

Effect of chloride on the decarburization and detoxification of washed incinerator fly ash

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ABSTRACT

Medical waste incinerator (MWI) fly ash has a high content of carbonaceous matter and dioxins as well as a large amount of chloride. The flotation behavior of washed fly ash (WFA) at NaCl concentrations of 0.05, 0.1, 0.15, and 0.2 mol/L was investigated to evaluate the effect of chloride on the flotation of MWI fly ash. The study results revealed that WFA without NaCl addition exhibited weak flotation. The removal efficiencies of carbon and dioxins from WFA were found to be similar and increased with increasing NaCl concentrations. Solubility of dioxins was also enhanced. The highest removal efficiencies of carbonaceous matter and total dioxins reached 88.5% and 84.4%, respectively, at 0.15 mol/L NaCl. Patterns of distribution of dioxin congeners in the remaining solution were similar to those in the WFA.

Keywords: Washed fly ash; Flotation; Chlorides; Surfactant; Dioxins

1. Introduction

Incineration has become popular for medical waste treatment in China [1]. Medical waste contains a large amount of chlorine-containing plastics as well as NaCl, which is used as a disinfectant. These chemicals may lead to the production of considerable quantities of polychlorinated dibenzo-*p*-dioxins and dibenzofurans (PCDD/Fs) and chlorines in medical waste incinerator (MWI) fly ash. In addition, MWI fly ash contains a high content of carbonaceous matter, including unburned carbon (UC) and powder-activated carbon (PAC), which are the main sources of PCDD/Fs because of their adsorptive behavior [1,2,3]. Thus, removal of carbon from MWI fly ash is an urgent concern. Our previous study suggested that the flotation process may concentrate dioxins and carbonaceous matter, including UC and PAC, from fresh MWI fly ash into the froths, leaving a low content of dioxins in the tailings [4].

Because the daily output of MWI fly ash is relatively small (approximately 300–500 kg/d), part of the fly ash is

usually packed and transported to designated stockpiles within incineration plants for 3–6 months or longer [5]. UC in fly ash might undergo oxidation due to weathering [6], which increases oxygen functional groups, such as carboxylic esters (–COOH) and phenols (–OH) [7], on its surface and makes flotation more difficult [8]. Thus, surfactants must be added to improve flotation efficiency [9].

The flotation process of weathered fly ash is complex. Many factors may affect carbon removal, such as the flotation reagent, current density, pulp pH, and co-existing ions. MWI fly ash contains particles with various chemical compositions. The chloride content, particularly of the MWI fly ash [2], which could reach approximately 20%, can increase the amount of dissolved ions and the ionic strength of the slurry, affecting flotation performance. Consequently, understanding the effect of chloride on flotation performance is important.

Previous studies have reported that chloride leads to the presence of ions, such as Ca²⁺, Mg²⁺, NO₃⁻, and that sulfate and chloride could change the hydrophobicity of minerals and then affect the flotation process [10]. Harvey

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pointed out that coal flotation improved with increasing electrolyte concentration [11]. Gao found that Cl^- addition had a promoting effect on algae removal during electro-coagulation-flotation [12]. Ozdemir proved that dissolved salts have a strong ion effect on coal flotation and the salt concentration of solutions plays an important role in coal flotation; the author also reported an optimum salt concentration [13]. Some studies have investigated the effect of addition of salt water as a frothing agent during flotation [14]. In addition, bubble size in the pulp phase was reduced in the electrolyte-containing solution [13]. An increase in electrolyte concentration was found to produce a large amount of fine bubbles, thus increasing froth stability [10]. Laskowski noted that 1 mol/L NaCl had a similar inhibitory effect on bubble coalescence as 10 mg/L MIBC [15]. Some research indicated that froths were more stable with increasing salt concentrations in the absence of a frothing agent and that a large amount of bubbles would increase the coal flotation efficiency of salt solutions [16,17]. Therefore, the performance of surfactants, which act as promoters, is strongly affected by the liquid environment, such as the ionic strength of the solution.

The presence of ions in the flotation system has been suggested to further increased the complexity of the problem [10]. When considering chloride unavoidably existent in MWI fly ash, it is important to understand the effect of chloride ions on carbon and dioxin removal during flotation. In this study, a moderate amount of NaCl was added to the washed fly ash (WFA) for testing the effect of NaCl concentration on carbon and dioxin removal. The results provided useful information about removing dioxins by flotation in the presence of high chloride in the ash.

2. Materials and methods

2.1. Materials

The raw fly ash (RFA) sample, which was weathered for six months, is collected from incineration plants in southern China, which equipped with a 15 t/d of gyration kiln incinerator. Then it was homogenized and screened by a sieve of 20 meshes to remove large particles, and dried for 24 h at 105°C. The loss on ignition (LOI) of this sample was determined in accordance with the standard for pollution control (GB18485-2014).

In order to investigate the effect of chloride salts on the flotation performance of MWI fly ash, the RFA sample was washed by 100 g/L of prepared distilled water with a motor stirrer at 500 rpm for 30 min to remove the soluble chloride salts, then filtered and dried to obtain the washed fly ash (WFA).

2.2. Flotation methods

Fly ash sample was tested with 1 L of Denver flotation cell (RK/FD111). To determine the optimal flotation conditions, a series of tests were carried out [2]. It has been revealed nonionic surfactant Tween 80 could promote the floatation effect of the weathered fly ash under a suitable dosage of 0.15 g/kg-ash [18]. Thus, before flotation, 3.0 g/kg-ash of kerosene, 0.15 g/kg-ash of Tween 80 were added

into the slurry one by one. Meanwhile, the conditioning time was controlled as 1 and 2 min, respectively. 0.1 g/kg-ash of MIBC would be subsequently added and conditioned for 3 min when MIBC frother was needed (Table 1). After pulp conditioning, the slurry obtained was transferred to the Denver flotation cell.

Considering most of the chlorides in MWI fly ash are sodium chlorides (NaCl), NaCl with analytical grade (>99%) was selected as an added salt to prepare a chloride solution of concentration with 0.05, 0.1, 0.15 or 0.2 mol/L, which were defined as WFA-5, WFA-10, WFA-15, and WFA-20, respectively. All samples were freshly prepared. And in order to facilitate the comparison of effects of chloride salts, pulp conditioning prior to flotation was carried out under identical conditions. In this work, all the experiments were carried out at a constant airflow rate of 1.2 L/min and an impeller speed of 2000 rpm a slurry concentration of 35.6 g/L determined by reference to slurry concentration of RFA [18]. The experiments were conducted at pH 7 and 23°. In order to maintain a stable liquid level, deionized water was added to the flotation cell during the whole flotation process. Finally, the tailings and froths obtained were separately filtered, dried, and weighed. As a result, dioxins contained in the fly ash were separated and then enriched as froths product by flotation. Meanwhile, concentration of dioxins and their congeners in both products were analyzed with isotope dilution high resolution gas chromatography-high resolution mass spectrometry (HRGC-HRMS). Prior to the analysis, the samples were milled for the purpose of homogenization. According to the modified version of the US EPA Method 23 (20), the sample was pretreated. And the $^{13}\text{C}^{12}$ isotopically labeled internal standard solution was purchased from Isotope Laboratory of Cambridge. And the total toxic equivalent (TEQ) of dioxin congeners of the sample can be calculated according to the international toxic equivalency factors. Three replicates of dioxin analyses were made for obtaining reliable data and the results were determined as the average of three measurements [19]. The removal efficiency of dioxins was defined as the ratio of the amount of dioxins in the froths over their total amount in the raw ash sample. The zeta potential measurements of slurry WFA were conducted with a Zeta potential analyzer (ZEN2500, Malvern, UK).

3. Results

3.1. Effect of washing on ash characteristics

The LOI and chemical compositions of the RFA and WFA are presented in Table 2. The measurements were repeated three times and then the average value was taken. The major components of the RFA were CaO , SiO_2 , Cl , and

Table 1
The dosages of added agents and the reaction time before flotation

| Agents | Dosages (g/kg-ash) | Reaction time (min) |
|----------|--------------------|---------------------|
| Kerosene | 3 | 1 |
| Tween 80 | 0.15 | 2 |
| MIBC | 0.1 | 3 |

Table 2
The LOI and chemical composition of the RFA and WFA

| Major compounds | Raw fly ash | Washed ash |
|--------------------------------|-------------|------------|
| SiO ₂ | 14.31 | 29.75 |
| CaO | 23.55 | 38.7 |
| Al ₂ O ₃ | 3.65 | 7.59 |
| Fe ₂ O ₃ | 3.31 | 5.93 |
| MgO | 1.09 | 2.54 |
| K ₂ O | 4.58 | 1.36 |
| Na ₂ O | 17.03 | 0.82 |
| SO ₃ | 4.83 | 4.74 |
| Cl | 22.38 | 3.25 |
| TiO ₂ | 0.99 | 2.15 |
| F | 1.19 | 0.37 |
| LOI | 15.84 | 44.13 |

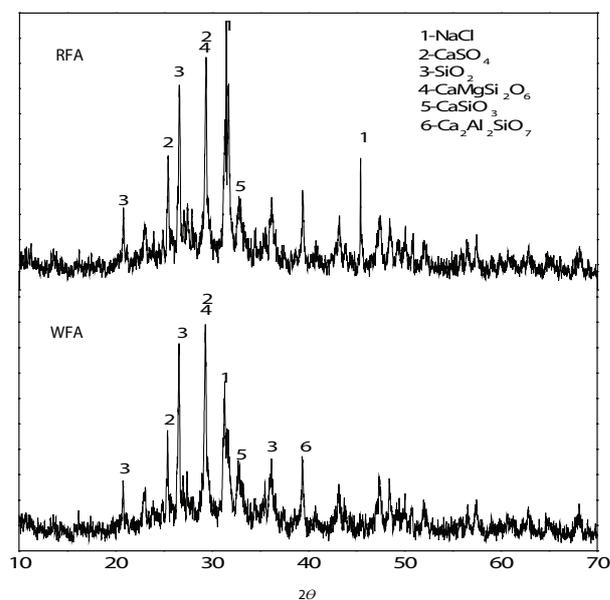


Fig. 1. X-ray diffractograms of RFA and WFA.

Na₂O, CaO, SiO₂, and Al₂O₃ emerged as major components in the WFA because these compounds were concentrated in the WFA when large amounts of chloride were removed. The results indicated that the LOI of the RFA reached 15.8%, which was due to the high content of carbonaceous matter in the RFA. Chloride content in the RFA reached 22.4% but significantly decreased to 3.3% because of the large amount of NaCl. X-ray diffractograms of the RFA and WFA are shown in Fig. 1. On comparing the two diffractograms, a weak peak for NaCl can be seen in the WFA, suggesting that quantities of NaCl in the RFA can be washed away by water. This would result in the WFA containing a relatively lower chlorine content. Similarly, the washing process of the RFA caused a 64.4% weight loss due to chloride solubilization. Carbonaceous matter remained in the solid phase, which may have caused the LOI of the WFA to increase to 44.1%.

Fig. 2 presents the concentration of each PCDD/F con-

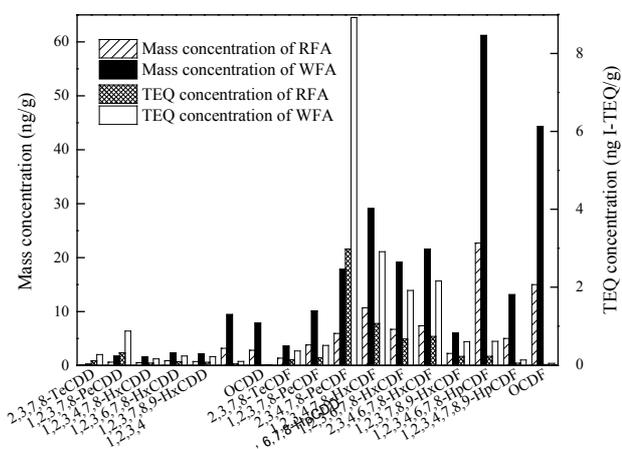


Fig. 2. Mass concentration of each PCDD/Fs congener for the RFA and WFA.

gener group for the RFA and WFA. The TEQ value of the PCDD/Fs in the RFA was 6.98 ng I-TEQ/g, which was higher than our previous reported value of 5.61 ng I-TEQ/g [1]. This difference in PCDD/F concentration may be attributed to the unstable combustion, different waste compositions, and the performance of APCDs [20]. 1,2,3,4,6,7,8-HpCDF showed the highest fraction of PCDD/F congeners, whereas a peak in total concentration was shown at OCDF. The concentration of PCDFs, particularly highly chlorinated homologs, in the RFA was greater than that of PCDDs. This might be related to the high chlorine content of the medical waste. When the chlorine content of the waste exceeded the threshold value of 0.8%–1.1%, the formation of PCDFs dominated [21]. However, 2,3,4,7,8-PeCDF gave the highest contribution of 2.98 ng I-TEQ/g, or 43% of the total TEQ of the RFA. This particular 2,3,4,7,8-PeCDF congener was the dominant contributor to TEQ, which was in accordance with other fly ash samples [22,23].

Congener distribution in the WFA was nearly the same as that in the RFA, with the first two dominant congeners being 1,2,3,4,6,7,8-HpCDF and OCDF. The total concentration of PCDD/Fs in the WFA was 251.6 ng/g and the I-TEQ concentration was 20.0 ng I-TEQ/g. Because dioxins are strongly hydrophobic and generally do not leach easily with distilled water [24], elution ratios of dioxins ranged from 0.001 to 0.01 ppm when using pure water [25]. Thus, after washing and filtration, almost all dioxins in the RFA would be separated into the WFA.

3.2. Effect of NaCl concentration on carbon removal

Almost no carbonaceous matter was removed during flotation when NaCl was not added (Fig. 3). Moreover, no bubbles were observed during flotation. Ozdemir proved that coal flotation in NaCl salt solutions was completely different from that in distilled water [13]. Carbon removal efficiency dramatically increased to 84.8% in 0.05 mol/L NaCl and gradually increased with increasing NaCl concentrations to 0.15 mol/L; at this point, the removal efficiency reached the maximum of 88.5%. Meanwhile, the LOI of the tailings decreased to 6.5%. However, an additional increase in NaCl

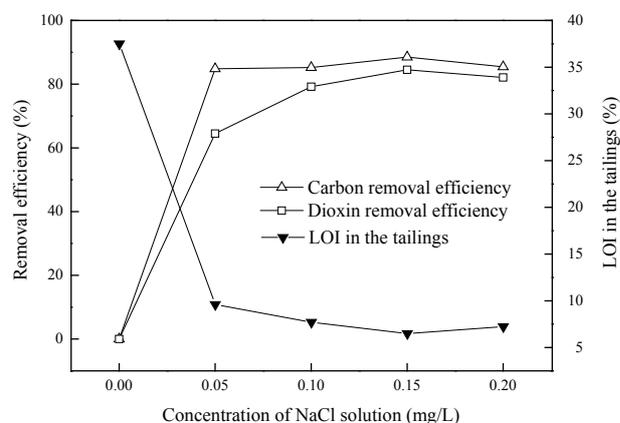


Fig. 3. Effect of NaCl concentration on removal efficiencies of carbonaceous matters, total dioxins, LOI in the tailings.

concentration to 0.2 mol/L brought significantly decreased the removal efficiency and increased the LOI of the tailings. The study results were similar to those reported by Gao for algae removal in the presence of Cl^- at a transition concentration by electro-coagulation-flotation [11,12].

The flotation behavior of carbonaceous matter in electrolytes can be explained as follows [13]: (1) salt ions destabilize the hydrated layers surrounding the carbon particles and thus reduce the surface hydration of the carbon, which enhances the attachment of the carbon particles to the bubbles [26]; (2) inorganic electrolyte solutions compress the electrical double layer on the surface of the particles and then reduce electrostatic repulsion between the bubbles and the particles [11,27]; and (3) inorganic electrolytes reduce the bubble size in the pulp phase and inhibit bubble coalescence, which may improve flotation performance [26]. Bubble coalescence can be inhibited in the presence of NaCl, KCl, or MgCl_2 [10]. Thus, to stabilize the froth zone, a relatively high salt concentration is required [27]. However, excess salt may easily decrease the hydrophobicity of coal and thus inhibit flotation performance [13,28].

The decarburization effects of the RFA with MIBC addition and of the WFA-15 with and without MIBC addition were compared (Fig. 4). For RFA flotation, 90.6% of carbon removal efficiency was achieved with a LOI of 5.7% in the tailings [18]. Compared with the results achieved for the WFA with MIBC addition, relatively high carbon removal efficiency was achieved for the RFA in this study. While the WFA contained NaCl, the RFA contained MgCl_2 and CaCl_2 in addition to NaCl. When the divalent cations Mg^{2+} and Ca^{2+} are present at certain concentrations, they might produce higher flotation recovery efficiencies than the monovalent cation Na^+ during flotation [10,14]. Harvey revealed that the effect of salt on coal recovery depended on the type of electrolyte and that MgCl_2 resulted in higher flotation recovery than NaCl [11]. A definite correlation exists among reduction of the bubble size in the pulp phase, valence of the electrolytes, and the transition concentration [23]. Furthermore, the transition concentration for each salt is quite different [10]. On comparing the WFA with and without MIBC addition, we found that MIBC did not improve the removal efficiencies significantly when a high content of NaCl was present since stable froths are formed at high chloride concentra-

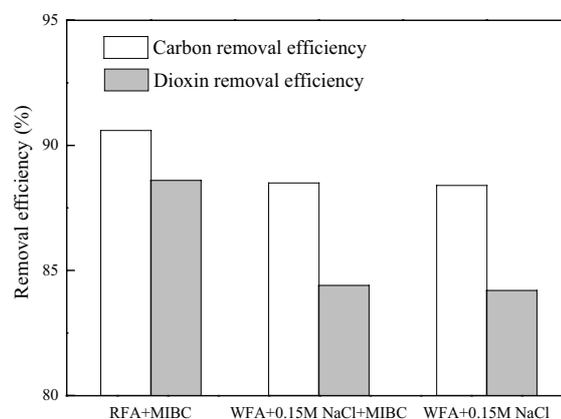


Fig. 4. Removal efficiency of carbonaceous matters and total dioxins under three different conditions.

tions. Ozdemir also proved that coal could be recovered in chloride salts without using any frothing agents [13].

3.3. Effect of NaCl concentration on Zeta potential

The Zeta potential values of the WFA slurry were negative, which was mainly attributable to the oxygenic functional groups on the surface of the fly ash, such as hydroxyl and carboxylic functional groups [29] (Fig. 5). The Zeta potential increased from the initial -6.1 mV to $+1.2$ mV with increasing NaCl concentration. These results were similar to that of Gao, who found that Zeta potential increased as Cl^- concentration increased [12]. The presence of electrolytes compressed the electrical double-layer between the bubbles and particles, which corresponded to the absolute value reduction of the Zeta potential of the slurry. The isoelectric point (IEP) of the washed ash was in the range of 0.1 to 0.15 mol/L. The flotation recovery can have a maximum below or above the IEP [13]. The resulting value of the Zeta potential is basically consistent with the highest level of carbon removal at an NaCl concentration of 0.15 mol/L.

3.4. Effect of NaCl concentration on dioxin removal

Dioxin removal performance at various NaCl concentrations is shown in Fig. 3. When NaCl was absent in the slurry, dioxin removal efficiency was nearly zero, similar to the removal efficiency of carbon. Compared with the results in distilled water, the presence of 0.05 mol/L NaCl increased dioxin removal in the WFA. Mouton also proved that the addition of NaCl could enhance PAH removal during flotation [30], implying that NaCl could not only improve carbon removal in the flotation process but also enhance the removal of dioxins. It could also be observed that the influence of NaCl on dioxin removal was similar to that on carbon. Dioxin removal efficiencies were 64.5%, 79.2%, and 84.4% with 0.05, 0.1, and 0.15 mol/L NaCl, respectively. However, an increase in NaCl concentration to 0.2 mol/L decreased the amount of dioxin removal and only 82.1% removal efficiency was observed.

Fig. 4 portrays the dioxin removal results of the RFA with addition of MIBC and of the WFA with and without addition

of MIBC. For flotation of the RFA, the removal efficiency of dioxins reached 88.6% and the dioxin content in the tailings was significantly reduced to 2.7 ng I-TEQ/g, which was satisfactory with the landfill regulation of municipal solid waste of 3ng I-TEQ/g [18]. For the WFA without MIBC, dioxin removal efficiency was 84.2%, close to the value of the WFA with MIBC.

Figs. 3 and 4 portray the changing trend of dioxin removal with the operating conditions corresponding to that of carbon removal, which indicates a relationship between them. In MWI fly ash, PAC/UC plays an important role in the partitioning of dioxins [2]. A possible explanation for the expected high dioxin removal efficiency is the relatively high sorption capacity of the PAC/UC. Therefore, the mechanism may occur as follows: (1) strong adsorption between gaseous phase dioxins and porous PAC occurs during the flue gas cleaning process of the incinerator and this strong adsorption persists in the flotation process [31]; (2) some of the dioxins originally bound to fly ash particles dissolve and enter into the aqueous phase of the slurry through surfactant-enhanced solubilization during the slurry pulp-conditioning process and then most of these dioxins are adsorbed again on sites of the PAC/UC while the sorptive-flotation processes occur simultaneously (Fig. 6); and (3) dioxins are easily adsorbed to kerosene drops with hydrophobicity because of their strong adsorptive tendency. Most of these aggregates of hydrophobic particles will tend to attach to air bubbles and float to the surface whereas a relatively low

amount may remain in the tailings or the remaining solution. Thus, the influence of NaCl concentration on dioxin removal is similar to that on carbon removal. In addition, the increase in ionic strength may increase dioxin adsorption on some adsorbents, such as PAC and UC [23].

3.5. Effect of NaCl concentration on dioxin concentration in the remaining solution

Dioxins are hydrophobic and characterized by extremely low water dissolution [32]. However, surfactants might solubilize hydrophobic compounds in aqueous solutions and increase their leachability [30]. Yasuhara found that dioxins in ash were leached more effectively by nonionic surfactant solutions than by pure water [32]. Because of the addition of surfactant, a relatively low quantity of dioxins in the RFA would remain in the remaining solution. The concentration of each dioxin congener in the remaining solution at various NaCl concentrations mimicked the increasing NaCl concentrations (Fig. 6). The patterns of distribution of the dioxin congeners in the remaining solution were similar to those in the WFA from which they were derived [32]. Compared with the WFA sample (Fig. 2), the concentrations of the highly chlorinated (hepta- and octa-) congeners in the remaining solutions were relatively higher, whereas those of the less chlorinated homologs were relatively low, particularly at high NaCl concentrations. This phenomenon might be due to the dependence of dioxin leachability on the chlorination degree of dioxins [23]. Highly chlorinated congeners were leached more easily than the less chlorinated congeners. Schramm also found that the concentration of highly chlorinated dioxins in the leaching solution was higher than that of the less chlorinated ones [33]. The effect of NaCl concentration on solubilization of each dioxin congener during flotation will be examined in a future study.

Calculated from the concentration of each dioxin congener in Fig. 7, the total mass concentration of dioxins in the remaining solutions increased from 447.0 to 675.7 ng/L when NaCl concentration increased from 0 to 0.2 mol/L, suggesting that NaCl could considerably enhance dioxin solubilization. Wang noted that the surfactant used with NaCl addition could promote the mobilization of organic compounds with relatively high lipid solubility and low water solubility [34]. Although the percentages of dioxins dissolved in the remaining solutions (approximately 5%–7.5%) are much lower than those in the froths and tailings, they should also still be degraded before final disposal through photodegradation or catalytic degradation [35].

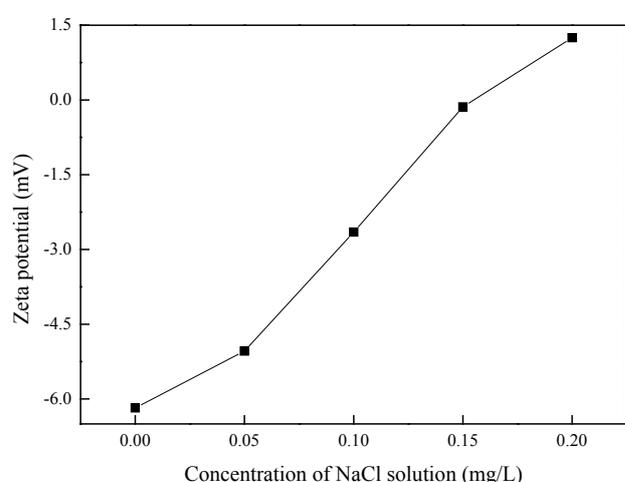


Fig. 5. Effect of concentration of NaCl concentration on Zeta potential of the slurry.

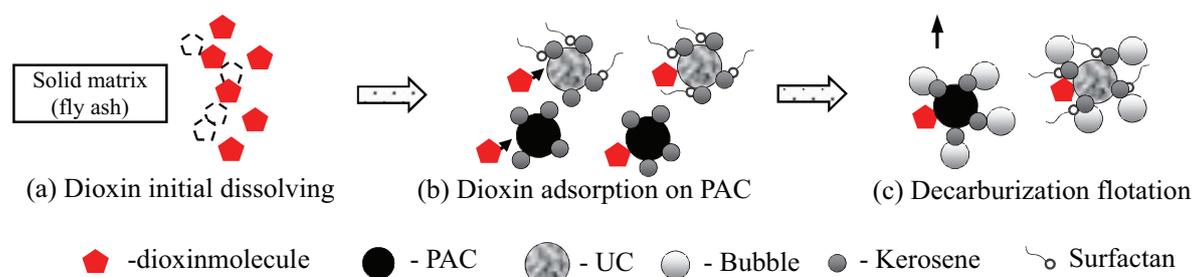


Fig. 6. Conceptual diagram of dioxin removal regarding interactions among PCDD/F molecule, PAC, UC, bubble and kerosene during flotation.

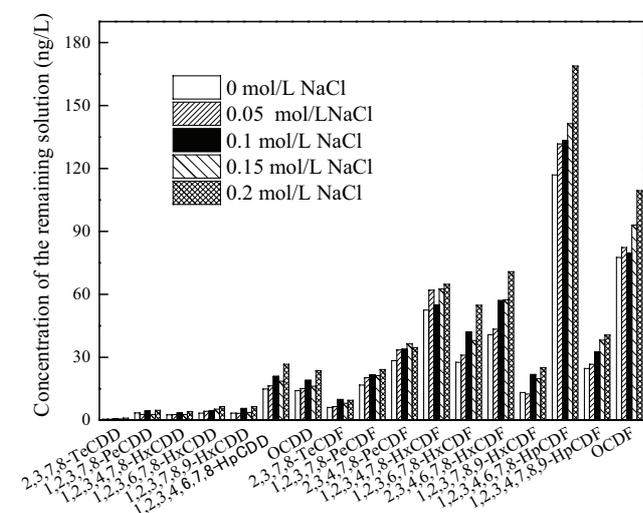


Fig. 7. Concentration of dioxin congeners in the remaining solution at different NaCl concentration.

4. Conclusions

MWI fly ash contains a high concentration of chloride, which may have a substantial influence on the performance of the flotation operation. The effect of different concentrations of NaCl solutions on the flotation behavior of the WFA was investigated in this paper. The results revealed that there was a synergistic effect when NaCl was added into the flotation system of the WFA. However, dioxins underwent a greater degree of solubilization in the presence of high concentrations of NaCl. The decarburization effects of different NaCl concentrations are validated by Zeta potential measurement results. The maximum removal efficiencies of carbon and dioxins from the WFA were 88.5% and 84.4%, respectively, and were simultaneously obtained when 0.15 mol/L NaCl was added. In addition to NaCl, Mg^{2+} and Ca^{2+} in raw fly ash might also have a positive effect on the removal process. In the presence of high NaCl content in solution, the addition of frothing agent MIBC is useless. This work confirmed that high concentrations of carbonaceous mass and dioxins enriched in the froths might result from flotation aided by the presence of chloride in MWI fly ash.

Acknowledgments

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