



## Factors controlling the formation and stability of foams used as precursors of porous materials



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### ARTICLE INFO

#### Article history:

Received 11 November 2013

Accepted 28 March 2014

Available online 3 April 2014

#### Keywords:

Foamability

Foam stability

Foam rheology

Suspension rheology

Particle stabilized foams

Porous materials

Solid foams

### ABSTRACT

The remarkable stability of particle-stabilized foams and the opportunity to use them for production of novel porous materials have been attracting the researchers' attention in the recent years. The major aim of the current study is to clarify the factors, controlling the foamability and stability of foams, formed from concentrated silica suspensions in the presence of the amphoteric surfactant CAPB. The experiments showed that: (1) two regions can be defined with respect to suspension foaminess: Region 1 – good foaming and Region 2 – strongly suppressed foaming. The foam volume decreased linearly with the increase of suspension viscosity, so that Region 2 appears as a result of the excessively high suspension viscosity. (2) Based on foam stability four sub-regions were observed in Region 1: region 1S – stable foams, which can be dried to form stable porous materials; 1UD – foams which are unstable with respect to water drainage and collapse upon drying; 1UC – foams which are stable to drainage, but are unstable to drying, due to crack formation; 1UF – unstable foams which completely fall apart upon drying. (3) Foams in Regions 1S and 1UC had yield stress above 10 Pa which prevented liquid drainage, while foams from Region 1UD drained because of their lower yield stress. (4) The particles in the foams assembled in a 3D network in the Plateau channels and the nodes, while surfactant stabilized the foam films between the bubbles. These results define the conditions, appropriate for formation of stable, highly porous silica materials with low mass density, which can be further modified (sintered, impregnated, hydrophobized, etc.) to serve as catalyst supports, porous filters, insulating materials, etc.

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### 1. Introduction

Particle-stabilized foams [1–26] and emulsions [25–36] have attracted considerable research interest recently, due to their unique properties and potential technological applications. Among the most interesting applications are those related to the possibility for fabrication of porous materials with complex hierarchical structure, by using particle-stabilized drops or bubbles as templates [18–28]. Several groups described procedures for generation and stabilization of foams by appropriate particle–surfactant mixtures [1–17]. In some of these studies it was shown that inorganic colloidal particles can be hydrophobized in situ, by adsorption of short-chain amphiphilic molecules, and these surface-modified particles can act as very efficient foam stabilizers [2,3,9,10,14,23]. The effect of the composition of the initial colloidal suspension on the air volume fraction, average bubble size, and bubble size

distribution in the formed wet foams was investigated [23]. In other studies, [12–14,16] aqueous foams prepared by dispersions of disk-shaped Laponite particle and containing the cationic surfactant cetyltrimethylammonium bromide (CTAB), were studied. All these studies showed that the foams could be stabilized by two basic, qualitatively different mechanisms: (a) adsorption of surfactant-modified particles on the bubble surface and the related stabilization of the foam films by the particles; (b) formation of a network by aggregated particles in the foaming phase [12,17].

The particle modification by adsorbing surfactant molecules is highly specific and depends on the type and concentration of surfactants and particles [2,10,12,15,16]. Therefore, it is still very difficult (or impossible) to predict what would be the foamability of such particle–surfactant mixtures and what would be the stability of the generated foams. For example, Sepulveda and Binner [19] claimed that suspension viscosity had a little effect on the final volume of the generated foam, whereas Dhara and Bhargava [22] observed that the total porosity of the final material decreased linearly with the increase of suspension viscosity. Thus we see that even the “simple” effect of suspension viscosity, which is governed

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by the surfactant–particle interactions, is under discussion and might be system dependent.

The major aims of the current study are: (1) to reveal the mechanisms of foam stabilization for concentrated silica dispersions, containing the zwitterionic surfactant cocoamidopropyl betaine, CAPB; (2) to determine the main physico-chemical factors which control the foamability and foam stability with respect to liquid drainage, bubble coarsening, bubble coalescence and foam drying for the foamed CAPB-silica suspensions.

CAPB surfactant was chosen for this study because, as shown in Section 3.6 below, it leads to formation of foams with foam films stabilized by surfactant molecules (not by particles). Accordingly, the final porous structure is with open cells – these foam films break in the process of drying, thus opening direct access between the neighboring bubbles. From this viewpoint, CAPB differs from most of the surfactants used in this area which provide particle-stabilized foam films, thus leading (usually) to closed cells in the final porous material.

To achieve these aims, we performed foaming experiments under well-defined conditions, combined with a set of complementary model experiments. Namely, the rheological properties of CAPB-silica dispersions were characterized to clarify their relation to the foamability of the dispersions and to the stability of the generated foams. Optical observations of the foam films and of the real foams were performed to clarify the mechanism of foam stabilization (by particles or by adsorbed surfactant molecules). Foam stability during long-term shelf storage and upon drying was monitored. Whenever possible, quantitative expressions are proposed to describe the relations between suspension and foam properties.

The focus of the current study is mainly on the formation and stability of the wet foam precursors. The subsequent process of foam drying to produce the final porous material (and the related changes in the foam structure during drying) is investigated in detail in a separate study, see Ref. [24].

## 2. Materials and methods

### 2.1. Materials

We used amorphous precipitated silica particles (Tixosil 365, Rhodia) with silica concentration in the batch powder  $\geq 87.5$  wt%. The commercial silica powder contains also 1.5 wt% soluble salts, and 11% physically and chemically bound water. According to the producer, specific surface area of the silica particles is  $150 \pm 10$  m<sup>2</sup>/g, mean agglomerate diameter is  $d_{50} \approx 3.5$   $\mu$ m, and mass density of the particle material is 2100 kg/m<sup>3</sup>.

Amphoteric surfactant cocoamidopropyl betaine (CAPB) was used as additive for foam stabilization. Commercial product Tego Betain F50 from Goldschmidt GmbH, containing 40 wt% of active CAPB, was used. According to Tzochcheva et al. [37] the commercial Tego betaine F50 contains also admixtures of 2.8 wt% non-reacted coco fatty acids and 7.3 wt% NaCl. The composition of the coco fatty acids is 5–9% octanoic acid (C<sub>8</sub>); 6–10% decanoic acid (C<sub>10</sub>), 44–52% dodecanoic acid (C<sub>12</sub>), 13–19% tetradecanoic acid (C<sub>14</sub>); 8–11% hexadecanoic acid (C<sub>16</sub>); 1–3% octadecanoic acid (C<sub>18</sub>) and 5–8% oleic acid. Respectively, the main surfactant CAPB is also a mixture of molecules with a distribution of the hydrocarbon chain lengths, reflecting the composition of the initial coco fatty acid source from which CAPB is synthesized.

Deionized water from Elix 3 module (Millipore) was used for preparation of all solutions and suspensions. Sodium chloride and sodium hydroxide from Sigma–Aldrich were used in suspension preparation, both being of analytical grade (>99.9% pure).

### 2.2. Suspension preparation

Initially, suspensions with 21 wt% particle concentration were prepared by mixing the necessary amounts of silica particles and deionized water. This mixture was placed on a ball mill drive (BML-2, Witeg) at 125 rpm for 30 min. Afterwards, the suspension was homogenized for 2 h with a pulse sonicator (SKL-650 W, Syclon) which was set to 1 s long pulses with power output of 650 W (followed by 0.5 s off), using a sonotrode with diameter of 10 mm. Next, portions of 2 M solution of NaOH (in proportion of 0.12 g solution per 1 g silica in the suspension) were added to adjust the pH to 8.5. Finally, this suspension was homogenized for additional 5 min at the ball mill and for 60 min at the sonicator. Dynamic light scattering measurements on diluted suspensions showed that the aggregate size has been reduced after this procedure down to the range between 50 and 300 nm.

Suspensions with lower particle concentrations were obtained by diluting the original 21 wt% suspension with the necessary amount of deionized water or CAPB solution, just before the foaming experiments. Suspensions with particle concentration above 21 wt% were prepared by stepwise addition of more silica and NaOH to the initial 21 wt% suspension, followed by homogenization for 5 min at the ball mill and for 1–2 h at the sonicator after each step. Maximum concentration of  $25.7 \pm 0.5$  wt% silica could be obtained in this way. Further increase of particle concentration led to severe suspension gelling.

The working pH = 8.5 was chosen after an initial series of screening experiments about the effects of pH and electrolytes (NaCl and CaCl<sub>2</sub>) on the foaming and foam stability. These experiments showed that best results with respect to foam stability are obtained at low electrolyte concentrations and high pH. Therefore, all systematic experiments are performed without additional electrolytes and at pH = 8.5. Higher pH was not used to avoid possible (partial) dissolution of the silica particles in the form of sodium silicate.

### 2.3. Foam generation and foam stability

Planetary mixer (Kenwood Chef Premier KMC 560, 1000 W) was used for foaming of the suspensions. A given mass of the suspension,  $m_s$ , was introduced in the mixer and a complementary quantity of surfactant stock solution was added, so that the total amount of suspension + surfactant solution was always 400 g. The mixer was initially set at minimum mixing speed (32 rpm) for 4 min to form a homogeneous silica–CAPB suspension without foaming. Then higher mixing speed of 165 rpm was applied for 10 min. According to the model of the flow inside this mixer, developed by Chesterton et al. [38] the mixing speed in our experiments corresponds to shear rates of  $\approx 100$  s<sup>-1</sup>. Afterwards, mixing was stopped and foam samples were taken for determination of the air volume fraction and for characterization of the foam stability with respect to liquid drainage, bubble Ostwald ripening, and foam drying.

To determine the volume fraction of trapped air,  $\Phi$ , we filled a petri dish with foam of known volume,  $V_F = 28$  mL, and measured its mass,  $m_F$ . Then  $\Phi$  was determined from the relation  $\Phi = (1 - m_F/V_F\rho_S)$ . Here,  $\rho_S$  is the mass density of the silica suspension, calculated assuming ideal mixing of particles with mass density 2100 kg/m<sup>3</sup> (as provided by the producer) and water (997 kg/m<sup>3</sup>).

To determine foam stability with respect to liquid drainage, a foam sample was transferred in a petri dish of 100 mL with height of 3.5 cm, and observed for several days to check whether aqueous phase is collected at the bottom of the container, as a result of drainage. Similar sample was used to monitor the stability of the foams with respect to bubble collapse and bubble Ostwald

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