



## Arsenic removal from contaminated soils for recycling via oil agglomerate flotation



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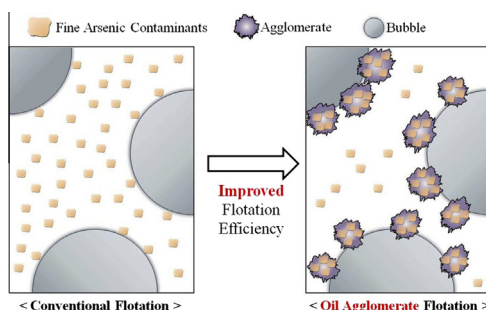
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### HIGHLIGHTS

- Oil agglomerate flotation process was developed for recycling contaminated soils.
- Adding nonpolar oils increased the arsenic removal efficiency.
- Fine particles were effectively removed by the addition of hydrocarbon oils.
- Nonpolar hydrocarbons bridged fine particles and favorably attached to the bubble.

### GRAPHICAL ABSTRACT



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### ABSTRACT

In this study, a flotation process is proposed for separating the arsenic-bearing minerals to recycle the soil surrounding the Janghang refinery in South Korea, which contains a high level of arsenic. To maximize the arsenic recovery, several experimental parameters were systematically investigated. X-ray diffraction revealed that the contaminated soil was mainly composed of arsenopyrite (FeAsS), arsenic trioxide (As<sub>2</sub>O<sub>3</sub>), arsenic pentoxide (As<sub>2</sub>O<sub>5</sub>), and quartz (SiO<sub>2</sub>). Flotation experiments with various concentrations of potassium amyl xanthate (PAX) showed that arsenic recovery increased with increasing PAX concentration. The addition of activators (Na<sub>2</sub>S, CuSO<sub>4</sub>, Na<sub>2</sub>S + CuSO<sub>4</sub>) in pulp pH greatly increased the arsenic recovery. In addition, oil agglomerate flotation was conducted to enhance the arsenic recovery by improving the separation of fine particles. The results showed that arsenic recovery increased with increasing nonpolar oil concentration. Based on the results obtained from the optimization tests, a flotation process by which the arsenic-contaminated soils can be recycled was successfully designed.

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## 1. Introduction

Flotation is a physicochemical separation technique that separates hydrophobic particles from a mixture of hydrophobic and hydrophilic particles [1–3]. Flotation takes advantage of the distinct capture capability of a bubble surface to hydrophobic

particles: more hydrophobic particles are easily attached to the bubble surface due to strong hydrophobic interaction whereas relatively fewer hydrophobic (or even hydrophilic) particles remain in the aqueous solution without the bubble attachment. However, when such a flotation technique is aimed at separating fine particles, it has not been very successful since its flotation efficiency becomes low [4–8]. In a flotation process, the bubble–particle collision and attachment, and the particle detachment from bubble surface simultaneously occur, which are known to govern the flotation efficiency [8,9]. It is generally accepted that

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a problem of fine particle flotation mainly originated from low collision probability due to their low mass, thereby hindering the attachment of the particles to air bubbles [5,10,11].

A typical route to improve such a poor flotation efficiency of fine particles is achieved by the addition of oil, termed oil agglomerate flotation [7,12–14]. When a small amount of oil is added, it acts as an adhesive between hydrophobic fine particles by the capillary force, resulting in big aggregates consisting of fine particles bridged by the added oil. Larger agglomerates are then produced instead of individual fine particles that interact with bubbles. Meanwhile, hydrophilic particles remain unaffected by the bridging reagents. This aggregation process lessens the problem of the low probability of collision and attachment of fine particles to bubbles [5,8,15,16]. Eventually, the agglomerated products can be separated using the conventional flotation technique [7,12–14,17].

Exploiting this strategy, several systems have been investigated to improve the recovery of fine particles such as coal [18–22], gold [23–25], and molybdenite [17,26–28]. Until now, however, studies on oil agglomerate flotation are limited to the recovery of minerals, and as far we are aware, there have been no attempt to investigate the remediation of soils using the agglomerate flotation technique. Since contaminated soil generally has a wide range of size distribution from 5  $\mu\text{m}$  to 5000  $\mu\text{m}$  [29–31] and it contains numerous fine particles, agglomerate flotation technique might be a proper direction to separate the contaminated particles from the soil.

Arsenic is one of the most dangerous inorganic pollutants, causing serious health and environmental emergencies in many areas of the world [32,33]. For this reason, all over the world, arsenic concentration in soil is being rigorously regulated, although it differs by orders of magnitudes for each region and country [34]. According to the Korea Soil Standard Criteria, the warning level for arsenic concentration in soil is 25 mg/kg for zone 1 areas [35], which include farmland, ranch lot, school lot, park and etc. [35]. The goal of this study is to find an optimum experimental condition for lowering arsenic concentration of contaminated soil below the warning level (25 mg/kg) by separating the arsenic-bearing minerals using flotation technique. Accordingly, we studied the effects of concentration of collector, type of activators, and more importantly, systematically investigated the effect of oil addition to a typical flotation technique on the purification of arsenic-contaminated soil. Experimental results suggest that the conventional flotation method cannot separate the arsenic-bearing minerals from soil effectively, in particular for fine particles. However, it is demonstrated that the agglomerate method by the addition of oil can better separate the arsenic-bearing minerals from soil by more effectively separating the fine particles that remained unseparated in the conventional flotation.

## 2. Experimental approach

### 2.1. Samples

The arsenic-containing soil surrounding Janghang refinery (Seocheon, South Korea) was selected for this study. The soil in this area consists mostly of sand; yet some particles fly in from

leaching factories. The main pollutant is arsenic (Table 1). For the flotation experiments, the soils surrounding Janghang refinery were ground in a ball mill (UBM 100S, SPG Co., Ltd, South Korea) at a rotational speed of 10 rpm for 5 min, reducing the particle to pass 100 mesh (150  $\mu\text{m}$ , Tyler Standard). It is worthwhile mentioning that arsenic concentration of the soils with the size above 150  $\mu\text{m}$  was found to be about 19 mg/kg (data not shown), which meets the maximum arsenic concentration for recycling (25 mg/kg), and thus all products are recyclable. Hence, the samples with the size below 150  $\mu\text{m}$  were only collected and used for the flotation experiments to lower the arsenic concentration to the warning level. To investigate the chemical composition and mineralogy of the contaminated soils, inductively-coupled plasma (ICP) (iCAP 7000 Series ICP-OES, Thermo Fisher SCIENTIFIC, USA), X-ray fluorescence (XRF) (Sequential XRF-1800, Shimadzu, Japan), and X-ray diffraction (XRD) analyses (D/Max-2200/PC, Rigaku, Japan) were performed.

### 2.2. Reagents

The collector used in this study was the potassium amyl xanthate (PAX,  $\text{C}_5\text{H}_{11}\text{OCS}_2\text{K}$ ) with purity higher than 90%, obtained from Hong Yuan Industry & Trade Co., China. In addition, sodium sulfide ( $\text{Na}_2\text{S}$ ) and copper sulfate ( $\text{CuSO}_4$ ) manufactured by Sigma-Aldrich were used as activators. Kerosene ( $\text{C}_n\text{H}_{2n+2}$ ,  $12 \leq n \leq 15$ ), diesel ( $\text{C}_n\text{H}_{2n+2}$ ,  $15 \leq n \leq 18$ ), and hexadecane ( $\text{C}_{16}\text{H}_{34}$ ) made by Samchun Pure Chemical Co., Ltd., South Korea were used as oil agglomerate reagents. DowFroth-250 (DF-250,  $\text{CH}_3(\text{OC}_3\text{H}_6)_4\text{OH}$ ) supplied by American Cyanamid, USA was used as a frother and analytical grade of HCl and NaOH (Fisher Scientific) were used as pH modifiers.

### 2.3. Flotation tests

For the flotation experiments, a 1 L flotation cell (Denver Sub-A, USA) was employed. For all tests, the following identical conditions were used: 120 g of contaminated soils, 880 mL deionized (DI) water, an impeller speed of 1200 rpm, and a flotation time of 10 min. In addition, the DF-250 (750 mL/ton) was conditioned for 3 min before air was introduced in the suspension. In the first set of experiments to choose the desired concentration of collector, PAX (50–200 g/ton) was added to the pulp for 5 min, followed by 3 min of frother conditioning and 10 min of flotation at pH 4. For the second set of experiments,  $\text{Na}_2\text{S}$  (200 g/ton),  $\text{CuSO}_4$  (200 g/ton), and mixed  $\text{Na}_2\text{S}$  and  $\text{CuSO}_4$  (200 g/ton) were added for 5 min to examine the influence of activators on arsenic recovery, followed by 5 min of collector conditioning, 3 min of frother conditioning, and 10 min of flotation at pH 4. For the third set of experiments, to improve the separation of arsenic contaminated fine particles, oil agglomerate experiments were also conducted. For oil agglomerate experiments, kerosene, diesel, and hexadecane were used. Mixed  $\text{Na}_2\text{S}$  and  $\text{CuSO}_4$  (200 g/ton) were added to the pulp at pH 4 for 5 min, followed by 5 min of collector conditioning, 5 min of kerosene, diesel, and hexadecane (100–300 g/ton) conditioning, respectively, 3 min and frother conditioning, and 10 min

**Table 1**  
Elemental and mineral composition of the Janghang refinery surrounding soils.

Elemental composition (mg/kg)										
As	Cd	Cr	Cu	Pb	Zn					
332.21	5.14	10.38	25.49	1.26	27.72					
Mineral composition (%)										
$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	$\text{Fe}_2\text{O}_3$	CaO	MgO	$\text{K}_2\text{O}$	$\text{Na}_2\text{O}$	$\text{TiO}_2$	MnO	$\text{P}_2\text{O}_5$	
58.17	13.41	8.87	1.54	0.29	0.34	1.12	0.05	0.01	0.38	

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