

UTILIZING GAS BUBBLE FLOTATION IN CAPTURING Al^{3+} DURING WATER TREATMENT

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TÓM TẮT

KHỬ ION NHÔM TRONG XỬ LÝ NƯỚC BẰNG PHƯƠNG PHÁP TUYỂN NỐI BỌT KHÍ

Hệ thống nước ngầm chứa một lượng lớn nhôm từ vỏ Trái Đất. Nguồn nước ngầm cần được xử lý đúng quy trình để đảm bảo an toàn phục vụ cho hộ gia đình. Nồng độ nhôm trong mạch nước ngầm có thể tăng phụ thuộc vào sự có mặt của chất làm đồng như phèn kali các hợp chất muối của nhôm. Chúng tôi nghiên cứu tính khả thi của việc ứng dụng phương pháp kết tủa nổi trong quá trình loại bỏ ion nhôm khỏi nước xử lý phèn. Natri dodecyl sulfat được sử dụng như chất điện hoạt để tăng lượng nhôm thu được. Mẫu nước thải được xử lý bằng phương pháp tuyển nổi bọt khí với 0.6 mM natri dodecyl sulfat cho thấy lượng nhôm giảm 98% so với mẫu nước trước khi xử lý.

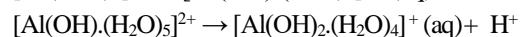
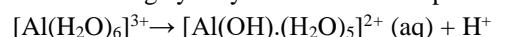
Từ khóa: Tuyển nổi; Khử phèn nhôm; Xử lý nước thải di động; Chuẩn độ ngược.

1. INTRODUCTION

Aluminum is the third most abundant element and the most common metal in the Earth's crust. It is predominately obtained as a complexation with other elements, including oxygen (oxides), silica (silicates) and fluorine (Greenwood 1997). The study on chemistry of aluminum in surface water is quite complex due to its various transformations. Aluminum has amphoteric property which allows the metal to alter its existing forms in accordance with the change in pH; when pH value ranges between 7.0 -7.5 (Gensemer and Playle 1999, Srinivasan, et al. 1999) or 6 – 8 (Bashkin 2007), aluminum exists as insoluble amorphous $\text{Al}(\text{OH})_3$. In acidic environment, aluminum appear in its soluble form of $\text{Al}(\text{OH})^{2+}$ and $\text{Al}(\text{OH})_2^{+}$. Another soluble form of aluminum, $\text{Al}(\text{OH})_4^-$ was recorded in the reverse of the mentioned conditions. Thus, the

fate of aluminum in the environment is strongly influenced by the variation of pH.

The following hydrolytic reactions are expected:



The end product is a precipitate of a colloidal hydroxide of aluminum, $[\text{Al}(\text{OH})_3.(\text{H}_2\text{O})_3] (\text{s})$ with a positive charge at a neutral pH (Saitoh, Yamaguchi et al. 2011).

Typically, aluminum in rocks and minerals are discharged to the water bodies through chemical weathering processes such as acid rain (Srinivasan, et al. 1999). The burning of fossil fuels emits sulfur and nitrogen oxides. Chemical reaction between sulfur and nitrogen oxides produces sulfuric acids and nitric acids. The mixture of such acids with water and other materials results in acid rain. Upon reaching the ground, acid rain reacts with mineral

particles of rocks to leach aluminum into water sources, raising the concentration of aluminum in natural water bodies. Human-derived sources of aluminum originates from industrial processes (i.e., mining and smelting). Solid wastes containing aluminum from agricultural activities such as fertilizers are discharged directly into the groundwater system, hence the elevated aluminum concentration (Srinivasan, et al. 1999).

Human exposure to aluminum has been generally considered unsafe. (Yase 1991, Safety 1997), (Inan-Eroglu and Ayaz 2018). According to the United States Environmental Protection Agency (EPA), aluminum is considered to affect human health adversely with concentrations higher than 0.2 mg/L.

One of the common aluminum salts, potassium alum, is employed as coagulants in water treatments to remove unwanted dirt and reduce turbidity. The alum-treated water will undergo filtration to produce safe drinking water. In particular, the aluminum salts added to untreated water will bind to the oppositely charged suspended particles, resulting in larger floc of particles that settle to the bottom of the water container. The supernatant is then filtered, either directly through a medium filter, a micro-filter or an ultrafiltration membrane.

The gelatinous form of aluminum hydroxide has paved way to the removal of aluminum ion by gas bubble flotation. In contrast to filtration, gas bubble flotation offers high quality outcome of the treated water with significantly reduced amount of sludge (Ferguson, Logsdon et al. 1995). This method was utilized prior to the straightforward operation, low cost of maintenance, economical outlay and energy conserving.

The principle of precipitate flotation is the use of air bubbles as a means of carrier to transport the precipitate onto the liquid surface (Capponi 2006). The introduction of gas bubbles coupled with the use of flotation reagents enhances the formation of agglomerates. The density of these floc is lower than water, hence the affinity to rise to surface where they will be collected.

In this study, sodium dodecyl sulfate (SDS) is utilized as a collector in the precipitate flotation process (Johnson and Tyrode 2005). SDS is commonly found in shampoos, cosmetics, and household cleaning products (Cowan-Ellsberry, Belanger et al. 2014, Committee for Human Medicinal Products (CHMP) 2015). A study conducted by Belanger classified SDS as an 'environmentally friendly' substance due to its rapid biodegradability and low bioaccumulation attributes (Belanger, Lee et al. 2004, Shipley 2014).

Toxicity of a product is examined on the basis of a whole combination of ingredients and not dependent on a specific substance. Hence, formulations containing insignificant concentrations of SDS are not considered toxicants. This could be the reason why the U.S Food and Drug Administration (FDA) lists SDS as a food additive (CFR - Code of Federal Regulations Title 21).

SDS was used in this study as a negatively charged surface active agent that attracts the positively charged aluminum ions. The high transfer energy of SDS facilitates its strong adsorption at the air/water interface of the bubbles. The use of SDS in combination with fine gas bubbles is potential in the elimination of excess aluminum ions in the water. Based on the aluminum's colloidal characteristic and its pH-dependent transformation, the removal of aluminum is feasible. The aluminum ion elimination approach proposed in this study is advantageous over the other existing methods using alum salts as it possesses high efficiency, relatively simple instrumentation and most importantly, it requires no filtering and thus no clogging will occur during the process.

2. MATERIALS AND METHODS

2.1. Materials

2.1.1. Chemicals and reagents

Potassium aluminum sulfate, Sodium hydroxide, Eriochrome Black T, Zinc sulfate, Disodium ethylenediaminetetraacetic acid and Sodium dodecyl sulfate were all purchased from Schalaur and used as received.

2.1.2. Instruments

Laboratory-scale model of flotation system consists of a container and a portable air pump, burette and stirrer.

2.2. Methods

2.2.1. Stock preparation

Potassium aluminum sulfate (Potassium alum) at 0.1M, 20mM sodium dodecyl sulfate (SDS) and 3.0M sodium hydroxide were used as stock solutions. The stocks were further diluted for experimental use. Disodium ethylenediaminetetraacetic acid (EDTA) 0.01M and Zinc sulfate 0.01M were prepared for the back titration with 0.5% Eriochrome Black T as an indicator.

Five standard solutions were prepared as followed. First, 47.4 grams of aluminum potassium sulfate ($\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$) was added to one liter of hot distilled water to obtain 0.1M Potassium alum stock solution. Sodium hydroxide 3M was attained by mixing 60 grams of powder into 0.5 liters of distilled water; this solution had to be prepared in ice cold environment since the reaction is highly exothermic and hence generates excessive heat. Sodium dodecyl sulfate (SDS) at 5.36 grams was dissolved in one liter of distilled water to produce 20mM of SDS solution. The stock solutions of 0.01M disodium ethylenediaminetetraacetic acid (EDTA) and 0.01M zinc sulfate were carefully prepared for back titration by mixing 3.72 grams and 2.9 grams of the two powders in one liter of distilled water each. An indicator solution was prepared by weighing 0.5 grams of Eriochrome Black T in 100 mL consisting of 80 mL of 95% Ethanol and 20 mL distilled water.

2.2.2. Standard curve construction

The reagents used were potassium alum 1 g/L, 0.01M disodium ethylenediamine tetra acetic acid (EDTA), 0.01M zinc sulfate, NH_3 solution, and 0.05% Eriochrome Black T.

One gram of potassium alum was added to one liter of distilled water to obtain the stock solution with a dilution factor of 1:2. Five 250mL Erlenmeyer flasks labelled 1:2, 1:4, 1:8, 1:16, 1:32 were filled with 40 mL distilled

water measured by a 50-mL cylinder. Serial dilution was carried out for five flasks.

The resulting diluted solutions along with the stock were titrated against zinc sulfate. The volume of zinc sulfate for each trial were recorded.

One gram of potassium alum was added to one liter of distilled water to obtain the stock solution with a dilution factor of 1:2. Five 250mL Erlenmeyer flasks labelled 1:2, 1:4, 1:8, 1:16, 1:32 were filled with 40 mL distilled water measured by a 50-mL cylinder. Serial dilution was carried out for five flasks. The resulting diluted solutions along with the stock were titrated against zinc sulfate. The volume of zinc sulfate for each trial were recorded.

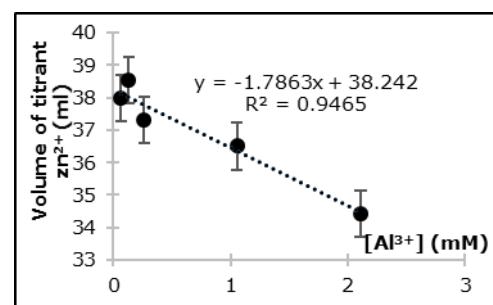


Figure 1. The standard curve between the zinc sulfate volume and the aluminum concentration

2.2.3. Precipitate flotation

The precipitate flotation process included 1 mM potassium alum solution and 3 mM sodium hydroxide. Different concentration of sodium dodecyl sulfate at varied concentrations were added to the mixture to obtain the concentration for efficient flotation.

The gas flotation was performed in a plastic container with the height of 20 cm and 15 cm in diameter. A nozzle connecting to a portable air pump was placed at the bottom of the container to generate fine air bubbles at a rate of 4.2 liters per minute. A magnetic stirrer was required throughout the flotation. Each set of experiment required two liters of distilled water; the reagents were prepared as described in Table 1. 20.2 mL of aluminum 0.1 mM and 2 mL of NaOH 3M were consecutively pipetted into two-liters of distilled water in a plastic container. Depending on the concentration, different volumes of SDS 20

mM were added according to **Table 1**. The pH of the solution was closely monitored to ensure no deviation from the value of pH 7.0. The container was then placed on a magnetic stirrer at the constant speed of 900 rpm with simultaneous introduction of constant air flow for 20 minutes. The solution containing aluminum was collected at four specific time periods: before floatation, after five-minutes, ten-minutes and twenty-minutes of flotation by withdrawing 25 mL solution from the bottom of the container using a 10-mL glass pipette.

Table 1. Sample preparation components

Al ³⁺ Stock 0.1M (mL)	NaOH 3M (mL)	SDS 20 mM (mL)	SDS concentration observed (mM)
20.2	2	105.3	1
20.2	2	94.2	0.9
20.2	2	83.3	0.8
20.2	2	72.5	0.7
20.2	2	61.9	0.6
20.2	2	51.3	0.5
20.2	2	40.8	0.4
20.2	2	30.5	0.3
20.2	2	20.2	0.2
20.2	2	10.1	0.1

2.2.4. Evaluation of aqueous medium

2.2.4.1. Determination of aluminum ions in aqueous medium

Standard 0.01M Ethylenediaminetetra-acetic acid disodium salt (EDTA) was used as the chelating agent that bind to the aluminum ions to form complexes. 0.01M Zinc sulfate solution was used as the titrant with 0.05% of Eriochrome Black T (EBT) as an indicator. The use of ammonia solution was to maintain the pH of the solution to be around 10.

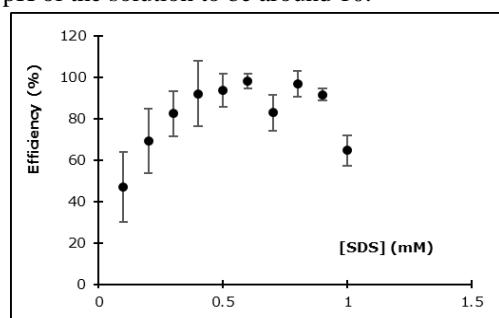
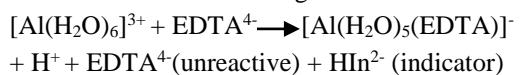
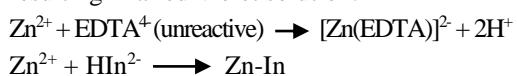


Figure 2. The flotation efficiency after 5 minutes

Back titration and surface tension measurements were performed to detect and measure the remaining aluminum ion and the abundance of SDS in the treated water. Back titration was used to quantify the concentration of aluminum residues since aluminum ion blocks an indicator, causing no color change observed at the endpoint. A back titration involves the reaction between a sample of unknown concentration and an excess of reagent with a known concentration. The first reagent is then titrated against another reagent. In this experiment, aluminum from an unknown solution reacted with an excess amount of standard EDTA, forming a ligand. It has been widely accepted that EDTA has a high affinity for metal ions, compared to the EBT indicator; thus, all aluminum ions will completely bind to the EDTA prior to the addition of indicator. Since the amount of EDTA is excessive, free EDTA and the indicator dianion HIn²⁻ will form when the pH of the solution is in the range of 7 - 11:



This resulting solution will react with the zinc solution. Zinc(II) solution reacts with free EDTA to form another ligand. The reaction between zinc and indicator will also take place, resulting in a red-violet solution:



The complex of aluminum and EDTA ($[\text{Al}(\text{H}_2\text{O})_5(\text{EDTA})]^-$) is still present in the solution. The metals complex with EDTA following the ratio of 1:1.

The potassium alum samples obtained at the four different time periods, were mixed with 40 mL of disodium EDTA in a 250-mL Erlenmeyer for two minutes. The solutions were then heated at 60°C in a water bath for three minutes to ensure complete complexation of aluminum. The solution was then cooled down to room temperature and an appropriate amount of ammonia stock solution (~25%) was added to maintain that pH value fluctuated around 10. The addition of 250 μL of

Eriochrome black T gave the solution a blue color. The burette was filled with 0.01M zinc sulfate as a titrant and this was used to titrate against the mixed solution containing excess disodium EDTA and potassium alum. The endpoint was reached once the solution turned violet, the volume of the titrant was recorded.

2.2.4.2. Assessment of SDS in the aqueous medium

Hydrogen bonds between water molecules at the surface of water bodies create a strong network of water molecules. The strength of this network at the air-liquid interface, is reflected by the surface tension of that interface. One distinctive characteristics of SDS is the ability to reduce the surface tension (Esteves 2016). Therefore, the measurement of surface tension can be used to examine the excessive amount of SDS in water after flotation. In our experiment, the Wilhelmy plate method was applied as a method to calculate the surface tension of four samples after the flotation process.

3. RESULTS

Table 2. The flotation efficiency after 5, 10, and 20 minutes.

	Surface tension (mN/m)	[SDS] (μ M)
Before	58.01	35.28
5 mins	62.19	26.09
10 mins	63.42	24.22
20 mins	67.05	17.29

Figure 1 illustrates the standard curve when plotting the remaining aluminum ion concentration against the volume of titrant Zn^{2+} . The concentration of aluminum ion is inversely proportional to the volume of zinc sulfate. The amount of aluminum ions being removed is dependent on the concentration of SDS used (**Figure 2**) and the floating time (**Table 2**). Aluminum ions are best eliminated from the water bodies by flotation at SDS concentration of 0.6mM. According to Table 3, the highest amount of aluminum removed after five minutes, with the use of 0.6 mM

SDS, accounted for approximately 98.14%. When 0.8 mM and 0.9 mM SDS were applied, roughly 96.8% and 91.5% of aluminum was respectively eliminated from the solution. However, there was a decrease in the flotation efficiency when using higher concentrations SDS, as only 64.6% of aluminum was eliminated. This suggests that an excess in SDS is likely to reduce the floatation efficiency, not meeting expectations. Similarly, after five minutes, the extent of aluminum ions eliminated from the solution declines, as smaller concentrations of SDS were used. To summarize, a downward trend in the aluminum removal was present in parallel to the smaller SDS concentration. Beginning at SDS 0.5 mM, approximately 92.5% of aluminum was removed. There was roughly a 91.6%, 84%, 62.5% and 47.5% of aluminum removal when the SDS concentrations 0.4mM, 0.3 mM, 0.2 mM, and 0.1 mM were respectively used. No aluminum remained after 10 minutes when the concentrations of SDS tested were in the range of 0.4 mM to 1 mM. For SDS concentration of 0.3 mM, 0.2 mM, and 0.1 mM, there were increases in the removal of aluminum after the entire twenty-minute flotation duration.

Table 3. Surface tension of the solution after further flotation process. The continual process was performed with the treated solution at SDS concentration of 0.6mM

[SDS] (mM)	Efficiency (%)		
	5 Mins	10 Mins	20 Mins
1	64.61	100	100
0.9	91.57	100	100
0.8	96.83	100	100
0.7	82.81	100	100
0.6	98.14	100	100
0.5	91.94	100	100
0.4	90.72	100	100
0.3	82.35	94.82	98.06
0.2	69.33	88.81	93.44
0.1	46.86	83.00	89.33

Table 3 portrays the correlation between surface tension of alum-treated water and the

concentration of SDS. In particular, an increase in surface tension of SDS is closely associated with decreasing SDS concentrations.

The surface tension of the alum-treated water measured before flotation and after 5, 10 and 20 minutes flotation show a steady increment whereas the corresponding SDS concentrations are halved. After 20 minutes flotation, the SDS concentration dropped from $35\text{ }\mu\text{M}$ to $17\text{ }\mu\text{M}$ ($\sim 2.8\%$ of the initial SDS concentration of 600 μM used to float Al^{3+}).

4.DISCUSION

In this study, positive colloidal of aluminum particles were produced by precipitating with SDS. The concentration of SDS used in the study is well below its critical micelle concentration value ($\sim 8.08\text{ mM}$) to prevent the formation of micelles (Fuguet, Ràfols et al. 2005). Below the CMC level, surfactants are adsorbed onto the bubble surface, resulting in a negatively charged surface that attract aluminum ion. These aluminum ion agglomerates are dragged onto the surface of the solution as bubbles rise.

Table 4. The calculation of aluminum concentration, based on the volume of zinc sulfate titrated, at four specific time periods with different SDS concentrations.

[SDS] (mM)	Volume of zinc sulfate (mL)				[Al ³⁺] (mM)			
	Before	After			Before	After		
		5 min	10 min	20 min		5 min	10 min	20 min
1	36.25 ± 0.49	37.55 ± 0.07	38.5 ± 0.28	39 ± 0.10	1.12 ± 0.28	0.36 ± 0.4	0	0
0.9	36.0 ± 0.28	38.05 ± 0.07	38.5 ± 0.14	38.75 ± 0.21	1.26 ± 0.15	0.11 ± 0.04	0	0
0.8	35.97 ± 0.15	38.23 ± 0.12	38.5 ± 0.26	38.57 ± 0.59	1.31 ± 0.08	0.024 ± 0.08	0	0
0.7	35.83 ± 0.83	37.87 ± 0.06	38.5 ± 0.26	38.68 ± 0.35	1.34 ± 0.47	0.21 ± 0.032	0	0
0.6	35.97 ± 0.25	38.17 ± 0.06	38.67 ± 0.29	38.8 ± 0.17	1.27 ± 0.14	0.042 ± 0.032	0	0
0.5	35.87 ± 0.32	38.10 ± 0.46	38.67 ± 0.23	38.73 ± 0.21	1.33 ± 0.18	0.098 ± 0.23	0	0
0.4	36.4 ± 0.10	38.05 ± 0.21	38.9 ± 0.14	38.95 ± 0.07	1.03 ± 0.10	0.11 ± 0.12	0	0
0.3	35.43 ± 0.40	37.80 ± 0.15	38.27 ± 0.40	38.57 ± 0.40	1.57 ± 0.23	0.27 ± 0.086	0	0
0.2	36.05 ± 0.35	37.60 ± 0.35	38 ± 0.10	38.1 ± 0.10	1.22 ± 0.20	0.39 ± 0.20	0.14 ± 0.01	0.08 ± 0.01
0.1	35.05 ± 0.07	36.55 ± 0.35	37.7 ± 0.14	37.9 ± 0.14	1.79 ± 0.04	0.95 ± 0.12	0.30 ± 0.08	0.19 ± 0.08

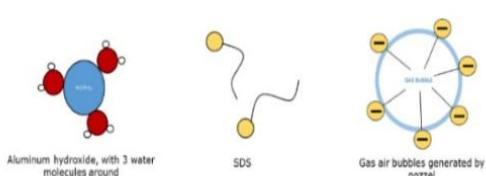


Figure 3. Interaction between SDS and fine gas bubbles

Different SDS concentrations were applied to examine the optimal concentration for flotation. The amount of aluminum ions

eliminated from the water bodies is projected to increase as a function of SDS concentration. As shown in **Figure 2**, after peaking at 0.6 mM , the floating efficiency fluctuates greatly at elevated SDS concentration. Varying the concentration of surfactants results in the change in adsorption of SDS molecules at the air/water interface. At low SDS concentration, little molecules are present as a binding site for aluminum ions. Raising the concentration of SDS leads to a deficiency of air bubbles as the site of adsorption for surfactants which

accounts for the unexpected fluctuation in floating efficiency. The flotation is thus ineffective at either enhanced or reduced concentration of surfactants. **Table 3** illustrates the floating efficiency at varied SDS concentration, with repeated collection of aluminum ions after the floating periods of 5, 10 and 20 minutes. High SDS concentrations (0.4 mM to 1 mM) demonstrate good aluminum ions removal capacity as no trace of aluminum ions were detected after floating the alum-treated water for 10 minutes at 1mM SDS. The removal of the comparable amount of aluminum ions takes longer time as lower SDS concentrations (0.1 mM to 0.3 mM) of SDS were used. After 20 minutes flotation, the efficiency of 89.33% and 98.06% were obtained at 0.1mM and 0.3mM, respectively. Increasing the concentration of SDS has been shown to enhance the flotation of aluminum ions. Despite the extended flotation time, high removal efficiency of 98.06% was obtained at such low concentration implies promising application of the technique in the field of water treatment.

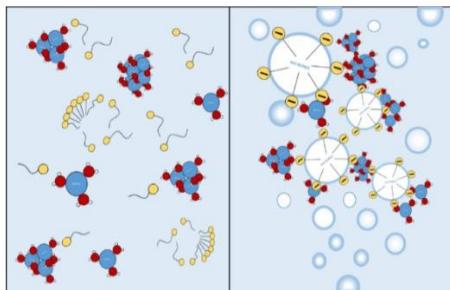


Figure 4. Illustrative comparison on the interaction of aluminum ion and SDS in the presence and absence of fine gas bubbles.

SDS particles are simultaneously removed with the aluminum ions during the flotation process, hence a low initial SDS concentration indicates minor presence of SDS particles in the water bodies after flotation. The resulting water source can be considered safe for household use prior to a reduced aluminum ions concentration and insignificant level of SDS. In addition to the low requirement of SDS in the process, the use of SDS itself poses as a great advantage due to the low production cost. Applying gas bubbles flotation in water treatment process invariably reduces capital investment on the costly filtering matrix in the current water treating plants.

Conventional water treatment plants generally require the elimination of toxic chemicals (i.e., Chlorine, Chlorine dioxide, Ozone, Monochloramine, etc.) before the treated water are qualified to enter the distribution system. The use of SDS in this study allows the negligence of the chemical removal step which could potentially shorten the throughput time of the process. Investment cost of the treating system might consequently be reduced.

The presence of heavy metals in drinking water has been reported to have deteriorate effect on human health (Rehman, Fatima et al. 2017); this calls for treatment method with high removal efficiency and simple operation. The gas bubble flotation system has proven to be a promising alternative. The adsorption of SDS onto the air/water interface followed by electrostatic interaction between the negatively charged bubbles and the positive metal ions can be applied to retrieve different metals from wastewater. The use of this method should be further extended to the removal of common metals in water sources such as magnesium, calcium, iron and heavy metals, namely arsenic, lead, mercury, cadmium. The bubble foam generated in the flotation process was expected to contain surfactant-coated aluminum hydroxide [surfactant-Al(OH)₃] as SDS molecules adsorb on the aluminum hydroxide precipitate due to the difference in electrical charge.

The SDS-coated aluminum hydroxide is highly hydrophobic. Such agglomerates can act as sites of attraction towards other hydrophobic organic pollutants (Saitoh, Yamaguchi et al. 2011).

5.CONCLUSION

The removal of aluminum ions by precipitate flotation, using SDS as the collector was investigated. The utilization of this method with appropriate combination of gas bubbles and flotation reagents is expected to promote a greater efficiency for aluminum ion removal from the aqueous media. Approximately 98% of aluminum ions were eliminated from the original solution after a brief 5 minutes flotation session. The longer the process, the larger the amount of aluminum ions removed, and the lesser SDS remained in the water, with only 17 μM after 20 minutes of floatation. The

minimized use of SDS offers the gas bubble flotation method great advantages over the conventional water treating method. The use of

SDS as surfactant to assist aluminum ions removal is economical due to the affordable production cost of the compound. Gas bubble flotation does not require the chemical elimination step in water treating process which is both time saving and environmental-friendly as no toxic chemical residues are produced during the process. The bubble foams containing aluminum hydroxide naturally coated with surfactant due to electrostatic interaction poses as a potential cleaning material. The hydrophobic-hydrophobic interaction between the coated aluminum hydroxide and other hydrophobic organic compounds had paved way to the application of the SDS- aluminum hydroxide complex in the removal of organic substances.

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