



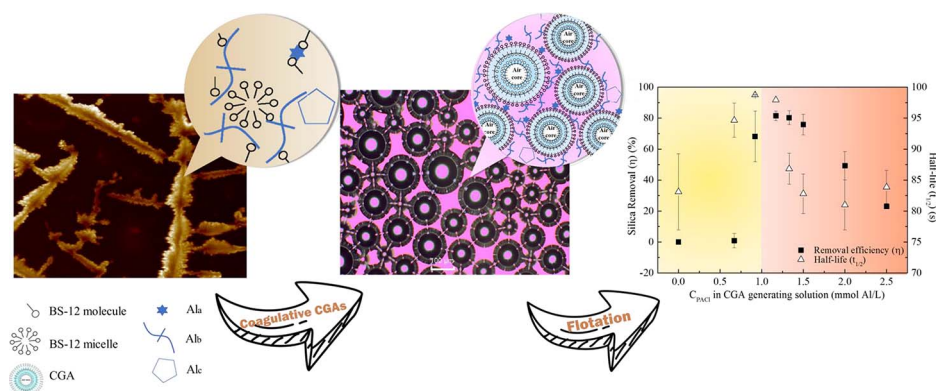
# Coagulative colloidal gas aphrons generated from polyaluminum chloride (PACl)/dodecyl dimethyl betaine (BS-12) solution: Interfacial characteristics and flotation potential



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## GRAPHICAL ABSTRACT



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## ABSTRACT

Bubble surface functionalization is promising in flotation in terms of stronger bubble-particle interaction, less chemical consumption, more flexible and pollutant-oriented. In this study, the coagulant-modified microbubbles were generated via colloidal gas aphrons (CGAs) technique; the specific bubble characteristics and flotation efficiency in removing nanosilica were investigated. The coagulative CGAs were generated from a mixed system consisting of zwitterionic surfactant (dodecyl dimethyl betaine, BS-12) and inorganic polymer coagulants (polyaluminium chloride, PACl). Results denoted that the bubble generation capability of the mixed solution was well maintained; meanwhile, the observed large aggregates indicated the possible interaction between PACl and BS-12. The generated PACl-modified CGAs exhibited a typical CGA morphology with the averaged diameter of 46–100  $\mu\text{m}$ . It should be noted that the stability of these positively surface-charged bubbles was strongly influenced by the PACl concentration. The half-life reached  $\sim 100$  s at the optimum PACl concentration range of 0.92–1.17 mmol Al/L. The interaction mechanisms between PACl and BS-12 as well as CGAs were speculated: one the one hand, the monomer Al species might interact with the BS-12 molecules and coat on the CGA surface; on the other hand, the polymerized Al species probably acted as bridge/net in which the BS-12 micelles and

**Abbreviations:** CGA, colloidal gas aphrons; PACl, polyaluminium chloride; BS-12, dodecyl dimethyl betaine; CMP, chemical mechanical polishing; CMC, critical micellar concentration; (BS-12)-CGA, colloidal gas aphrons generated from pure BS-12 solution; IEP, isoelectric point; SDS, sodium dodecyl sulfate

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CGAs were embedded. Particularly, the coagulative CGA-involved flotation could remove approximately 81% of nanosilica, and a reinforced flotation process was thus achieved.

## 1. Introduction

Microbubbles-involved flotation technique has gained an increasing interest in water treatment [1,2]. Featured as high interfacial area, great bubble density [3] and long reservation time [4], the bubbles of 100–700  $\mu\text{m}$  in diameter [5–7] are capable of improving the flotation performance through enhancing the effective bubble-particle collision [1,4,8]. Thus, they are especially preferred in the separation of ultrafine particles [9], colloids and algae from water and wastewater [8]. In traditional flotation techniques, an individual step of coagulation is inevitable, which enlarges the floc size via changing the physiochemical properties of particle surface and hence reinforces the attachment between bubbles and particles. However, it requires a significant amount of coagulants, probably giving rise to a secondary pollution to the aqueous environment as well as a large volume of sludge [10]. Given this context, the microbubbles surface functionalization with coagulant is proposed in this study: good bubbles are produced using surfactant and meanwhile strong adherence is achieved via the surface modification with coagulant.

Colloidal gas aphrons (CGAs) are a system of colloidal microbubbles (10–100  $\mu\text{m}$  in diameter) [11], which can be fabricated by intensively stirring the surfactant solution (> 4000 rpm) [12]. According to Sebba, the CGA is postulated to contain a gaseous core surrounded by multi-layers of surfactant shells [12]. The striking characteristics, such as extraordinarily high stability and controllable surface properties, endow CGAs with great advantages in bubble surface modification and flotation separation. The chemicals used for CGA production are determined by the treating pollutants. Through adapting the bubble generation solution with complex systems, the colloid and surface properties of bubbles can be tailored and the flotation performance will then be improved. One of the most attractive surfactant types for bubble modification is zwitterionic surfactant. With positive and negative hydrophilic groups as well as hydrophobic carbon chains, the zwitterionic surfactants may interact with the inorganic and organic matter by electrostatically and hydrophobic attractions [13]. Thereby, the CGA surface modification can be realized by combining zwitterionic surfactants with modifiers (coagulants herein). Moreover, it should be noted that the environmental impact of surfactants and coagulants has been of great concern owing to their toxicity [14–16]. On the one hand, those toxic functional groups (i.e. benzene ring) and elements (i.e. bromine) should be avoided, and the environmentally friendly reagents are needed for the CGA creation; on the other hand, the dosage of chemicals is expected to be low by adapting the CGA generation solution.

Previous research has been dedicated to exploring the most appropriate chemical characters for bubble modification, including the type of metal coagulants and cationic surfactants, the length of carbon chain, and the molecular weight of polymers [17–20]. Three criteria have been drawn from the successful generation and application of positively charged bubbles: 1) the positively charged region for the electrostatic attraction between bubbles and negatively charged algae; 2) the hydrophobic component to encourage adsorbance at the bubble surface; 3) the high molecular weight to increase the swept volume and to enlarge the enmeshment area [18–20]. To meet the above mentioned requirements, Rita K. Henderson et al. synthesized the hydrophobically associating cationic polymers to modify the surface of air bubbles, and around 99% of algae cells were removed by the improved dissolved air flotation technique [17]. However, to the authors' knowledge, no reports have specifically investigated the characteristics of the surface-modified bubbles, which is one of the significant targets of the present study.

The fast development of nanotechnology brings about a growingly

environmental problem—nanoparticles and colloids pollution in water and wastewater [21,22]. Due to the high dispersion stability of nanoparticles and colloids in water, the related coagulation-flotation separation demands a great amount of coagulant and causes a bulky sludge [23]. Moreover, the performance of flotation is limited by the low bubble-particle collision efficiency caused by Brownian diffusion [24]. A promising solution may be surface-modifying the microbubbles, such as CGAs, with coagulants, through which the hydrophobic interaction and electrostatic attraction between bubble surfaces and nanoparticles can be strengthened. Consequently, an enhanced flotation technique for effective separation of nanoparticles and colloids can be developed.

In this study, a novel coagulant surface-functionalized microbubbles system was created from the inorganic polymer/surfactant mixed system, named coagulative CGAs. The highly efficient coagulant, polyaluminum chloride (PACl), and the zwitterionic surfactant, dodecyl dimethyl betaine (BS-12), were employed for the generation of coagulative CGAs. The microbubbles were investigated with respect to the characteristics generating suspension and bubble features. Possible formation mechanisms of the coagulative CGAs were speculated based on the experimental results and analysis. Furthermore, the coagulative CGAs were adopted in the flotation separation of nanosilica which had widely existed in the industrial effluents, such as CMP (Chemical mechanical polishing) wastewater [25].

## 2. Materials and methods

The reagents used in this study were analytical grade products except those used for instrumental analysis. All the solutions were prepared with deionized water. Ultrapure water with a resistivity of 18 M $\Omega$  cm was used in PACl preparation.

### 2.1. Preparation and characterization of PACl/(BS-12) mixed solution

#### 2.1.1. Surfactant solution

Dodecyl dimethyl betaine (BS-12,  $\text{C}_{15}\text{H}_{31}\text{N}^+(\text{CH}_3)_2\text{CH}_2\text{COO}^-$ ) (Sinopharm Chemical Reagent Co., Ltd.) was employed in the current study. BS-12 contains an anionic functional group with carboxyl terminal, a cationic functional group with quaternary-N, and a hydrocarbon chain with 15 carbons. Being accepted as hazard-free, it finds a wide application in the industrial manufacture of detergents, dispersants and emulsifiers [26,27]. As a mild zwitterionic surfactant, BS-12 is of excellent stability under acidic or basic conditions [27]. The critical micelle concentration (CMC) of BS-12 was determined to be 0.29 mmol/L in the present work by measuring the surface tension of BS-12 solutions within a series of concentrations at 25  $^\circ\text{C}$  (Fig. S1 in Supporting Information (SI)) [28]. The CGAs generated from BS-12 solution are briefly named as (BS-12)-CGAs in the following discussion.

#### 2.1.2. Preparation of PACl

PACl solution with a basicity of 2.0 was prepared by the base titration method in laboratory at 25  $^\circ\text{C}$  [29]. Prior to the preparation of PACl2.0, 0.4 mol Al/L  $\text{AlCl}_3$  as well as 1 mol/L  $\text{NaHCO}_3$  was prepared as stock solution. The  $\text{NaHCO}_3$  solution was titrated into the  $\text{AlCl}_3$  solution at 0.5 mL/min by peristaltic pump (BT600-2J, Longe CO., China), during which the  $\text{AlCl}_3$  solution was rapidly stirred. The PACl2.0 solution with the concentration of 0.2 mol Al/L was finally obtained after aging for 24 h. The pH of PACl2.0 was measured to be 4.0 with pH meter (PHC101, Hach, USA).

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