



Research Article

## Novel water treatment processes

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**Abstract.** The effect of a natural, biodegradable surfactant obtained using a novel and efficient chemical reaction between cysteine (a thiol-based amino acid) and an octanoyl (C8) compound, was investigated for its application to the ion flotation removal of low levels of different contaminant ions from aqueous solution. The synthesised amino acid-based single-chain surfactant shows a high water solubility and exhibits extensive foaming in a typical flotation chamber over a wide pH range. In a batch ion flotation process, this surfactant was able to remove 97–99% of the 5ppm of strontium, lanthanum, arsenic and different heavy metal ion levels present in contaminated water, in a simple, single-stage physiochemical process. Also, significant differences in ion binding selectivities could be used as the basis for the complete separation of some specific ions from mixed solutions, using the ion flotation process. Recycled water is an invaluable resource but it often also contains inorganic and organic nutrients, and chemical and biological substances, such as enteric microbial pathogens, which are often not monitored. This is a key inhibition to its reuse. The application of a novel CO<sub>2</sub> bubble column sterilization process is presented here and compared with other commonly used processes.

**Keywords.** ICP-MS, surfactant, ion flotation, heavy metals, cysteine, octanoyl chloride, water reuse, sterilization.

### 1. INTRODUCTION

The demand for developing new techniques to treat contaminated water containing hazardous ions, has grown significantly in recent years. Some of these ions like strontium<sup>1</sup> and lanthanum<sup>2</sup> or heavy-metal ions like arsenic<sup>3</sup>, mercury<sup>4</sup>, cadmium<sup>5</sup>, chromium<sup>6</sup>, lead<sup>7</sup> and copper<sup>8</sup> can accumulate in the body and cause serious damage to health due to their high affinity for binding with proteins in biological cells<sup>9</sup>. Some of these ions and their environmental effects are listed below.

Strontium (Sr) from radioactive wastewater is considered to be one of the most dangerous radionuclides to public health because of its high transferability, large consumption, high solubility and easy bioaccumulation. Radioactive strontium can be interchanged with calcium ions, which have major

functional activities to preserve bone and teeth as well as performing many essential enzymatic reactions in the body; therefore, this exchange can produce genetic damages to the living organisms and consequently can cause serious harm to the human health<sup>10</sup>. Among different strontium isotopes, the long-lived unstable one, with a half-time of 28.9 years is <sup>90</sup>Sr which emits beta particles that can cause severe damage to seawater creatures. Therefore, removing radioactive Sr from water is of great importance since, especially, the Fukushima Daiichi accident (Japan 2011), which led to disastrous seawater contamination by radioactive <sup>90</sup>Sr<sup>1</sup>.

Moreover, the Fukushima event became a motivation for researchers to focus on removing the released contaminant toxic elements, such as caesium, from leached soil and polluted water. It was shown that caesium (Cs<sup>+</sup>) can be removed from contaminated aqueous solutions by employing the flotation of copper-based Prussian blue nanoparticle analogues as a pH-sensitive surfactant<sup>11</sup>.

Contamination of groundwater and natural water with heavy-metal ions, especially arsenic, is a worldwide problem. It has become a major challenge for scientists and policy makers<sup>13,14</sup>. In addition, groundwater contamination with heavy metal ions causes contamination of agricultural products, like rice, which has been reported in several countries including China, Canada, the USA, and Bangladesh<sup>15</sup>, and Taiwan, Mexico, Argentina, Mexico, Poland, Hungary, Japan and India<sup>16</sup>, during recent years.

In addition, heavy-metal ions contaminate drinking water even at low concentrations, and hence they can be taken up via human consumption, which can ultimately lead to the risk of cancer, in the bladder, lung and also skin<sup>17-19</sup>.

To remove these ions in water treatment, adsorption can be used as a general process, especially when there is a strong affinity to bind hazardous ions in an efficient and simple alternative<sup>20</sup>. Using natural adsorbers which are green and non-toxic to the health like L-cysteine have been shown to be very promising in the treatment of water contaminated with these ions<sup>21</sup>.

Cysteine, used as the polar head group of a surfactant, could provide selective and efficient ion capture in an ion flotation removal process, in one step. Cysteine chelates with dissolved ions in aqueous solution, which can be effectively removed by rising bubbles into a foam on the surface of the aqueous solution.

In this study, the natural amino acid, L-cysteine was reacted with octanoyl chloride to form a single chain N-octanoyl cysteine surfactant (denoted N-octanoyl-cys).

Our motivation was to use the surfactant for removal by selective ion flotation of low levels of strontium, lanthanum and other heavy-metal ions from drinking water. This was achieved by the use of this new surfactant with cysteine head-groups which adsorbs at the water-air interface of a bubble. Rising bubbles within a flotation column can then offer a continuous supply of cysteine coated monolayers, where the surfactant and head-group will be relatively mobile, at room temperature. Collisions between the dissolved ionic species and the cysteine coated rising bubbles might be useful for selective and efficient ion capture and removal at a level of more than 97% for most of the discussed ions, in a one-step, continuous water treatment process. In addition, the surfactant is readily decomposed into natural products; therefore, this offers an environmentally acceptable process.

The World Health Organization (WHO) considers three reference pathogens in drinking water: for bacteria (*Campylobacter jejuni*); for protozoans (*Cryptosporidium*); and for viruses (rotavirus)<sup>22</sup>.

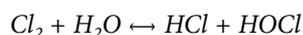
WHO guidelines for drinking-water quality compare thermal inactivation rates for different types of bacteria and viruses in hot liquids, concluding that temperatures above 60°C effectively inactivate both viruses and bacteria. When the temperature range lies between 60°C and 65°C, bacterial inactivation occurs faster than viral inactivation. Such studies show that, at a water temperature of 60°C, *E.coli* needs 300 seconds to reach a 1.5 log-unit reduction in viability; it takes 1800 seconds for viruses, such as enterovirus, echovirus 6, coxsackievirus B4 and coxsackievirus B5, to reach a 4 log-unit reduction (i.e. 99.99%)<sup>23</sup>.

### 1.1 Common water-sterilisation technologies

Collivignarelli et al.<sup>24</sup> found that UV irradiation and chemical treatments using chlorine, chlorine dioxide, peracetic acid or ozone were the most used technologies for wastewater disinfection.

**Chlorine** in the form of sodium hypochlorite, chloramines or chlorine gas is the most common disinfectant in water treatment, as it is cheap and relatively easy to handle<sup>25</sup>.

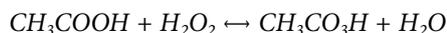
The bactericidal power of chlorine lies in its ability to affect the chemical structure of the bacterial enzymes through complex mechanisms. Hypochlorous acid and hydrochloric acid are produced when chlorine gas is added to water<sup>24</sup>:



**Chlorine dioxide** is a water-soluble bactericidal gas with high oxidizing power that affects protein synthesis and enzymes in bacteria. This gas is produced by the reaction<sup>26</sup>:

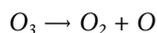


**Paracetic acid** is able to inactivate bacteria through the destruction of their membranes and enzymes due to the generation of reactive hydroxyl radicals and active oxygen. It is used as a chlorination alternative and is produced from the reaction<sup>26</sup>:



**Ozone** is a highly oxidizing agent produced by ozone generators when  $\text{O}_2$  is exposed to UV light or electric shocks. This produces individual oxygen atoms that combine with  $\text{O}_2$  generating  $\text{O}_3$ .

Ozone affects the cell wall of the pathogens, inactivating them. It is a very unstable gas that dissociates as<sup>26</sup>:



Water-disinfection UV lamps work in the spectrum 100-400 nm. There are four regions in the spectrum: UV (100-200 nm); UV-C (200-280 nm); UV-B (280-315 nm); and UV-A (315-400 nm). In water disinfection, UV-A and UV-C are the most effective; they are able to penetrate the pathogen membranes, inactivating them<sup>27</sup>.

However, all these water-disinfection technologies have limitations. For example, chlorine and chlorine dioxide react with organic compounds and form reactive chlorinated organic compounds that are hazardous to humans. In addition, chlorine needs at least 30 minutes contact time and is not able to eliminate *Cryptosporidium*. Chlorine dioxide has high management costs and is very unstable. Other disinfection methods such as ozone and UV irradiation are complex to operate and maintain. Rotavirus can be resistant to UV treatments and its efficiency is affected by the dissolved organic and inorganics in the wastewater, as well as its colour and turbidity<sup>28</sup>. Paracetic acid increases COD (Chemical Oxygen Demand) and BOD (Biochemical Oxygen Demand) due to the formation of acetic acid<sup>24</sup>.

Therefore, a major challenge exists to develop new, energy-efficient technologies to address these problems. One such candidate is the newly developed hot-bubble column evaporator (HBCE). This technique is able to inactivate pathogens for water reuse without the need for boiling and does not produce toxic side products.

## 2. MATERIALS AND METHODS

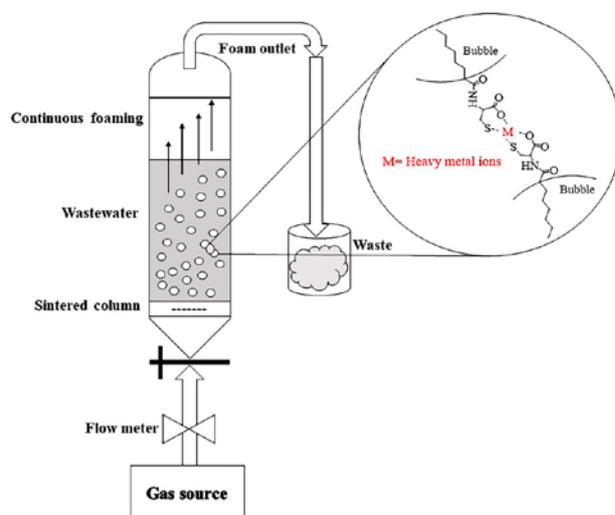
### 2.1 Ion flotation system

The solution obtained, after taking one sample for detecting the initial concentration of ions by ICP-MS (inductively coupled plasma mass spectrometry) analysis, from which removal rates will be calculated according to this, was then poured into a column (with 30cm height and 3cm diameter, as shown schematically in Figure 1) while a 1L/min flow of air gas was passed through a glass sinter, pore size no. 2, using an air pump (Hiblow HP40, Philippines). Two samples were taken after each of 30 and 60 minutes from around 2cm above the sinter, and the ion concentration of each sample was determined by ICP-MS analysis. The upper-outlet foam was also collected in a waste container using an outlet tube. A schematic diagram of the laboratory batch ion flotation column setup is shown in Figure 1.

It should be noted that in this protocol the surfactant, unlike the ions, is not fully depleted from the column during 60mins of bubbling time. That is, the surfactant concentration was approximately halved in the column during the bubbling experiments.

### 2.2 Hot bubble column evaporator

The HBCE process produces hot gas bubbles of 1-3 mm of diameter. The collision between these hot bubbles and the dispersed pathogens sterilizes the water<sup>29</sup>. At the same time, low solution temperatures (<50°C) are maintained, leading to good energy efficiency. The HBCE



**Figure 1.** Schematic diagram of the batch ion flotation apparatus. Reproduced from [21].

process requires less thermal energy than solution boiling because the heat capacity of the inlet hot dry air is much lower than that of water<sup>29</sup>. The hot, dry gas bubbles were produced continuously at the base of the bubble column using a glass sinter. The evaporation of water into the rising bubbles requires a substantial amount of latent heat and this produces only a modest temperature rise in the column solution. Experiments have been carried out using hot inlet gases at up to 275°C.

### 3. RESULTS AND DISCUSSION

#### 3.1 Ion flotation for heavy metal ion removal

The experimental results obtained using the Na<sup>+</sup> form of the N-octanoyl cys surfactant in an ion flotation process for the separation of a range of heavy metal ion contaminants from water, as individual ions and in appropriate ion mixtures, is summarized in the following Tables.

**Table 1.** Batch ion flotation results using air bubbles with 100mL solutions of 5mg/L each with single solutions of strontium, selenium and calcium ions using crystallised N-octanoyl-cys surfactant. The batch process started with a surfactant concentration,  $C_{\text{surfactant}}=0.01\text{M}$  and was maintained a pH=8.

Ion	C (mg/L) after 30min	Removal (%) after 30min	C (mg/L) after 60min	Removal (%) after 60min
Sr	0.03	99.4	0.009	99.8
Ca	0.72	85.6	0.13	97.3
Se	4.6	8	4.4	14

**Table 2.** Batch ion flotation results using air bubbles for 100mL solutions of 5mg/L calcium and strontium mixtures, using N-octanoyl-cys surfactant at an initial concentration,  $C_{\text{surfactant}}=0.01\text{M}$  and pH=8.

Mix Ions	C (mg/L) after 30min	Removal (%) after 30min	C (mg/L) after 60min	Removal (%) after 60min
Sr	2.14	57.2	0.13	97.4
Ca	3.39	32.2	1.3	73.8

These results indicate that, in mixed solutions, the favourable binding of strontium ions to the cysteine surfactant can depress the removal rate of calcium ions, and this effect may be related to the stronger hydration of the smaller calcium ion.

**Table 3.** Flotation results for 100mL mixed ion solutions of 5mg/L lanthanum and iron (initially) using crystallised N-octanoyl-cys surfactant with dry air ion flotation, the batch process had an initial surfactant concentration,  $C_{\text{surfactant}}=0.01\text{M}$  and pH=8.

Mix Pollutant	C (mg/L) after 30min	Removal (%) after 30min	C (mg/L) after 60min	Removal (%) after 60min
La	1.00	80	0.27	94.6
Fe	4.78	4.4	4.6	8.0

The significant differences in selectivity found here for the La/Fe mixture suggests that the ion flotation could be used to efficiently separate specific ion mixtures, possibly using multi-stage separation processes.

**Table 4.** A comparison of removal rates observed in a mixture of cadmium, chromium, copper, lead, lanthanum, magnesium, and iron ions (each initially at 5mg/L) using the ion flotation process with N-octanoyl-cys surfactant. The initial concentration of the surfactant was  $C_{\text{surfactant}}=0.01\text{M}$  and the solution was maintained at pH=8.

Mix Ions	C (mg/L) after 30min	Removal (%) after 30min	C (mg/L) after 60min	Removal (%) after 60min
Cd	4.4	12.0	2.8	43.8
Cr	3.57	28.6	1.07	78.5
Cu	1.53	69.4	0.82	83.6
Pb	4.76	4.8	3.72	25.6
La	4.5	10.0	3.2	34.6
Mg	4.8	4.0	4.2	16.3
Fe	4.98	0.4	4.7	6.3

Again, these results suggest that specific ion mixtures could be efficiently separated using multi-stage ion flotation processes.

**Table 5.** Ion flotation results for 100mL solutions of 50mg/L ( $7.87 \times 10^{-4}$  M) copper using crystallised N-octanoyl-cys surfactant with dry air flotation. The batch process had an initial surfactant concentration of 0.01M and pH=8.

Time (min)	C (mg/L)	Removal (%)
15	9.712	80.6
30	3.903	92
45	0.6	98.8

**Table 6.** A comparison of arsenic, cadmium, lead and nickel removal rates observed in the ion flotation process using N-octanoyl-cys surfactant. The batch process had an initial surfactant concentration of 0.01M and pH=8.

Ion species	C (mg/L) at t <sub>0</sub>	C (mg/L) after 30min	Removal (%) after 30min	C (mg/L) after 60min	Removal (%) after 60min
Arsenic	5.0	0.04	99.2%	0.03	99.4%
Cadmium	2.0	1.64	18%	1.55	22.2%
Lead	2.5	2.44	2.2%	2.25	9.8%
Nickel	3.5	3.83	2.9%	3.3	5.9%

**Table 7.** Comparison of removal rates of Au<sup>3+</sup> and Hg<sup>2+</sup> in binary mixtures of heavy metal ions (each initially at 5mg/L) using the batch ion flotation process with N-octanoyl-cys surfactant at an initial concentration of C<sub>surfactant</sub>=0.01M and pH=8.

Ion	C (mg/L) after 30min	Removal (%) after 30 min	C (mg/L) after 60min	Removal (%) after 60 min
Au <sup>3+</sup>	3.12	37.6	2.88	42.4
Au <sup>3+</sup> as (Au <sup>3+</sup> + Hg <sup>2+</sup> )	3.20	36.0	3.13	37.4
Hg <sup>2+</sup> as (Au <sup>3+</sup> + Hg <sup>2+</sup> )	3.88	22.4	3.50	30.0
Au <sup>3+</sup> as (Au <sup>3+</sup> + Fe <sup>3+</sup> )	3.62	27.6	3.38	32.4

The removal rates observed for most individual ions were typically reduced in mixed ion solutions, even with an excess of surfactant at an initial concentration of 0.01M of (Na<sup>+</sup>) N-octanoyl-cys surfactant, in the batch ion flotation process, summarized in the Tables above. The results obtained for Ca and Sr ions alone and as a mixture are a good illustration of these effects. The consistent removal rates observed, suggests that there

is strong selectivity of the N-octanoyl-cys surfactant toward arsenic, strontium, copper and chromium ions, compared with the other heavy metal ions. By comparison, there was very low removal rates observed for iron, selenium, gold and magnesium ions in these experiments. These relative selectivities could be developed further for use in specific ion separation processes.

### 3.2 HBCE sterilization. Comparison with other technologies

In Table 8, *E.coli* and MS2 virus inactivation rates using the HBCE process are compared with different studies of the most common disinfection technologies. For both pathogen groups HBCE and UV technologies presented the best inactivation results, with 3-log inactivation after 230 seconds and 3.5-log after 180 seconds, respectively when inactivating MS2 viruses and 2.3-log after 300 seconds for HBCE and 3.8-log after 300 seconds for UV, when inactivating *E.coli*. Ozone and chlorination sterilisation rates could be improved by increasing the dosage but at the concentrations used in these studies they present less or similar inactivation rates than the HBCE process<sup>30</sup> (Table 8).

The novel HBCE technology could become a new disinfection technology candidate able to compete with the existing ones. The fact that the process can use heated CO<sub>2</sub> gas and the possibility of reusing exhaust gas from combustion processes makes the HBCE process potentially more energy efficient. If pure CO<sub>2</sub> or combustion gas from gas generators is used, the only by-product that the system will generate will be 1% of carbonic acid at pH 4.1. The lack of potentially hazardous side products gives this new process a substantial advantage over other common processes.

**Table 8.** Summary of studies of inactivation of *E.coli* and MS2 virus with different technologies [30].

Pathogen	Treatment	Log 10 reduction	Time (s)	Source
Escherichia Coli	Thermal inactivation 60° C	1.5 log	300	WHO[22]
	2.0 mg O3/L	1.3 log	300	De Souza et al.[31]
	2.0 mg Cl2/L	2 log	300	De Souza et al.[31]
	UV (0.78mW/cm <sup>2</sup> ) at 295-400 nm.	3.8 log	300	Mamane et al.[32]
	DPCD, CO2 at 197 atm. and 34°C	1.25 log	300	Richard et al.[33]
	Bubble column, CO2 at 200°C, 1 atm.	2.3 log	300	Garrido Sanchis A. et al [30]
MS2 virus	0.1 mg O3/L	1.2	180	Fang et al.[34]
	1.0 mg H2O2/L	0.001 log	90	Richard et al.[35]
	30 mg Cl2/L	1 log	300	Tree et al.[36]
	UV (0.19mW/cm <sup>2</sup> )	3.5 log	180	Fang et al.[34]
	Bubble column, CO2 at 205°C, 1 atm.	3 log	230	Garrido Sanchis A. et al [30]
	Bubble column, Air at 200°C, 1 atm.	0.17 log	300	A.G. Sanchis et al.[37]

It is interesting to note from these results that hot CO<sub>2</sub> inlet gases are effective on both coliforms and waterborne viruses. By comparison, hot air is not as effective with viruses. Previous studies have used high pressure CO<sub>2</sub> for water sterilization but the high density bubble column is effective even at atmospheric pressure.

#### 4. CONCLUSIONS

Ion flotation should be more widely considered for the treatment of contaminated waters containing hazardous ions, especially when environmentally acceptable surfactants are available, which can be efficiently used in this process. For example, the recrystallised Na<sup>+</sup> form of the N-octanoyl-cys surfactant was found to be successful in removing a range of low levels of hazardous ions from water using an ion flotation process. This process offers the potential for a simple industrial physiochemical process to treat waters contaminated with a wide range of hazardous, mostly heavy metal ions, to achieve removal rates at more than 97-99% of these ions from drinking water. This process needs further study to extend its application to the recovery of valuable ions, such as gold, to reduce the significant costs of current refining processes. The surfactant also showed high efficiency for the removal of relatively high concentrations of copper ions, which could be used as a promising alternative for the treatment of industrial and mining wastewater. The significant selectivity of this surfactant for some of the ions studied suggests that the ion flotation process could be used to completely separate some specific ion mixtures. In addition, the surfactant collected in the foam can be released from the ion and then re-used to improve recycling of the surfactant to help with the commercial development of this process with potentially wide applications in several countries where these hazardous ions are present in the drinking water. This surfactant also readily decomposes into products which are acceptable for human ingestion.

Water is a very valuable resource therefore its reuse is imperative, but if this water carries pathogens these usually have to be inactivated. The HBCE process has proved its efficiency inactivating different types of pathogens in water by heating the gas, not the solution.

The HBCE process can be effectively used to inactivate viruses and bacteria in different solutions at modest temperatures of around 47°C, where normal water bath heating produces no effect. The inactivation is apparently caused by the collisions between viruses and the transient, rising hot bubbles.

Many industries, such as pig farms, landfill, bio-gas plants and coal power plants, emit large amounts of hot

combustion gases. The potential use of these hot combustion gas bubbles in water-treatment processes to sterilize water offers an attractive new energy-efficient technology. This new technology would then be able to compete with other water-disinfection technologies, such as UV irradiation, ozonation, and even chlorination, due to its low operating costs and its energy efficiency and also because of its complete absence of potentially hazardous side products.

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