



# Studies on stability and properties of micro and nano-particle-laden ionic microbubbles

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## ABSTRACT

Ionic microbubbles are miniature gas bubbles of  $<100\ \mu\text{m}$  in diameter, which are surrounded by charged ions. Stability of ionic microbubble is an important parameter which is used to analyse the efficiency of microbubble for fine particle separation. This article reports the experimental observations of stability of ionic microbubbles in presence of micro and nano particles. The effects of concentration of surfactant, temperature, and pH on the stability of the ionic microbubble are studied. A model has been developed to interpret the stability of the ionic microbubble based on drainage kinetics. Bubble size and its holdup, affecting the stability of the suspension are also enunciated in the present article. The study may be useful for fine particle separation in batch and continuous mode with the help of ionic microbubble.

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## 1. Introduction

Microbubble structure and its stability are known to play a significant role in determining the grade and recovery by its flotation. Sebba [1,2] has first described about the microbubble, having some colloidal properties with size range  $25\text{--}100\ \mu\text{m}$ . He proposed that microbubbles consist of a gaseous inner core surrounded by a shell composed of two surfactant layers in addition to the third layer of surfactant that stabilizes its structure. Numerous studies are presented in literature on microbubble generation [3] and applications such as: removal of pulp fibre [4], predispersed solvent extraction of dilute products [5], protein recovery [6,7], clarification of suspensions [8,9], separation of organic dyes from wastewater [10], removal of heavy metals from aqueous solutions [11], separation of fine particles [12,13], removal of methyl orange and methylene blue dye from water [14], removal of hazardous oily waste from a soil matrix [15], fractionation of a red grape marc extract [16], extraction of erythromycin [17], removal of pyrene [18] etc. The microbubble indicates a large interfacial area per unit volume which increases the adsorption of molecules on its surface. Its holdup enhances the probability of particles to attach on its surface [10].

Conventional flotation has been incorporated with microbubbles for application in the recovery of fine mineral particles (below  $13\ \mu\text{m}$ ) and in solid/liquid separation to remove pollutants [7]. Surface forces and electrostatic forces are two main interactive forces present in dispersed microbubbles which directs their application in various fields [18].

Microbubbles formed by use of ionic surfactants contribute electrostatic interaction to the stability of its dispersion, which is governed by the charged or polar groups in the surfactant molecules at the gas-liquid interface [19]. Microbubbles in non-ionic surfactants are governed by steric forces which also play an important role in stabilizing their dispersions [20]. The literature reports stability of microbubbles depends on many variables [3]. Lamentably, there is a knowledge gap on hydrodynamic characteristics of microbubbles in various liquids in presence and absence of fine solid particles and its stability. Modifying the surface tension of liquid can not only shift the bubble size distribution, it also influences the stability. But there are no significant studies available in literature that can correlate the stability of microbubble with concentration of surfactant in presence of micro and nano particles. Also there is no such quantitative research available in literature that can demonstrate the effect of particle concentration and other effects like temperature, pH on the stability of microbubble. Based on this scope of study, the objective of the present work is to study the effects of micro-nano particle concentration, temperature, pH and surfactants on the stability of microbubble, its holdup and size. The present work is also aimed to develop an empirical model to interpret the effect of variables to them.

## 2. Experimental

### 2.1. Materials

Sodium dodecyl sulfate (Sodium Lauryl sulfate) purchased from MERCK, nanoclay a surface modifier containing 25–30 wt% trimethyl stearyl ammonium purchased from ALDRICH chemistry and a bio

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**Table 1**  
Properties of the micro and nano particles.

Type of mineral	Copper oxide	Aluminum oxide (nanosize)	Zinc oxide	Aluminum oxide
Density (g/cm <sup>3</sup> )	6.31	3.95	5.61 g/cm <sup>3</sup> (20 °C)	3.94 g/cm <sup>3</sup> (20 °C)
Size (μm)	<50 nm (TEM)	13 nm (TEM)	4.47	19.6
Molar mass (g/mol)	79.55	101.96	81.37	101.96
Boiling point	–	2980 °C	2360 °C	2980 °C (1013 hPa)
pH value	–	–	7 (50 g/l, H <sub>2</sub> O, 20 °C) (slurry)	6.8–7.8 (10% suspension)
Melting point	–	2050 °C	1975 °C	2050 °C
Surface area (m <sup>2</sup> /g)	29 m <sup>2</sup> /g	85–115 m <sup>2</sup> /g (BET)		

surfactant saponin (non-ionic) purchased from SIGMA-Aldrich were used for experimentation. The various properties of materials used in the present study are shown in Tables 1–3.

## 2.2. Methodology

### 2.2.1. Generation of ionic microbubbles

A mixture assembled with a high-speed stirrer (max 20,000 rpm) (Butterfly Matchless 750 W Mixer) was used to generate ionic microbubbles in surfactant solution according to method given by Sebba [2]. The surfactant solutions were prepared using SDS, CTAB and saponin in 1 l of millipore water with and without particles. The solutions were stirred at a speed of 20,000 rpm to generate ionic microbubbles at room temperature, 25 ± 1 °C. The schematic diagram of the experimental setup is shown in Fig. 1.

### 2.2.2. Measurement of zeta potential

The zeta potential was measured by Zetasizer Nano ZS (Delsa Nano C, Japan, Se. No. 131410). pH of the suspensions was adjusted by using hydrochloric acid (0.1 N) and sodium hydroxide (0.1 N). Before zeta potential measurements all samples were sonicated (PCI Analytics Pvt. Ltd., Model No.: 3.5L100H/DTC) for 15 min. As the absolute value of zeta potential is larger, many colloidal particles show good dispersity as the electrostatic repulsion becomes stronger. However, as the zeta potential registers close to zero, the particles become unstable and are likely to aggregate which decreases the dispersity. Addition of surfactant significantly influences the zeta potential of particle as shown in Fig. 2. The zeta potential of ZnO particle is approximately –10 mV. Addition of SDS increases the negative charge on the ZnO surface. It does not create any significant attraction between the ionic microbubbles and the ZnO particles. This may increase the dispersity phenomena in the suspension. The zeta potential of Al<sub>2</sub>O<sub>3</sub> in water is approximately –3.6 mV which is low in magnitude as compared to ZnO.

### 2.2.3. Estimation of degree of stability of ionic microbubble

The degree of stability of colloidal microbubbles was estimated by measuring the drained volume of liquid with respect to time as per method proposed by Longe [21]. The schematic diagram of the stability analysis is shown in the Fig. 3 (a). After preparation of charged microbubble, it is transferred to a 50 ml measuring cylinder which is kept in a constant temperature bath. To maintain the constant temperature, a circulating water bath (ANM Industries Pvt. Ltd., Thane, Model No.: WB 2000) is used. A video camera (Nikon, Coolpix L340) is used to record the drainage of liquid for different concentration with various operating parameters. Each experiment is carried out three to five times, and the average value was calculated. The measurement is

**Table 2**  
Properties of surfactants.

Surfactant	SDS	CTAB	Saponin
Molecular formula	C <sub>12</sub> H <sub>25</sub> OSO <sub>2</sub> ONa	C <sub>19</sub> H <sub>42</sub> BrN	C <sub>45</sub> H <sub>73</sub> NO <sub>15</sub>
Molar Mass (g/mol)	288.37	364.46	868.06
Appearance	White powder	White powder	White crystalline solid
Melting point (°C)	204–207 °C	248–251 °C	271–273 °C
Solubility in water	2 g/l at 20 °C	36.4 g/l at 20 °C	74 g/l at 30 °C

done immediately just after the preparation of ionic microbubbles to avoid error. Drainage of liquid as a function of time is plotted for different parameters. The plotted curve was used to estimate the time taken to drain half of the liquid from the colloidal microbubbles suspension as shown in Fig. 3 (b). The ratio of half-life to the total time of liquid drainage is referred to degree of stability.

### 2.2.4. Determination of microbubble gas holdup

Knowledge of gas holdup is necessary for estimation of the interfacial area, which influence the efficacy to microbubble aided fine particle separation. The gas holdup depends on the gas flow rate, physico-chemical properties of liquid and gas phase, operating temperature, and pressure [22]. The gas holdup also depends on the number of bubbles generated, average bubble size and the bubble rise velocity [23]. The various methods to estimate gas phase fraction are phase isolation, conductivity, pressure drop, gamma ray attenuation, electrical resistant tomography (ERT) and dynamic gas disengagement (DGD). In the present work the phase isolation method is used. Microbubbles solution is filled in the transparent glass cylinder and the camera is used to record the change in the height of the clean liquid with time. After certain time the bubbles at the top starts disappearing as they are in contact with atmosphere directly. Few snap shots of microbubbles height or volume taken at different times are shown in Fig. 4.

The method proposed by Amiri and Woodburn [24] is used to determine the size of microbubbles based on the rise velocity of the microbubbles as a function of gas holdup in the ionic microbubble dispersion. The change of gas holdup ( $\varepsilon_g$ ) with time in the ionic microbubbles solution can be expressed as

$$\varepsilon_{g,t} = \frac{V_{g,t}}{V_t} \quad (1)$$

where  $V_{g,t}$  and  $V_t$  are the volume of gas in the microbubbles solution and volume of microbubbles solution at time  $t$ , respectively. The volume of gas at time  $t$  is

$$V_{g,t} = V_t - V_{d,t} \quad (2)$$

where  $V_{d,t}$  is the volume of liquid drained at time  $t$ . Therefore, from Eqs. (1) and (2), one can write:

$$\varepsilon_{g,t} = \frac{h_t - h_{d,t}}{h_t} \quad (3)$$

At  $t = 0$  of drainage,  $h_t = h_o$ ,  $h_{d,t} = h_{d,o}$  and  $\varepsilon_{g,t} = \varepsilon_{g,o}$ . Hence one can write

$$\varepsilon_{g,o} = \varepsilon_o = \frac{h_o - h_{d,o}}{h_o} \quad (4)$$

**Table 3**  
Physical properties of glycerol.

Properties	Value
Density	1.26 g/cm <sup>3</sup> (20 °C)
pH value	5 (100 g/l, H <sub>2</sub> O, 20 °C)
Vapour pressure	<0.001 hPa (20 °C)
Molar mass	92.1 g/mol

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