Polychlorinated biphenyls removal from weathered municipal solid waste incineration fly ash by collector-assisted column flotation

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Abstract

Polychlorinated biphenyls (PCBs), polychlorinated dibenzo-*p*-dioxins (PCDDs) and dibenzofurans (PCDFs) are highly toxic micropollutants emitted from municipal solid waste incineration (MSWI), in particular, concentrated in the unburned carbon (UC) of MSWI fly ash. Because of concerns over their adverse health effects, a number of countries have classified MSWI fly ash as hazardous material and required further treatment before its final disposal in landfills. The technologies for removing the toxic chlorinated micropollutants in the MSWI fly ash have been studied, however, until now no mature technique has been obtained in this purpose. In this research, we used a technique of collector-assisted column flotation to remove PCBs-enriched UC from MSWI fly ash. We found that 36.9% PCBs could be removed from fresh MSWI fly ash with 61.7% UC removal efficiency, whereas only 21.7% PCBs could be removed from weathered MSWI fly ash with a low UC removal efficiency of 33.7%. By adding a mixture of two kinds of surfactants: sorbitan mono-oleate and polyoxyethylene (20) sorbitan mono-oleate to the weathered fly ash slurry as the collector assistant, 39.3% PCBs was removed at the hydrophile–lipophile balance (HLB) value of 13.5, while the UC removal efficiency increased to 49.0%. The results showed that the collector assistant could enhance PCBs and UC removal efficiencies during the column flotation process, and the mechanism has been discussed in detail. Higher PCBs and UC removal efficiencies could be expected by further optimizing the conditions of collector-assisted column flotation.

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1. Introduction

The reduction of the emissions of persistent organic pollutants (POPs), including polychlorinated dibenzo-*p*-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs) and polychlorinated biphenyls (PCBs), to the environment is a contemporary issue of global efforts, as considering their high toxicity and great threat to the environment [1,2]. Among many emission sources of PCDD/Fs, PCBs, such as metallurgical industry, chemical manufacturing and combustion sources, municipal solid waste incineration (MSWI) is one major source because of the large quantity of the municipal solid waste and the immature incineration technique [3,4]. In particular, about a half of all the PCDD/Fs, PCBs and other organic micropollutants emitted from MSW incinerators are absorbed on the surface of fly ash particles [3]. The environmental protection agencies of many countries have classified MSWI fly ash as hazardous material and required the further treatment of MSWI fly ash before its disposal in final landfills [5]. Up to date, intermediate treatments such as thermal destruction [6], base catalyzed decomposition [7] and hydrothermal decomposition [8] have been studied and developed to decompose the toxic organic micropollutants in MSWI fly ash. However, there is the possibility of PCDD/Fs, PCBs regeneration in the low-temperature zone of processes using a thermal destruction technique, but alternative methods are still in laboratory testing stage. Therefore, it is important to find an effective technique to remove the organic contaminants in the MSWI fly ash.

The UC is the major source of organic micropollutants in MSWI fly ash due to its large adsorptive surface area and its role as the origin of the de-novo synthesis [6,9]. For this reason, it is supposed that most organic micropollutants can be effectively removed with the removal of UC from fly ash.

Column flotation is a low energy consumption technique widely used in the solids separation encountered in primary mineral and chemical industries, such as coal cleaning [10–12]. We used this technique to remove PCBs by removing PCBs-enriched UC from MSWI fly ash. However, from our former experimental results, we found that although the common collector kerosene was efficient in collecting UC in the flotation of fresh fly ash [13], it could not collect the UC efficiently in the flotation of weathered fly ash. This is because the weathering process results in the formation of oxygen functional groups, which reduce the hydrophobicity of the UC surface [14,15].

It was found that the use of non-ionic surfactants as the collector assistant or alternative collector, which acted on the surface of the weathered coal particles, could increase the coal recovery [16,17]. Moxon and Keast-Jones [16] used ethoxylated nonyl phenol with an average of five ethoxy groups as the collector assistant for dodecane in the flotation of coal with MIBC as the frother. They found that the recovery of coal increased about 12% at the ethoxylated nonyl phenol addition of approximately 0.5%. Jia et al. [17] reported the improvement of flotation of lower rank and oxidized coals by using non-ionic oxygenated surfactants as the collector.

Since the removal efficiency of UC and PCBs from the weathered fly ash by column flotation will increase with the increase of the UC collection efficiency of the collector, a mixture of two kinds of surfactants: sorbitan mono-oleate and polyoxyethylene (20) sorbitan mono-oleate was added to kerosene as the collector assistant in the flotation procedure. The objectives of our study are, therefore: to investigate the effects of the collector assistant (the

surfactant mixture) on the PCBs and UC removal efficiencies have been investigated; and to discuss the mechanism of the enhancement of the flotation procedure by the collector assistant.

2. Materials and methods

2.1. MSWI fly ash

Fly ash was obtained from an electrostatic precipitator (ESP) of a stoker type MSW incinerator. There was neither slaked lime nor activated carbon injection in the gas treatment system. The chemical analysis result of the fly ash contents is shown in Table 1. The UC concentration 5.24% (Table 1) is the weight concentration of the UC in the total fly ash sample. Before analyzing the concentrations of PCBs in different particle size parts, the fly ash was divided into five particle size ranges ($d > 500$, $250 < d < 500$, $106 < d < 250$, $44 < d < 106$ and $d < 44\,\mu m$) by sieves (as shown in Table 2), and the weight distribution was measured.

In order to study the effects of the collector assistant, the fly ash was classified into two types according to their storage time, i.e. the fresh and the weathered MSWI fly ash. The weathered MSWI fly ash was stored in a plastic tank within air atmosphere for 10 months.

2.2. Collector-assisted column flotation

The column flotation system was schematically shown in Fig. 1. The prepared slurry as described below and 0.6 ml methyl *iso*-butylcarbinol (MIBC) were fed from the top of the glass column after starting the air compressor. MIBC was added as the frother. Compressed

Table 1
Chemical analysis of fly ash

UC concentration (wt.%)	5.24
Na (mg/kg)	$6.37E + 06$
Mg (mg/kg)	$5.96E + 05$
Al (mg/kg)	$7.45E + 06$
K (mg/kg)	$4.87E + 06$
Ca (mg/kg)	$4.81E + 06$
Ti (mg/kg)	$1.23E + 06$
Fe (mg/kg)	$1.76E + 06$
Zn (mg/kg)	$2.13E + 06$
Cr (mg/kg)	$1.21E + 02$
Mn (mg/kg)	$1.31E + 03$
Cu (mg/kg)	$1.74E + 03$
Mo (mg/kg)	$2.76E + 01$
Cd (mg/kg)	$2.04E + 02$
Sb (mg/kg)	$9.53E + 02$
Pb (mg/kg)	$8.88E + 02$

Table 2
The GC–MS analysis and calculated PCBs concentrations

IUPAC nos.	GC–MS analysis PCBs concentration (ng/g)						Calculated PCBs concentrations in fly ash, $\widehat{C}$
	Original fly ash, C	$d > 500$	$500 > d > 250$	$250 > d > 106$	$106 > d > 44$	$44 > d$	
77	0.7	0.9	0.3	0.4	0.5	1.3	0.6
81	0.0	0.0	0.0	0.0	0.0	0.0	0.0
126	0.7	0.2	0.2	0.3	0.4	1.4	0.6
169	0.5	0.2	0.1	0.2	0.3	1.0	0.4
123, 106	0.1	0.0	0.0	0.0	0.0	0.1	0.0
118	1.4	0.3	0.2	0.2	0.3	0.7	0.4
105	1.1	0.3	0.2	0.2	0.3	0.7	0.3
114	0.2	0.5	0.1	0.1	0.1	0.3	0.2
156	1.0	0.5	0.1	0.5	0.6	1.7	0.8
157	0.3	0.1	0.1	0.2	0.2	0.6	0.3
128, 167	1.0	0.3	0.2	0.4	0.5	1.4	0.6
189	0.8	0.3	0.2	0.4	0.4	1.5	0.6
180	0.4	0.1	0.1	0.1	0.1	0.5	0.2
170	1.3	0.7	0.4	0.7	0.8	2.4	1.1
T3	7.9	16	7.6	7.3	7.0	13	9.3
T4	24	13	7.7	10	13	32	16
P5	23	7.6	6.6	11	12	34	16
H6	7.0	3.3	1.5	2.9	3.4	9.3	4.4
H7	4.8	2.5	1.3	2.3	2.4	8.0	3.6
O8	4.0	1.8	1.1	2.0	2.1	7.4	3.2
Total	71	44	26	36	40	103	52

IUPAC no: the number of the PCBs congeners, from the International Union of Pure and Applied Chemists (IUPAC).

air with a pressure of $6.86 \, \text{N/cm}^2$ was passed through the gas distributor and sheared into small bubbles which attached and transported the hydrophobic particles (mainly consisting of UC particles) to the upper region of the column and form a froth zone. The volumetric gas flow rate was 0.7 l/min. The hydrophilic mineral particles remained at the bottom of the column as the residue. The froth was collected together by scraping the froth zone with spoon 5 min once during the flotation process. The flotation time was kept for 30 min. Then, the slurry was filtered and divided into residue and solution parts. The whole collected froth during the flotation process, residue and solution were kept as samples. More details of the chosen of optimum experiment conditions were described in our former paper [13].

For the column flotation without using the collector assistant, the slurry was made by mixing 10 g fly ash sample with 750 ml distilled water for 5 min using a jar-tester at 200 rpm. Then 3 ml collector kerosene was added to the slurry, the slurry was agitated for more 5 min. The prepared slurry was used for the flotation using no collector assistant.

It has been indicated by our previous work that the collector kerosene was effective to remove UC from fresh MSWI fly ash [13], but for the weathered fly ash it lost effect. Therefore, we added a surfactant mixture as the collector assistant in the slurry to enhance the collector kerosene's collection efficiency. Two kinds of surfactants: 86.0 wt.% of sorbitan
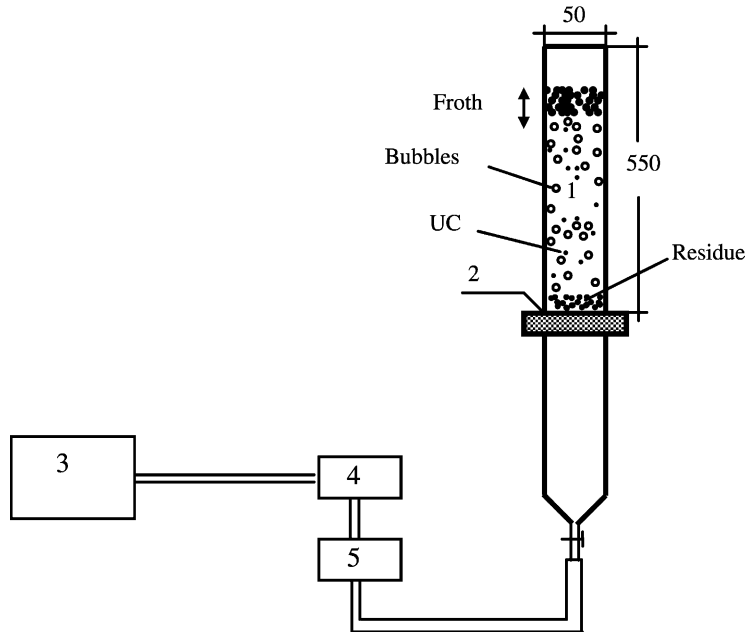
Fig. 1. Schematic diagram of the flotation column: 1, column; 2, gas distributor (porous plate); 3, air compressor; 4, flow controller; 5, flow meter.

mono-oleate (HLB = 4.3) and 14.0 wt.% of polyoxyethylene (20) sorbitan mono-oleate (HLB = 15.0) were mixed to obtain a surfactant mixture with hydrophile–lipophile balance (HLB) value of 13.5. This surfactant mixture was added to the kerosene at the concentration of 3% by volume to get a kerosene mixture. According to previous experiment results, it was found that the optimum UC removal efficiency for the weathered fly ash could be achieved when the HLB value of the surfactant mixture was 13.5 and the concentration of the surfactant mixture in the kerosene was 3% [18]. The kerosene mixture can be looked as modified collector. The 3.09 ml prepared kerosene mixture was added to 350 ml distilled water and stirred for 10 min using jar-tester at 200 rpm to get the kerosene emulsion. After 10 g fly ash sample was mixed with 400 ml distilled water for 5 min, the kerosene emulsion was added to the fly ash slurry and mixed for 5 min more. The pH values of the original fly ash slurry (pH 6.9) were adjusted to the optimum pH value of 5.9 for the UC flotation by using 0.1 mol/l HCl or 5 mol/l NaOH solution [13]. The prepared slurry was used for the flotation using the collector assistant.

Initially, we prepared slurry with the direct addition of kerosene mixture instead of the addition of the kerosene emulsion. However, we found that the UC removal efficiency of the flotation process using kerosene mixture was worse than the process using kerosene emulsion. This is due to the reason that a stable kerosene emulsion can well distribute the kerosene mixture in distilled water. When the kerosene emulsion was mixed with the fly ash slurry, the UC solid particle surface can be well attached by the oily particle of the kerosene mixture.

2.3. TOC measurement GC–MS analysis

The UC concentrations of fly ash, froth and residue samples were measured by total organic carbon analyser (TOC-5000A/SSM-5000A: Shimadzu). The collected froth, residue and original MSWI fly ash were dried at 105 °C for 24 h. Then, the samples were treated with 2 mol/l HCl for 2 h, filtrated and dried at 105 °C for 12 h before the TOC value was measured.

The concentration of PCBs in froth, residue and MSWI fly ash samples were analyzed by HRGC/LRMS (HP6890/HP5973: Agilent) with the internal standards of ^{13}C-PCBs (nos.: 28, 52, 101, 118, 138) (Wellington Lab.). The solid samples were digested with 2 mol/l HCl at the ratio of 20-mmol/g sample for 2 h. The filtered samples were dried at the room temperature for 48 h and extracted by refluxing with 200 ml toluene for 24 h. The extracts were concentrated to 100 μl by the rotary evaporation and nitrogen blowing after having been cleaned in multilayer silica columns with silica gel (7 *g*) + 50% AgNO$_3$ (5 *g*) + Na$_2$SO$_4$ (6 *g*). The solution sample (approximately 1 l) was extracted three times by 100 ml dichloromethane after adding the internal standards (^{13}C-PCBs), then concentrated to 100 μl by the rotary evaporation and nitrogen blowing.

2.4. Measurement of the metal concentrations in the fly ash

The metal concentrations (Table 1) were measured by ICP-AES (ICP-8000: Shimadzu). Before the measurement, a 100 mg sample was digested in an acid mixture of 5 ml HNO$_3$, 2 ml HCl and 3 ml HF using a microwave oven (MDS-2000: CEM corp.) for 60 min. The acidic solution was then neutralized by 18 ml boric acid and put in the microwave oven for 5 min. Finally, the sample was filtrated and the filtrate was used as the measuring sample.

3. Results and discussion

3.1. UC and PCBs concentrations and distributions in the fly ash

The UC and PCBs concentrations were measured at each particle size range by TOC and GC–MS measurement, respectively, as shown in Table 2. The corresponding balance calculation values of PCBs concentrations were derived from the following equation:

$$\widehat{C} = \sum_{i=1}^{5} C_i D_i \tag{1}$$

where $\widehat{C}$ is calculated PCBs concentrations in fly ash (ng/g); C_i the concentration of the PCB isomer in i-th particle size range (ng/g); D_i the weight fraction of the i-th particle size range.

In Table 2, C represents the GC–MS analysis PCBs concentrations of the original fly ash sample. The calculated $\widehat{C}$ values show good consistency with those of the original GC–MS analysis concentrations C.
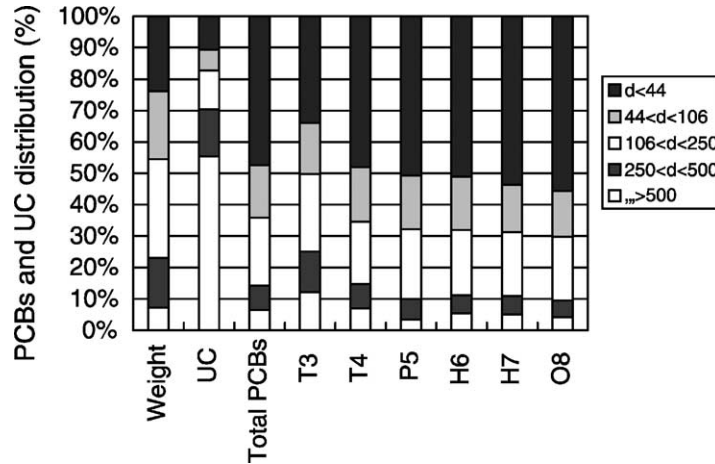
Fig. 2. PCBs and UC distribution in MSWI fly ash with different particle size.

The concentrations of total trichlorobiphenyls (T_3), tetrachlorobiphenyls (T_4), pentachlorobiphenyls (P_5), hexachlorobiphenyls (H_6), heptachlorobiphenyls (H_7) and octachlorobiphenyls (O_8) were measured by GC–MS and counted up.

The distributions of PCBs and UC in different particle size ranges are shown in Fig. 2. It is obvious that the smaller particle size fraction (e.g. PCBs = 47.6% in $d < 44\,\mu m$) contains more PCBs than the larger particle size parts (e.g. PCBs = 6.4% in $d > 500\,\mu m$ and PCBs = 7.9% in $250 < d < 500\,\mu m$). This is because PCBs are more easily generated and absorbed on the surface of the small size particles, where there are more active sites for the PCBs synthesis reaction under the heavy metals catalysis [19,20].

3.2. PCBs removal under different column flotation conditions

The recovery efficiency of UC R_c and the recovery efficiency of PCBs homologues R_p are defined by:

$$R_c(\%) = \frac{m_f C_f}{m_0 C_0} \times 100 \tag{2}$$

$$R_p(\%) = \frac{m_f C_{pf}}{m_0 C_{p0}} \times 100 \tag{3}$$

where m_f and m_0 are the weights of the froth and the original fly ash sample used in the flotation experiment; C_f and C_0 the UC concentrations in the froth and the fly ash; C_{pf} and C_{p0} the concentrations of each PCBs homologues in the froth and the fly ash samples.

The removal efficiencies of each homologue of PCBs under different column flotation conditions were shown in Fig. 3. From Fig. 3(a) and (b), we can see that the removal efficiencies of all homologues of PCBs for the weathered fly ash are lower than those of the
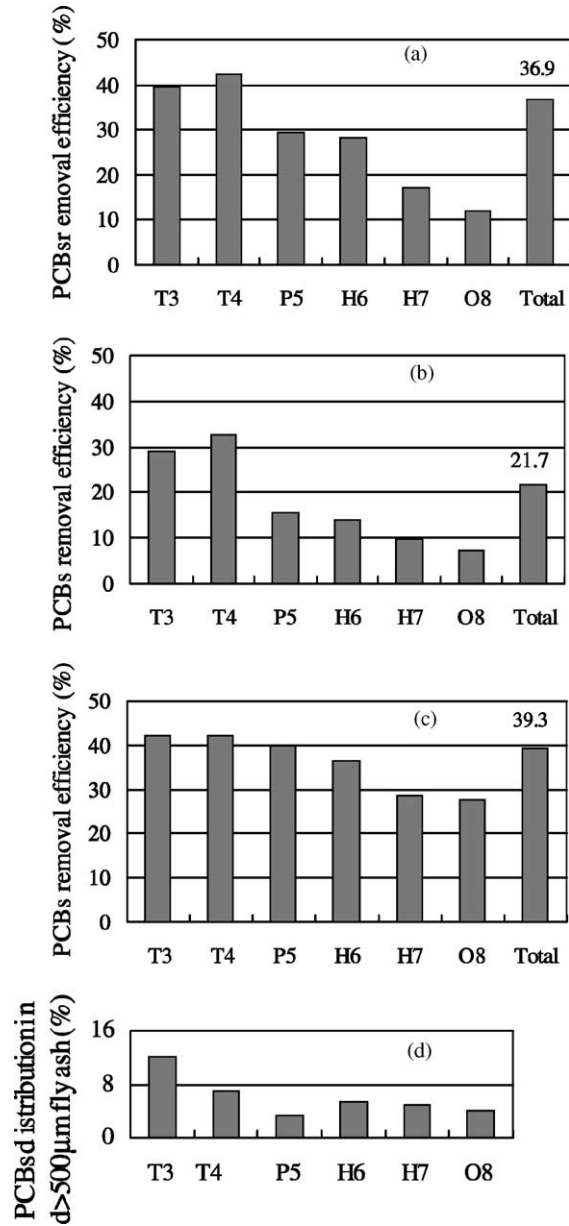
Fig. 3. The removal efficiency of PCBs under different flotation conditions: (a) flotation of the fresh fly ash without using collector assistant; (b) flotation of the weathered fly ash without using collector assistant; (c) flotation of the weathered fly ash enhanced by collector assistant; (d) PCBs distribution in $d > 500\,\mu m$ fraction of the fly ash.

fresh fly ash. By adding the surfactants in the weathered fly ash, the removal efficiencies of T_3, T_4, P_5, H_6, H_7 and O_8CBs increased by 13.3, 9.6, 24.6, 22.3, 18.7 and 20.3%, respectively (Fig. 3(c)). The removal efficiencies of T_3 and T_4CBs are higher than those of other homologues under the flotation conditions (a) and (b). This result is coincident with the particle size distributions of $d > 500\,\mu m$ (seeing Figs. 2 and 3(d)). As most UC particle size in the froth sample of the flotation without using the collector assistant are in the range of $d > 500\,\mu m$ [13]. Therefore, the difference among the PCBs removal efficiencies is mainly attributed to the effect of fly ash particles with the size of $d > 500\,\mu m$. This tendency is not so obvious in Fig. 3(c) compared with those of Fig. 3(a) and (b), because more fine UC particles are floated up and removed by the addition of surfactants.

3.3. Effects of the collector assistant on the PCBs and UC removal efficiencies during collector-assisted column flotation

The total PCBs and UC removal efficiencies under different flotation conditions corresponding to Fig. 3(a)–(c) are shown in Fig. 4. We find that 36.9% PCBs can be removed from fresh MSWI fly ash with 61.7% UC removal efficiency, whereas only 21.7% PCBs can be removed from weathered MSWI fly ash with a low UC removal efficiency of 33.7%. The decreases of PCBs and UC removal efficiencies are due to the weathering of the UC in the fly ash. Most UC is susceptible to oxidation by weathering, which can occur during the storage. Weathering processes results in the formation of more oxygen functional groups, such as carboxyl, phenolic and carbonyl functionalities on the UC surface, which reduce the hydrophobicity of the coal surface by increasing the number of sites that hydrogen bond with water molecules [14,15]. This is the reason why the weathered UC is more difficult to float with common oily collectors, such as kerosene used in our experiments.

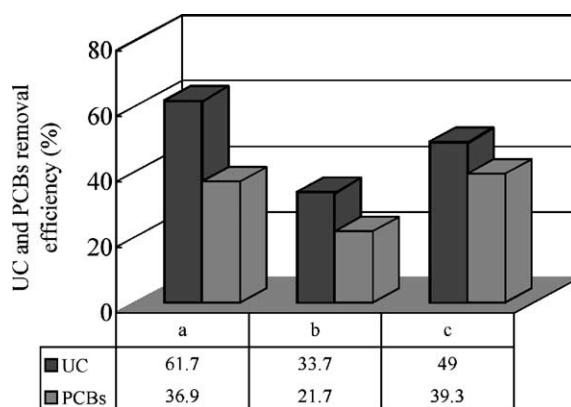|	a	b	c
■ UC	61.7	33.7	49
▨ PCBs	36.9	21.7	39.3

Fig. 4. Comparison of the removal efficiencies of UC and total PCBs under different flotation conditions: (a) flotation of the fresh fly ash without using collector assistant; (b) flotation of the weathered fly ash without using collector assistant; (c) flotation of the weathered fly ash enhanced by collector assistant.

HO(CH$_2$CH$_2$O)$_w$ (OCH$_2$CH$_2$)$_x$OH

CH(OCH$_2$CH$_2$)$_y$OH

CH$_2$(OCH$_2$CH$_2$)$_z$OOCCH$_2$(CH$_2$)$_5$CH$_2$CH=CHCH$_2$(CH$_2$)$_6$CH$_3$

A   B

w+ x + y +z = 20

Polyoxyethylene (20) sorbitan mono-ole ate

HO   OH

CH OH

CH$_2$OOCCH$_2$(CH$_2$)$_5$CH$_2$CH =CHCH$_2$(CH$_2$)$_6$CH$_3$

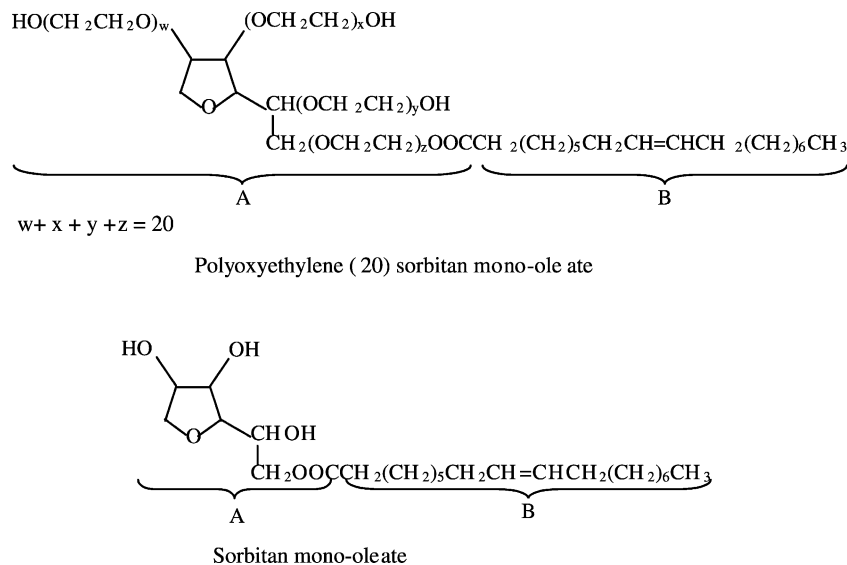A   B

Sorbitan mono-oleate

Fig. 5. Structures of polyoxyethylene (20) sorbitan mono-oleate and sorbitan mono-oleate.

In order to improve the total PCBs and UC removal efficiencies, we add the surfactant mixture (HLB $=$ 13.5) of sorbitan mono-oleate and polyoxyethylene (20) sorbitan mono-oleate to the slurry as the collector assistant. The total PCBs and UC removal efficiencies increase by 17.6 and 15.3%, respectively. The 39.3% PCBs is removed while the UC removal efficiency increases to 49.0%. The total PCBs and UC removal efficiencies show a good correlation, which also gives the experimental evidence that UC is the major source of PCBs.

The assistance of the surfactant mixture (the collector assistant) to the PCBs and UC removal efficiencies in the column flotation can be understood from the chemical structure of these two kinds of surfactants (polyoxyethylene (20) sorbitan mono-oleate and sorbitan mono-oleate) as shown in Fig. 5 [21]. These two kinds of non-ionic surfactants are bifunctional: they possess oxygenated functional groups (A) and a hydrocarbon chain (B). In the oxygenated functional part (A), there are carboxyl, hydroxyl, esters of sorbitol and its mono- and di-anhydrides groups; in the hydrocarbon chain part (B), there is the oleic acid moiety. As we know, the surface of UC consists of inherently hydrophobic areas (carbonaceous sites) and also sites containing oxygenated moieties such as carboxyl, carbonyl, phenolic and ester groups. Therefore, the attachment of the collector assistant to the UC particle surface can take place by two mechanisms:

1. Hydrogen bonding of polar oxygenated functional groups of the surfactant mixture (collect assistant) with the oxygenated functional groups on the UC surface.
2. van der Waals interaction (hydrophobic bonding) of the non-polar hydrocarbon chain of the surfactant mixture with the carbonaceous sites on the UC surface.

The hydrogen bonding interaction is much stronger than the van der Waals interaction. For the weathered UC particle that contains a large fraction of oxygen functional groups,

attachment of the collector is mainly through hydrogen bonding with the polar oxygen atoms of the collector. Since there are not almost any oxygenated functional groups on the molecule of kerosene, kerosene has limited affinity for a weathered UC particle surface. Thereby the attachment and collection efficiency of the weathered UC by kerosene is lower than those of the kerosene mixture with the collector assistant which contains oxygenated functional groups. By using the collector assistant the UC removal efficiency can be dramatically improved during the flotation.

3.4. Leaching of PCBs in flotation process

The leaching ratios of each PCBs homologues in the liquid phase are shown in Fig. 6. The leaching ratio is the partition ratio of the PCBs desorbed from fly ash and dissolved in the liquid phase during flotation process. The total PCBs leaches to the liquid phase under condition (c) is higher than that under condition (b). This is because that the surfactant mixture used in condition (c) can enhance the affinity between the UC and the kerosene, which make more PCBs dissolve in the kerosene, and then transfer to the liquid phase.

At all the ratios of PCBs dissolved in the liquid phase were much lower than the PCBs left in froth and residue parts. Only 2.1 and 5.8% PCBs dissolved in the liquid phase under conditions (b) and (c). The PCBs left in froth, residue after flotation process under condition (b) and (c) were from 21.7 to 76.2 and 39.3 to 54.9%, respectively.
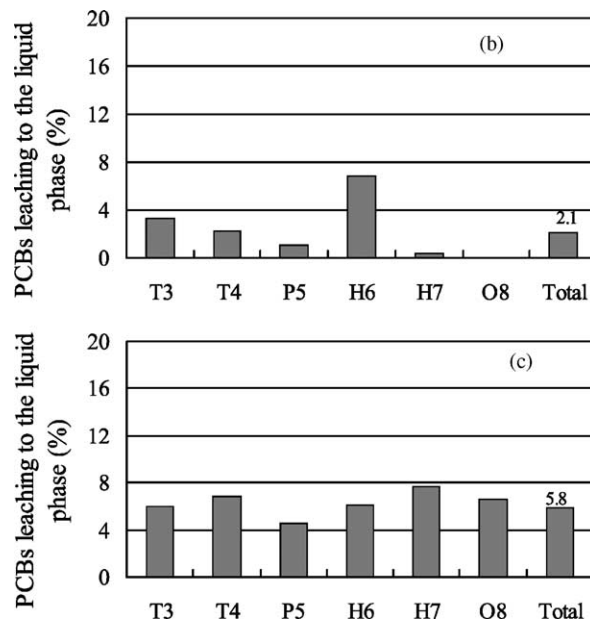

Fig. 6. Leaching ratios of PCBs homologues in the flotation conditions: (b) flotation of weathered fly ash without using collector assistant; (c) flotation of the weathered fly ash enhanced by collector assistant.

4. Conclusions

1. Collector assistant (the surfactant mixture of polyoxyethylene (20) sorbitan mono-oleate and sorbitan mono-oleate) has important effects on enhancing the PCBs and UC removal from weathered MSWI fly ash during column flotation compared with the column flotation without using the collector assistant. The PCBs and UC removal efficiencies increased from 21.7 to 39.3%, and 33.7 to 49.0%, respectively.
2. The assistance of the collector assistant (the surfactant mixture of polyoxyethylene (20) sorbitan mono-oleate and sorbitan mono-oleate) to the UC and PCBs removal during the column flotation is attributed to the following reasons: (1) polar groups of the surfactant mixture interact with the oxygenated functional groups on the UC surface by hydrogen bonding; (2) the non-polar chains of the surfactant mixture interact with the carbonaceous sites on the UC surface.
3. Higher PCBs and UC removal efficiencies can be expected by further optimizing the conditions of collector-assisted column flotation.
4. Column flotation can be effectively used to remove PCBs from fresh MSWI fly ash. In this experiment, 36.9% PCBs are successfully removed with 61.7% UC removal efficiency from fresh MSWI fly ash.

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