 이러한 거품안정성이 향상된 이유는 제염제 내에 공기와 용액간의 계면에서 실리카 나노입자와 계면활성제의 반응으로 생각된다. 또한, CTAB는 실리카 나노입자의 분리특성에서도 우수한 성능을 나타냈었다. 실리카 나노입자 분리시, CTAB의 탄화수소기에 의한 소수성과 전하중성화에 의해서 실리카 나노입자의 응집현상을 향상시켰다.

중심단어: 거품제염, 거품안정성, 실리카 나노입자, Cetyltrimethylammonium bromide, 실리카 나노입자 분리

1. Introduction

Decontamination technology is necessary to reduce radioactive component and contamination of complex equipment during decommissioning of nuclear power plants. There are a number of methods for decontamination process including chemical, electrochemical, foam, and mechanical contamination technologies. Among them, decontamination foam is considered to have potential application for the cleaning of radioactive contaminants from metallic walls, overhead surfaces, and complex components [1]. Moreover, decontamination foam could generate a small amount of secondary waste owing to its volume expansion. In order to increase decontamination efficiency, it is essential to improve the foam stability with a small amount of chemical decontamination agent. Recently, silica nanoparticles have been used as foam stabilizers for the decontamination technology [2]. Silica nanoparticles have also been widely used for catalyst, biomolecule separations, chromatographic supports, miniaturization of electronic devices, and semiconductor nanostructures [3,4]. Yoon et al. reported that the silica nanoparticles showed increased foam stability compared to surfactant solution alone [5]. The nanoparticle has been used as foam stabilizer, which adsorbed at fluid/fluid interface and stabilized emulsions or bubbles in foams [6]. In spite of improvements of foam stability, the surfactant, silica nanoparticle (1wt%), and viscosifier (foam stabilizer) are still used. Moreover, studies on high decontamination

efficiency with foam, and on reducing the use of chemical agents and secondary waste are not very common. At present, it is difficult to separate and recover silica nanoparticles during the decontamination waste treatment process. Nanoparticles are different from classical solid particles due to their very small particle size and consequent specific properties. Thus, the separation of nanoparticle, with high recovery rate, should be also developed.

Cetyltrimethylammonium bromide (CTAB) is a typical cationic surfactant with sixteen long carbon chains and positively charged surface. CTAB can be used to synthesize silica nanoparticles of various sizes and shapes by controlling its amount. It is also well known that the adsorption of CTAB on the silica nanoparticles surface modifies its hydrophilic/hydrophobic state and determines its affinity for the fluid interface [6]. Recently, it was reported that the silica surface was modified as a function of the amount of CTAB added, and that the conjunction of CTAB and SiO₂ to form an interfacial nanocomposite could influence the physical and chemical properties of the nanocomposite, including surface tension and wettability [7,8]. In addition, the CTAB-SiO₂ interaction influences the viscoelastic properties of interfacial films, as well as the formation and stability of hexadecane in water emulsions in the presence of NaCl at constant ionic strength [9].

In line with achieving decontamination efficiency, the disposal of secondary radioactive waste is another important issue. Because these wastes are hazardous to humans

and the environment, it is necessary to treat them with carefully selected methods. The addition of nanoparticles to decontamination waste solution was attempted to increase the efficiency of classical water treatment processes. Recently, Liu et al. investigate the addition of CTAB in SiO₂ nanoparticle suspensions [10]. Hu et al., and Lien and Liu performed a coagulation-flotation process using a CTAB to address the treatment of chemical-mechanical polishing (CMP) waste water [11,12]. The addition of CTAB reduced the sludge volume after the coagulation-flotation process, better than the addition of cationic polymers did.

In this work, we synthesized SiO₂ particles and investigated the foam properties associated with the CTAB-SiO₂ interaction reaction. We also evaluate the separation of SiO₂ nanoparticle from the waste solution by using CTAB. The hydrophobic interaction between SiO₂ particles and the hydrocarbon tail of the surfactant, could improve the foam stability. The flocculation of silica nanoparticles could be quickly induced by adding CTAB and the SiO₂ particle size became larger with an increase in the amount of CTAB added.

2. Experimental

All the chemicals are commercially available and used as received unless otherwise stated.

For the SiO₂ preparation, the cetyltrimethylammonium bromide (CTAB, 2.0 g) and sodium hydroxide solution (2 M NaOH, 7.0 ml) were added in deionized water and the mixture was stirred and heated at 80°C. To clear this solution, tetraethylorthosilicate (TEOS, 9.3 ml) was added to the solution via rapid injection. The white precipitate was observed within 3 min and the solution was maintained at 80°C for 2 h. After the reaction time, the SiO₂ particles were washed by water and methanol, and dried under vacuum oven. The morphology and structure of SiO₂ were analyzed by a field emission scanning electron microscopy (FE-SEM, Hitachi) and powder X-ray diffraction (XRD, PANalytical)

using Cu K α radiation ($\lambda=1.5405$) at 40 kV and 15 mA. Thermogravimetric analysis (TGA, Mettler-Toledo) was carried out to measure the amount of CTAB in SiO₂ composite under flowing N₂ with a heating rate of 5°C · min⁻¹.

The foam stability and liquid volume of the foam were analyzed with commercially available Foamscan instrument (Teclis/IT Concept, France) which observe the foam properties such as foamability, foam volume and liquid fraction. The foam stability and drainage of liquid from the foams were determined by conductivity measurements of the foam volume. In order to measure the foam properties, the SiO₂ solution that SiO₂ particles (1wt%) are dispersed in 100 ml of deionized water at pH 2 was prepared. The concentrated 1 M HNO₃ solutions was used to adjust pH of SiO₂ solution. The SiO₂ solution was stirred at 400 rpm for 1 h. The solution was poured into cylinder glass column and bubbled by sparging nitrogen gas at a flow rate of 200 ml · min⁻¹ through the SiO₂ solution. After the foam volume produced by porous disk was achieved at 200 ml, and its foam and liquid volume were recorded by monitoring the change in foam volume and liquid volume using a CCD camera and measuring the conductivity as a function of time. For the comparison, 1wt% fumed silica nanoparticle (M-5, Cabosil) with 1wt% ElotantTM Milcoside 100 (EM 100, LG Household and Health Care) as a surfactant was also dispersed in 100 ml deionized water.

For the silica nanoparticle separation, the CTAB was also used as additive to aggregate the silica nanoparticles in the SiO₂ solution, and its separation efficiency was investigated. The desired concentration of CTAB (powder) was added into the SiO₂ solution in the range of 0.05 mM to 5 mM. The SiO₂ amount in the solution was 1wt%. The SiO₂ solution included CTAB was stirred at 400 rpm for 60 min. The SiO₂ solution was centrifuged at 3500 rpm for 30 min. After centrifugation, the top clear solution in the centrifuge tube was used to measure the transmittance of solution by Turbiscan (Formulation, France). Turbiscan detected the backscattering and transmission of monochromatic light ($\lambda=880$ nm) through the sample. Measurement

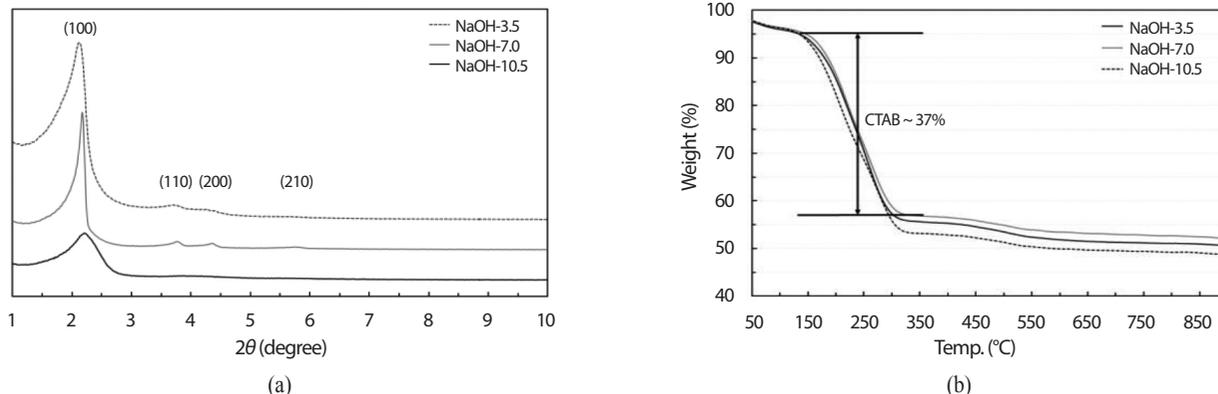


Fig. 1. XRD patterns (a) and TGA profiles (b) of SiO₂ synthesized by different NaOH amount 3.5, 7.0, and 10.5 ml, respectively.

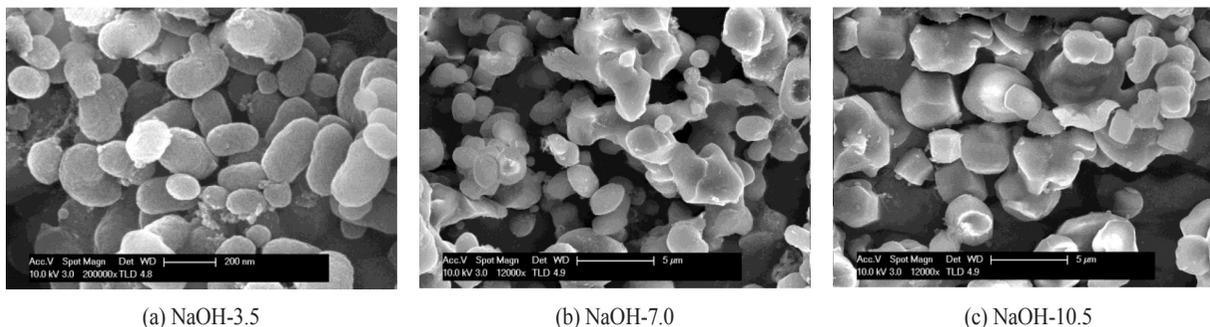


Fig. 2. SEM images of SiO₂ particles synthesized by different NaOH amount 3.5, 7.0, and 10.5 ml, respectively.

was conducted for 60 min. The results showed the change of transmittance of light versus time. Inductively coupled plasma atomic emission spectroscopy (ICP-AES, OPTIMA) was used to measure the SiO₂ content in the solution after centrifugation. The particle size of SiO₂ after adding CTAB was also measured using the particle size analyzer. All the measurements were carried out at room temperature.

3. Results and discussion

Fig. 1(a) shows the small-angle XRD patterns of as-synthesized SiO₂ particles. The SiO₂ samples show an intense (100) peak and three weak (110), (200), and (210) peaks (except for NaOH-10). In the case of SiO₂ synthesized with 7 ml of NaOH (NaOH-7), the sharp intensive

(100) peaks are observed at $2\theta = 2.18^\circ$, which is associated with the formation of a highly ordered hexagonal symmetry (p6mm structure). As shown in Fig. 1, however, NaOH-10 shows broad (100) peak and no weak peaks appears even though the spherical morphology and smaller particle size are observed in SEM image. In addition, the main peak (100) of SiO₂ particle was slightly shifted toward higher 2θ angles with increasing to the NaOH amount, indicating the creation of larger internal d-spacing. The pH values with NaOH amount of 3.5, 7, and 10 ml were 11.7, 12.4, and 12.8, respectively. Because the amount of TEOS and CTAB was fixed, the optimal condition of NaOH amount is 7 ml with a pH value of 12.4 for highly ordered structure of SiO₂ in this study.

The TAG profiles of as-synthesized SiO₂ particles are depicted in Fig. 1(b). The first region from room temperature

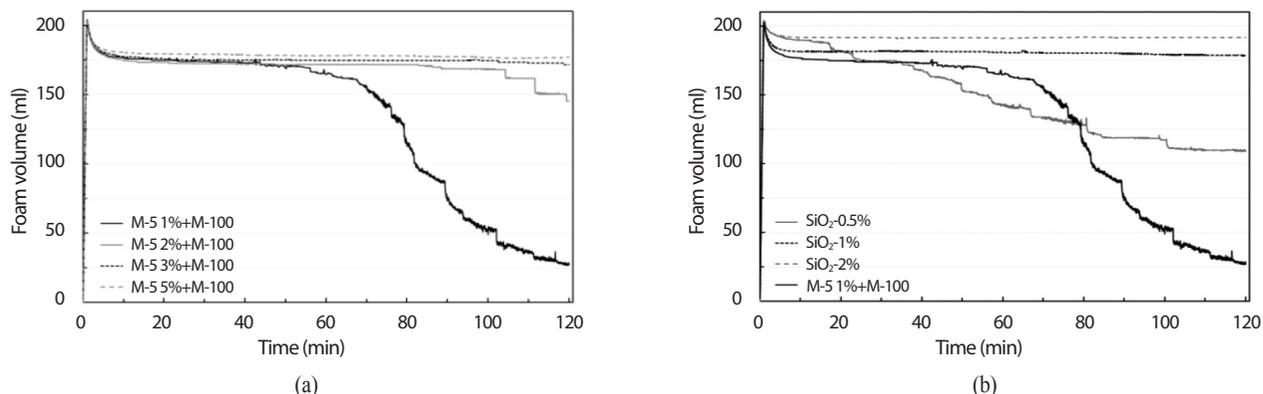


Fig. 3. Foam stability of surfactant solution with different SiO₂ (M-5) amount (a), and foam stability of as-synthesized SiO₂ with different amount (b).

to 150°C is attributed to the release of absorbed water on the silica surface. The drastic weight loss (40%) caused by decomposition of organic molecule and CTAB in the SiO₂ matrix was observed in the temperature range of 150-350°C. No obvious weight loss was observed at above 550°C, indicating that the pure SiO₂ particles can be obtained. As to the large weight loss for as synthesized SiO₂, the CTAB was mainly decomposed about 37% weight loss in SiO₂ particles. In comparison to the weight loss, the NaOH-7 was smaller than other samples, indicating that surfactant probably interacts more strongly.

The morphology of SiO₂ particles was observed by FE-SEM and shown in Fig. 2. It is clearly reveal that the particle size of SiO₂ increased as a function of NaOH amount. The particles exhibited tubular shape (Fig. 2(a)), but the other samples show irregular and micron-sized particles. It is well known that the rate of hydrolysis of silane and condensation of the siloxane bond depend strongly on the charge state [13]. The pH is increased by adding the NaOH amount, indicating the pH value 11.7, 12.4, and 12.8, respectively. Even though the difference of pH is relatively small, the lower pH value results in the smaller SiO₂ particle size compared to higher pH because of relatively slow rates of hydrolysis and condensation of TEOS.

For the foam stability measurement, the aqueous foam was generated and its stability was recorded by using

Foamsan analyzer. Fig. 3(a) shows the foam volume of silica nanoparticle solution (M-5, Cabosil) containing EM-100 (1wt%) as a surfactant at pH 2. The foam volume of SiO₂ particle (1wt%) with surfactant (1wt% EM-100) retained at 165 ml until 50 min, but dramatically decreased after 60 min due to the liquid drainage during the foam aging. However, the foam stability was improved with increasing SiO₂ amount. The high SiO₂ amount could be favor the foam stability because of higher interactions between SiO₂ nanoparticles and surfactant at the air-water interface and aqueous solution. Compared to the foam stability of SiO₂ nanoparticle, as-synthesized SiO₂ particles also investigated their foam stability without any surfactant. In contrast to the SiO₂ nanoparticle (M-5), the foam stability of as-synthesized SiO₂ particles is higher than SiO₂ nanoparticle (M-5) except for 0.5wt% of as-synthesized SiO₂. As can be seen in Fig. 3(b), the foam volume of 1 and 2wt% as-synthesized SiO₂ noticeably decreased within few minutes and retained their foam volume for 120 min with low foam volume decay. Especially, the foam volume of 2wt% as-synthesized SiO₂ remained at a constant value of 191 ml (95% of initial volume (200 ml)), which is significantly higher than that of all SiO₂ nanoparticles (M-5).

In order to further investigate the foam properties of as-synthesized SiO₂ particles, the foam stability was also measured according to the amount of NaOH, and CTAB during

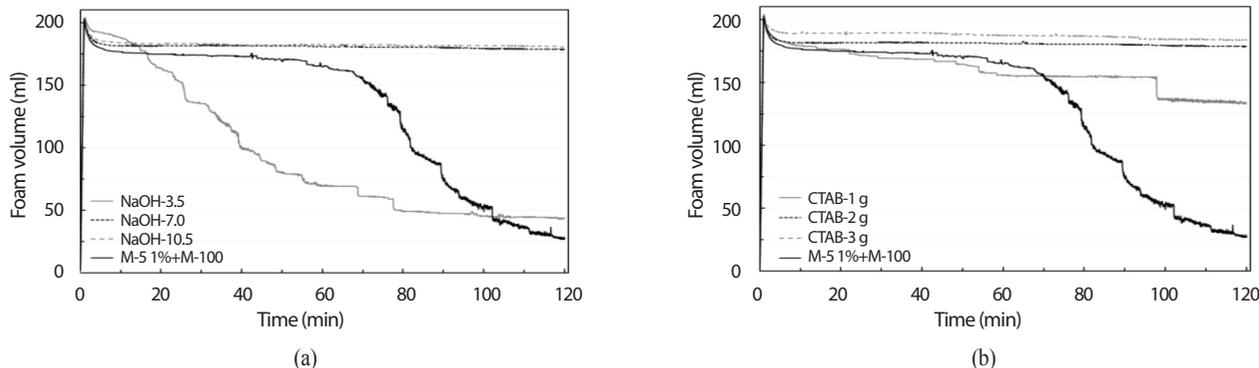


Fig. 4. Foam stability of as-synthesized SiO₂ particles as a function of NaOH (a) and CTAB (b).

the synthesis and depicted in Fig. 4. As shown in Fig. 4(a) and 4(b), the synthesis condition for SiO₂ particle could influence the foaming stability. For example, as the amount of NaOH and CTAB increase, the foam stability retained at highest foam volume retention during foam scanning, indicating that the higher amount of NaOH and CTAB leads to improve the foam stability of as-synthesized SiO₂ particles. The lower pH value results in the smaller SiO₂ particle size compared to higher pH because of relative slow rates of hydrolysis and condensation of TEOS (Fig. 2(a)). It was reported that more hydroxyl groups could be existed on the silica surface at higher pH value, thus the surface charges of silica are negative [8]. When the CTAB and silica were added into the NaOH solution, the ionic interaction between the positive and negative charges on the silica surface make the organic chain of CTAB array around the silica particle. The CTAB reacts with the hydroxyl groups on the silica surface and surface energy of silica particles decreases. Moreover, higher amount of NaOH leads to enlarge the contact area of silica and CTAB interaction resulting in large agglomerated silica particle with high CTAB amount. The CTAB amount is also important factor to determine the wettability of the particle-network at the air-water interface due to the balance between electrostatic and hydrophobic interactions [14]. As the electrostatic interaction CTA⁺ cations and SiO⁻ moieties continues takes place leading to the formation hemimicelles. Further adsorption of CTA⁺ cations on the

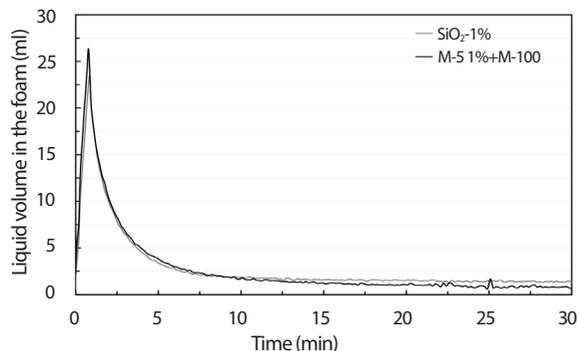


Fig. 5. Liquid volume in the foam of as-synthesized SiO₂ (1wt%) and surfactant solution (1wt% M-5).

particle surface may then lead to the formation of reverse hemimicelles [15,16]. The addition of a low concentration of CTAB to the dispersion of silica nanoparticle leads to an increase of hydrophobicity of the silica solution [17]. At the high CTAB concentration, the hydrophobic interaction between silica and hydrocarbon tails of surfactant molecules attached to the particle surface also increases consequently, favoring the formation of silica particles with a larger size. During the formation of a CTAB monolayer around the particles, the pH value is varied due to the chemical exchange between silanol groups and the CTA⁺ cations.



The flocculation of silica particles with CTAB could

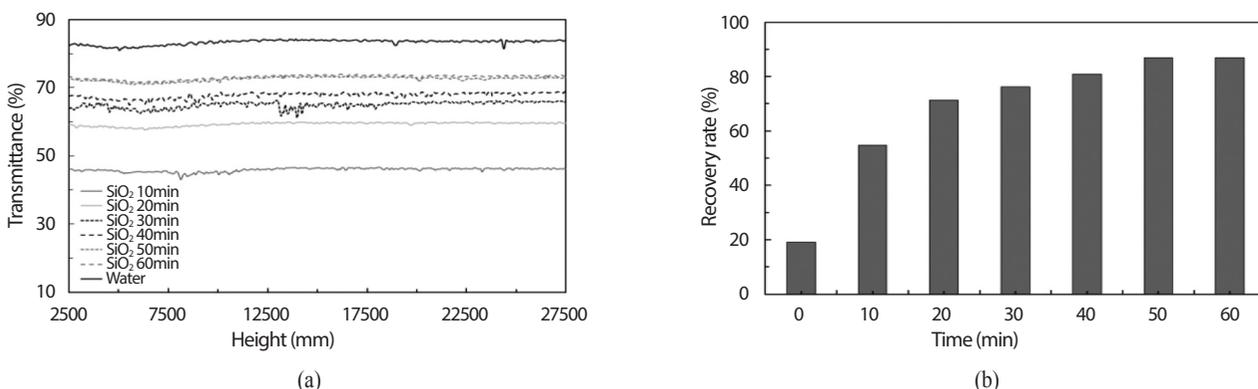


Fig. 6. Transmittance (a) and recovery rate (b) of SiO₂ solution without CTAB according to the centrifugation time.

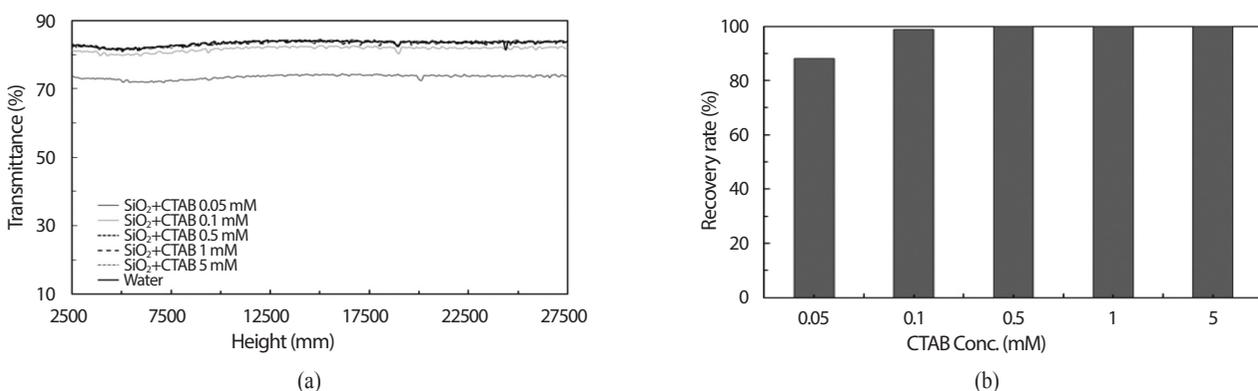


Fig. 7. Transmittance (a) and recovery rate (b) of SiO₂ solution as a function of CTAB amount after centrifugation (for 30 min).

also modify their surface wettability from hydrophilic surface due to the negative surface charges to hydrophobic surface [9]. So, we concluded that high amount of NaOH and CTAB during the silica synthesis is beneficial for improving the foam stability of silica particles. The liquid contents in the foam are also important factor to measure the foam stability. Fig. 5 shows the liquid volume of silica solutions (1wt% as-synthesized SiO₂, 1wt% M-5 with M-100). It should be noted that the liquid volume in foam is the overall liquid volume in the total foam at a specific height of the foam column. As shown in Fig. 5, the liquid volume of two samples is progressively decayed over time due to drainage. There is no difference of liquid volume, which is comparable with surfactant solution.

CTAB can be also used to separate the SiO₂ particles as additive for the aggregation of silica nanoparticles [11,18]. Generally, silica nanoparticle shows a negative charge on the particle surface and the zeta potential $|\zeta|$ remains lower than 30 mV leading to their stability [19]. Liu et al. reported the aggregation of silica nanoparticles by cationic surfactant CTAB and proposed the coagulation mechanism of silica nanoparticle with CTAB. We used the CTAB as additive for separation of silica nanoparticle after foam stability measurement and the effect of CTAB for the SiO₂ separation. Fig. 6 shows the transmittance of SiO₂ solution after centrifugation without CTAB. The interpretation of the transmission profiles was based on the change in the light transmission caused by change of SiO₂ contents. As

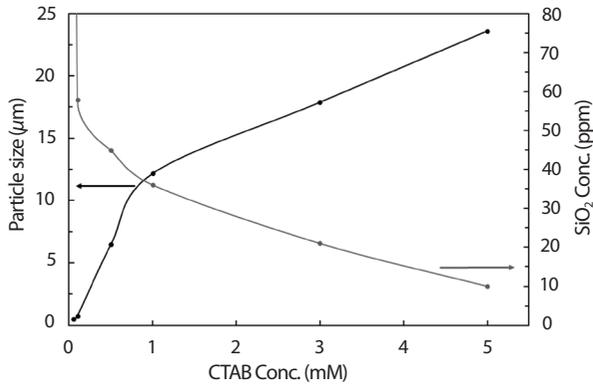


Fig. 8. Particle size and SiO₂ concentration as a function of CTAB amount after centrifugation (for 30 min).

shown in Fig 6(a), the transmittance of 1wt% SiO₂ solution without CTAB linearly increased according to the centrifugation time. However, no difference of transmittance increasing time was observed after 50 min, which means SiO₂ nanoparticles are still suspended in the solution. We also calculated the recovery rate to determine the separation efficiency. The reference was based on the transmittance of distilled water. About 83% of recovery rate of SiO₂ solution was achieved after 60 min by centrifugation. Fig. 7 shows the transmittance of SiO₂ solution with a series of CTAB containing different CTAB amount. The SiO₂ solution was centrifuged for 30 min. When the CTAB added in the SiO₂ solution, the transmittance becomes more increase and similar with that of water even at small concentration of CTAB (0.1 mM). It is indicating that efficient agglomeration

of SiO₂ particles can be obtained because of interaction SiO₂ and CTAB. The recovery rate of SiO₂ with CTAB was also similar with that of water (except for 0.05 mM of CTAB). From Fig. 7, the concentration of CTAB ≥ 0.1 mM could effectively agglomerate the SiO₂ particles and very few silica contents remain in the solution. The Fig. 8 shows the particle size and SiO₂ contents according to the addition of CTAB amount. All measurements were carried out after reaching the equilibrium. At low concentration of CTAB (0.1 mM), no increasing particle size of SiO₂ was observed and SiO₂ concentration was also high, indicating that it is low to induce the efficient aggregation. However, the particle size of SiO₂ was dramatically increased according to the CTAB amount (0.5 mM), and the SiO₂ amount was also decreased after centrifugation. The particle size and contents of SiO₂ are dependent on the CTAB concentration. It is reported that the charge neutralization seems to be main reason for silica aggregation, the hydrophobic effect from hydrocarbon tails of CTAB may also contribute to the aggregation as well [20]. Liu et al. proposed the aggregation mechanism of silica nanoparticle; when the low concentration of CTAB, little CTAB can be absorbed on the silica surface. As increasing the CTAB, negative surface charge is reduced and agglomeration may occur until charge neutralization is achieved due to the hydrophobic interaction (Fig. 9). Finally, when the CTAB concentration is too high, a bilayer of CTAB could envelop the particle surface and thus re-stabilize the silica nanoparticle suspensions [19].

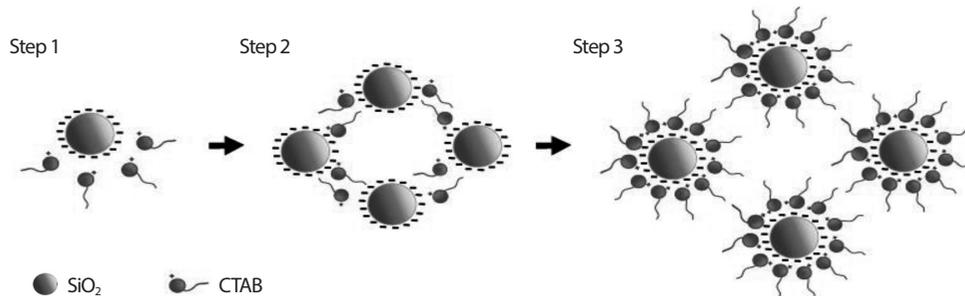


Fig. 9. Schematic mechanism of SiO₂ aggregation with CTAB on SiO₂/water interface.

4. Conclusion

In this study, we synthesized SiO₂ particles via co-condensation method based on the NaOH-TEOS catalytic reaction with CTAB and SiO₂ nanoparticle separation by CTAB was also investigated. From the results of XRD, TGA, and SEM, NaOH amount could influence their physical properties such as d-spacing and morphology of primary particles. We confirmed that amount of SiO₂, NaOH, and CTAB affected foam stability compared to silica nanoparticle (M-5) with surfactant (M-100). From the foam stability test, the foam volume of as-synthesized SiO₂ (2wt%) remained at a constant value of 191 ml (95% of initial volume (200 ml)), which is significantly higher than that of all SiO₂ nanoparticles (M-5). The addition of CTAB produced the flocculation of SiO₂ particles and increased the hydrophobic interaction between SiO₂ particle and hydrocarbon tail of surfactant, which in turn improved the foam stability. The flocculation of silica nanoparticle added by CTAB could be quickly achieved for only 30 min. The particle size of SiO₂ was larger as CTAB amount increased, and SiO₂ contents in the top solution were decreased after centrifugation.

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