Dechlorination of Municipal Solid Waste Incineration Fly Ash by Leaching with Fermentation Liquid of Food Waste

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Abstract: Cement kiln collaborative disposal of municipal solid waste incineration (MSWI) fly ash (referred to as fly ash) can achieve harmless recycling of fly ash. However, because of high chlorine (Cl) content in fly ash, the practical application of this technology is seriously restricted. In order to find a suitable leaching solvent for dechlorination of fly ash, this study compared the effect of lactic acid fermentation liquid (FL) and sludge FL of food waste on dechlorination. Results show that 90% of water-insoluble Cl in fly ash can be removed by a three-step leaching process with lactic acid FL and sludge FL, and the Cl content in leached fly ash residue is 0.44% and 0.39%, respectively. According to calculation, permissible fraction of the residue after three-step leaching with addition of lactic acid FL and sludge FL in kiln is 4.28% and 4.99% higher than that of the residue after three-step leaching with pure water, respectively. Furthermore, the properties of leaching solvents after leaching experiments indicate that organic acids with low pH value and high concentration are more conducive for removal of water-insoluble Cl. Therefore, it is feasible to use two kinds of FL as leaching solvent for fly ash dechlorination.

Keywords: MSWI fly ash; dechlorination; food waste; lactic acid fermentation; sludge fermentation

1. Introduction

In recent years, the municipal solid waste incineration (MSWI) technology has developed rapidly in China because of obvious advantages of reduction, stabilisation, and resource utilisation [1,2]. As one of the by-products of MSWI, the output of incineration fly ash (accounting for about 2%–5% of waste incineration quality) is also increasing by the day [3,4]. Due to the volatilization of heavy metals in MSW during high-temperature incineration, the fly ash contains a large number of heavy metal compounds with high leaching concentration, which will cause great harm to the natural environment and human health [5,6]. Fly ash belongs to the CaO·SiO2·Al2O3·Fe2O3 system with fine particles and large specific surface area. It can be used as alternative raw material for cement
production [7,8]. However, high chlorine (Cl) content in fly ash is a bottleneck if it becomes a raw material of Portland cement [9–12]. Fly ash with high Cl content calcined directly in a cement kiln easily causes high-temperature corrosion, crusting, and blocking of the kiln. Too much Cl content in cement easily causes corrosion of reinforced concrete [13,14]. Therefore, Chinese standard HJ662-2013 (technical code for environmental protection of collaborative disposal of solid waste by cement kiln) clearly stipulates that Cl content in raw materials entering a kiln shall not be more than 0.04% (mass metre). Therefore, before using fly ash as a cement raw material, it is necessary to reduce its Cl content as much as possible.

The Cl content in fly ash can be divided into water-soluble Cl (i.e., NaCl, KCl, and CaCl$_2$ [15,16]) and water-insoluble Cl (i.e., Friedel’s salt, AlOCl [15,17]). At present, the water leaching pretreatment technology is one of the main methods for fly ash dechlorination, and the rate of Cl removal from fly ash can reach 90% [18,19]. However, because of the existence of water-insoluble Cl in fly ash even after multi-step water leaching, the Cl content in leached fly ash residue is still 1%–4% [20–22], which greatly limits the proportion of fly ash in the materials entering the kiln. In the current situation of fly ash production increasing, it is difficult to realise ‘absorption treatment’ of fly ash in a true sense. In addition, some studies have shown that the leaching effect of acid solvent on Cl in fly ash is significantly better than that of water, which can effectively remove water-insoluble Cl from fly ash [23,24], but if pure acid is directly used for leaching, the cost will be greatly increased. Therefore, it is of great significance to develop a highly economical dechlorination technology.

Food waste has the characteristics of high moisture content and high content of available organic matter containing. It is an ideal material for anaerobic fermentation to produce organic acids [25]. If food waste, which is a kind of solid waste, can be treated together with fly ash, not only can the consumption of water in the process of fly ash dechlorination be reduced, but also the pH value of fermentation liquid (FL) of food waste will be lower, which is expected to remove water-insoluble Cl from fly ash [26,27]. Therefore, this study compares the effect of FL that is formed by inoculation of different inoculums on the removal of Cl from fly ash with the effect of dechlorination in which different steps of leaching are used and explores the feasibility and advantages of deep dechlorination of fly ash by FL, which provided technical support in the application of collaborative disposal technology for fly ash and food waste.

2. Materials and Methods

2.1. Experimental Materials

The fly ash samples used in this study were collected from the discharge port of a bag filter from the same batch in a MSWI power plant in Beijing. After being transported to a laboratory, the fly ash was dried, ground, and homogenised, and then sealed with air. The basic properties of fly ash are shown in Table 1.

| Properties          | Unit     | Value  |
|---------------------|----------|--------|
| pH                  | -        | 12.8   |
| BET                 | m$^2$/kg | 912.8  |
| Total Cl            | g/(g fly ash) | 0.237 |
| Water-soluble Cl    | g/(g fly ash) | 0.207 |
| Water-insoluble Cl  | g/(g fly ash) | 0.030 |

Food waste for the experiment was taken from a canteen, and generated by the three meals of breakfast, lunch, and dinner in the canteen, which was evenly mixed with equal quality ratio. The bones, sawdust, and sundries that cannot be fermented from the food waste were removed, and then crushed with a grinder. The food waste was placed inside a −20 °C refrigerator for sealed storage, and before the experiment, it was placed inside a 4 °C refrigerator for thawing. The basic properties of
food waste are shown in Table 2. The total Cl and water-soluble Cl in the food waste (dry weight) after determination were 7.5 and 6.8 mg/g, respectively.

Table 2. Physicochemical properties of food waste.

| Properties      | Value          |
|-----------------|----------------|
| TS (%)          | 22.6 ± 0.05    |
| VS (%)          | 22.2 ± 0.06    |
| Fat* (%)        | 30.82 ± 0.51   |
| Protein* (%)    | 14.51 ± 0.07   |
| Carbohydrate* (%) | 44.81 ± 0.03  |
| C* (%)          | 48.98 ± 0.04   |
| H* (%)          | 7.15 ± 0.15    |
| O* (%)          | 30.26 ± 0.17   |

* The percentages listed in the table are in dry matter.

The anaerobic sludge for the experiment was taken from a biogas station in Beijing. After 5 days of sedimentation, the anaerobic sludge was drained of supernatant, and then the substrate was cultured at 37 °C of constant temperature. A certain amount of food waste was added into the substrate every day for domestication, and the inoculated sludge required for the experiment was obtained after continuous cultivation for 1 month [28].

2.2. Experimental Method

2.2.1. Fermentation Experiment of Food Waste

The experimental method for food waste fermentation by inoculation of anaerobic sludge (referred to as sludge fermentation group) was as follows: Food waste and anaerobic sludge were mixed in a ratio of 4:1.

The experimental method for food waste fermentation by inoculation of Lactobacillus casei (referred to as lactic acid fermentation group) was as follows: In this experiment, MRS (i.e., it is a kind of microbial culture medium, which can be used for the culture of Lactobacillus) broth was used as culture medium. A certain amount of bacterial solvent was added into the new MRS broth with 10% inoculation amount, and cultured for 12 h at 37 °C. The bacteria solvent after the cultivation was inoculated into the food waste according to a 10% inoculation ratio.

The sludge fermentation group and the lactic acid fermentation group were fermented. Fermentation temperature was 37 °C. Anaerobic fermentation lasted for 4 days. Supernatant was taken after centrifugation. Type and concentration of organic acids were determined by HPLC, as shown in Table 3.

Table 3. Organic acid content of fermentation liquid of food waste.

| Type             | Concentration (g/L) | Lactic Acid Fermentation Group | Sludge Fermentation Group |
|------------------|---------------------|--------------------------------|---------------------------|
| Lactic acid      | 55.6                | 0.7                            |                           |
| Formic acid      | 1.5                 | 0                              |                           |
| Acetic acid      | 2.1                 | 11.6                           |                           |
| Propionic acid   | 0                   | 0.6                            |                           |
| N-butyric acid   | 0                   | 14.7                           |                           |
2.2.2. Dechlorination Experiment of Fly Ash

In the pre-experiment, the best experimental conditions for dechlorination of fly ash by water leaching were investigated by changing the solid–liquid ratio and leaching time. In addition, the leaching effect of lactic acid FL and sludge FL on Cl in fly ash was investigated. Under 2 h total leaching time, 1:10 solid–liquid ratio, and leaching 25 °C temperature conditions, the same volume of leaching solvent was added to fly ash two, three, and five times (also known as two-step, three-step, and five-step leaching) to investigate the influence of the number of leaching times on dechlorination effect and make comparisons with one-step leaching. The experimental scheme is shown in Table 4. One-step leaching involved adding 100 mL FL to leach the fly ash, whereas two-step leaching involved dividing 100 mL of leaching solvent into two to leach the fly ash, each step with 50 mL, and so on.

The experimental method was as follows: 10 g of fly ash was weighed and placed in a 500 mL conical flask. Different amounts of leaching solvent were added step by step and put into a shaker. After shaking at 200 rpm for different times at 25 °C, it was taken out and centrifuged at 4000 rpm for 15 min. The supernatant was filtered after centrifugation with a 0.45 µm nylon filter membrane, and chloride concentration was measured. The remaining solid matter (referred to as residue) was dried in the centrifuge tube at 105 °C to a constant weight, and then used to detect its physical and chemical properties. Two parallel groups were set for each experimental condition. The average of the test indicators was taken into account for the final evaluation.

Table 4. Experimental design.

| Leaching Steps | Fly Ash (g) | Leaching Time of Each Step (h) | Leaching Solvents Volume of Each Step (mL) |
|----------------|-------------|-------------------------------|------------------------------------------|
| One-step       | 10          | 2                             | 100                                      |
| Two-step       | 10          | 1                             | 50                                       |
| Three-step     | 10          | 0.67                          | 33                                       |
| Five-step      | 10          | 0.4                           | 20                                       |

2.3. Analysis Method

The pH value was measured according to the Chinese soil pH measurement standard (solid–liquid ratio 1:2.5, stirring for 30 min). The BET was measured according to the method of determination for specific surface of cement (GB/T 8074-1987). After pretreatment of fly ash with JIS A1154-2003, total Cl content was determined by intelligent ion chromatography (YC3000, Qingdao Allen Chromatography Technology Co., Ltd.) and converted to soil mass concentration. Water-soluble Cl is determined by the method provided in appendix F of GB5085.3-2007 (identification standard for hazardous waste). Organic acid content in the FL was determined by HPLC (LC-20AT, Shimadzu International Trade Shanghai Co., Ltd). Relevant physical and chemical indexes (pH value and conductivity) of leaching solvents after leaching experiments were determined by pH meter (pHSJ-3F, Shanghai Yidian Science Instrument Co., Ltd.).

3. Results and Discussion

3.1. Discussion on Best Experimental Conditions for Dechlorination of Fly Ash by Pure Water Leaching

Previous studies of our research group have shown that the dechlorination effect on fly ash leaching is affected by many factors such as temperature, solid–liquid ratio, and leaching time [3,29,30]. Therefore, the pre-experiment of this study adopted the control variable method to simulate the leaching process of Cl in fly ash by shaking bottle experiment under 25 °C temperature conditions and comparing the dechlorination effect of different solid–liquid ratios and leaching times of water leaching on fly ash, as shown in Figure 1.
Figure 1. Dechlorination effect of pure water leaching on fly ash under different experimental conditions: (a) Solid–liquid ratio; (b) leaching time.

The dechlorination effect of pure water on fly ash was studied under solid–liquid ratio 1:2–1:20 and leaching time of 2 h. As shown in Figure 1a, the Cl removal percentage increased with the increase in solid–liquid ratio, but when the solid–liquid ratio exceeded 1:15, the Cl removal percentage would not increase any more, which may be because most of the water-soluble Cl salt in fly ash is completely dissolved at this time, and only a small amount of water-insoluble Cl is left in the fly ash. Therefore, to further improve the dechlorination efficiency, the key is whether water-insoluble Cl can be leached. It should be noted that when the solid–liquid ratio increased from 1:10 to 1:15, pure water consumption increased by one-third, but the Cl removal percentage only increased by 2.95% (from 81.40% to 84.35%). Therefore, from the perspective of water conservation, 1:10 was selected as an optimal solid–liquid ratio for this study.

Under solid–liquid ratio 1:10 conditions, fly ash was leached with pure water for 8 h and samples were taken for analysis regularly. It can be found that with increase in leaching time, Cl removal percentage increased gradually and then tended to be stable (Figure 1b). In the first 0.5 h, the Cl removal percentage was 79.07%, which is mainly because water-soluble types of Cl such as KCl and NaCl in fly ash can be quickly dissolved in water [31]. In a period of 0.5–2 h, growth was 3.91%. After 2 h, leaching was basically finished. Therefore, 2 h was selected as optimal leaching time for this study. The following leaching experiments with pure water and FL were conducted under a leaching temperature of 25 °C, a solid–liquid ratio of 1:10, and a leaching time of 2 h.

3.2. Dechlorination Effect of Different Leaching Solvents on Fly Ash

Because of the existence of water-insoluble Cl, the Cl removal percentage could only reach 82.98%, and 17.02% of total Cl remained in leached fly ash residue (i.e., the Cl content in leached fly ash residue was 4.03%), which is still not ideal as additional material for cement kiln. Therefore, on the basis of the above-mentioned dechlorination conditions for fly ash, one-step leaching experiment for dechlorination of fly ash was conducted with sludge FL and lactic acid FL as described in 2.2.2 to remove the remaining water-insoluble Cl from fly ash in depth. The dechlorination effect is shown in Figure 2. It can be seen from Figure 2 that when lactic acid FL, sludge FL, and pure water were used as leaching solvents, the leached Cl mass from each gram of fly ash was 0.220, 0.219, and 0.206 g, respectively, and the leached Cl mass accounted for 92.83%, 92.41%, and 86.92% of total Cl mass, respectively. The addition of FL of food waste can significantly improve the dechlorination effect on fly ash, with 43.33% and 40.00% of water-insoluble Cl in fly ash removed using lactic acid FL and sludge FL as leaching solvents. Compared with pure water leaching, the removal of water-insoluble Cl from fly ash by FL may be related to the physical and chemical properties of FL (acid ion, pH value etc.). In addition, it has been pointed out that high concentration of acids can destroy the internal...
structure of water-insoluble Cl (such as AlOCl) to a certain extent, thus releasing the Cl bound by a strong covalent bond and then becoming water soluble [32], which also explains the results of this study to a certain extent.

![Figure 2. Dechlorination effect of different leaching solvents on fly ash with one-step leaching. 'FL' refers to fermentation liquid.](image)

3.3. Dechlorination Effect of Different Leaching Steps on Fly Ash

In order to further improve the dechlorination effect of FL on fly ash, lactic acid FL and sludge FL were used as leaching solvents in this study. The results of leaching are shown in Figure 3.

![Figure 3. Dechlorination effect of different leaching steps with different leaching solvent: (a) Lactic acid fermentation liquid; (b) sludge fermentation liquid.](image)

As shown in Figure 3a, when lactic acid FL was used as leaching solvent, the leached Cl mass from each gram of fly ash was 0.220, 0.228, 0.234, and 0.235 g, respectively, with one-step, two-step, three-step, and five-step leaching, and the leached Cl mass accounted for 92.83%, 96.20%, 98.73%, and 99.16% of total Cl mass, respectively. Compared with one-step leaching, the water-insoluble Cl leached from the fly ash increased by 26.67%, 46.67%, and 50.00% with two-step, three-step, and five-step leaching, respectively. From the comparison of different leaching steps, increasing the number of leaching steps had a significant effect on the removal of Cl from fly ash. This is mainly because one-step
leaching can ensure high solid–liquid ratio (1:10), but with the reaction going on, FL constantly reacts with alkaline substances in the fly ash, which consumes the acid substances and weakens the acid in FL, so it is difficult to leach water-insoluble Cl. Also, compared with one-step leaching, the solid–liquid ratio in each step of multi-step leaching was lower (Table 4), but it could guarantee complete leaching of water-soluble Cl in the first few steps, which provides an acidic environment for subsequent leaching. The acid ions in FL can exchange with water-insoluble Cl in the fly ash, and H⁺ can destroy the internal structure of fly ash, so water-insoluble Cl can be dissolved [32]. Multi-step leaching can make full use of the acid added with each step and then improve the leached Cl mass from fly ash.

Similarly, as shown in Figure 3b, when sludge FL was used as leaching solvent, the overall trend of dechlorination effect on fly ash with different leaching steps was the same as that of lactic acid FL. That is to say, both of them could be used to replace pure water for deep dechlorination of fly ash.

3.4. Cl Content in Leached Fly Ash Residue

After the fly ash was leached by leaching solvents, Cl content in the residue (i.e., percentage of Cl mass in total solid mass) is shown in Figure 4. The Cl content in raw fly ash was 23.70%, and the Cl content in the residue decreased with an increase in the number of leaching steps.

![Figure 4](image_url)

**Figure 4.** Cl content in residue after leaching with different leaching solvent: (a) Lactic acid fermentation liquid; (b) sludge fermentation liquid. ‘Pure water’ refers to three-step pure water leaching, which is the control group.

By the following, this study compares different leaching conditions and analyzes the feasibility of the residue as one of the raw materials in cement kiln. In 6.6.8 of Chinese standard HJ662-2013 (technical code for environmental protection of cement kiln collaborative disposal of solid waste), it is clearly stipulated that Cl content in materials entering a kiln shall not be more than 0.04% (mass metre). In this study, it is assumed that the Cl content in conventional raw materials (limestone, clay, etc.) is 0.02%, and the calculation formula is as follows:

\[
C = \frac{\text{Total CI content in materials (fly ash and conventional raw material) entering the kiln}}{\text{Total material entering the kiln}}
\]

\[
= \frac{C(\text{fly ash}) \times m(\text{fly ash}) + C(\text{conventional raw material}) \times m(\text{conventional raw material})}{m(\text{fly ash}) + m(\text{conventional raw material})}
\]

C: Cl content in total raw materials entering kiln, %; C (fly ash) and C (conventional raw material): Cl content in fly ash and conventional raw materials, %; m (fly ash) and m (conventional raw material): dosage of fly ash and conventional raw materials in unit time, kg/h.
According to the above calculation formula, the proportion of residue added into cement kiln after each step of leaching is calculated (Table 5).

| Leaching Steps | Pure water | Lactic acid fermentation liquid | Sludge fermentation liquid |
|----------------|------------|---------------------------------|----------------------------|
|                | Three-step | One-step                        | One-step                   |
| The Cl Content in the Residue (%) | 2.81 | 1.88 | 2.33 |
| The water-Insoluble Cl Removal Percentage (%) | 6.68 | 43.33 | 40.00 |
| Permissible Added Fraction of Leaching Residue (%) | 0.72 | 1.09 | 0.87 |
| Increase in the Permissible Added Fraction of Leaching Residue (%) | - | 0.37 | 0.15 |

* In the light of the HJ 662 standard, the permissible added fraction of leaching residue was obtained by assuming that the Cl content in conventional raw materials was 0.02%; ** refers to the increase of permissible added fraction of leaching residue compared with three-step pure water leaching group.

Taking lactic acid fermentation group as an example, three-step leaching can significantly improve the leaching of water-insoluble Cl from fly ash compared with two-step leaching and reduce total Cl content in residue by 0.74%. However, when the number of leaching steps is increased from three to five, Cl content in the residue does not decrease significantly. From the point of view of easy operation and energy saving, if the residue after three-step leaching is applied to the collaborative disposal of cement kiln, permissible added fraction of leaching residue is 5.00%, which is 4.28% higher than that of three-step pure water leaching.

According to Tables 3 and 5, the types and concentrations of organic acids in the two kinds of FL are different, but their capacity to remove water-insoluble Cl from fly ash is similar. Therefore, the factors that play a major role in dechlorination with FLs need to be further studied.

### 3.5. Change in pH Value and Conductivity of Leaching Solvents after Leaching Experiments

It can be seen from the previous analysis that there are obvious differences between dechlorination effect of pure water and two kinds of FLs on fly ash, which may be mainly due to the presence of H$^+$ and acid ions in the FLs. pH value and conductivity can well show the change in properties of the solvent after each step of leaching. Therefore, this study further discusses the change in pH value and conductivity of the leaching solvents after the leaching experiments. It can be seen from Figure 5 that the pH value of FL after the leaching experiments is significantly lower than that of pure water, and all of them show a downward trend with the increase in number of leaching steps. The pH value of the leaching solvents after leaching experiments is about 7 after five-step leaching, which indicates that the alkaline substances in fly ash are completely neutralised by H$^+$. This result is consistent with the result in 2.3 of this study, which states that low pH value is more conducive to leaching of water-insoluble Cl from fly ash.

In addition, the results for conductivity showed that the conductivity of original lactic acid FL and sludge FL is 151.9 and 153.7 mS/cm, respectively. With increase in the number of leaching steps, the conductivity of leaching solvents after leaching experiments decreased and is lower than that of pure water. This may be due to the large specific surface area of fly ash (Table 1), which is a good adsorbent [33]. A large number of acid ions in FL are adsorbed on the surface of fly ash, which makes the ion concentration decrease and then leads to decrease in conductivity. Although the dissolved heavy metal can also increase conductivity [34], its effect is lower than that of adsorption, so the conductivity of the leaching solvents after the leaching experiments shows a downward trend.
Figure 5. Change in pH value and conductivity of leaching solvents before and after leaching experiments. ‘FL’ refers to fermentation liquid.

3.6. Potential Disposal Scheme for the Leaching Solvents after Leaching Experiments

This study takes fly ash dechlorination as the main line, and the follow-up research will focus on the removal of heavy metals. From the change of conductivity in Section 3.5 and other research results, it can be seen that organic acids in FL can also remove heavy metals in fly ash. Therefore, not only is the concentration of heavy metals and Cl high, but also some organic matters are contained in the leaching solvents after the leaching experiments, which also becomes the last difficulty of the application of the technology. In the current research, we propose a potential disposal scheme. The heavy metals can be removed in the form of precipitation by introducing carbon dioxide or carbonate ion into the leaching solvent. The chloride ion in the leaching solvent without heavy metal is converted into Cl₂ by electrolysis to achieve the purpose of removal. After the above two-step treatment, the water quality indicators shall be tested and further treatment shall be conducted according to the relevant discharge standards. The feasibility of the disposal scheme will also be the focus of the next research.

4. Conclusions

In this study, dechlorination of MSWI fly ash is studied with pure water and both lactic acid and sludge FL in food waste. Results show that under a leaching of temperature 25 °C, a solid–liquid ratio of 1:10, and a leaching time of 2 h, the dechlorination effect of the two kinds of FL on fly ash was better than that of pure water. During one-step leaching, 43.33% and 40.00% of water-insoluble Cl in fly ash are removed by lactic acid FL and sludge FL, respectively, and Cl content in the leached fly ash residue after three-step leaching is 0.44% and 0.39%, respectively. As one of the raw materials in cement kiln, the permissible added fraction of the fly ash residue after leaching with lactic acid FL and sludge FL is 5.00% and 5.71%, respectively, which is 4.28% and 4.99% higher compared with three-step pure water leaching. In addition, the low pH value and high concentration of organic acids are more conducive to leaching of water-insoluble Cl from fly ash. To sum up, the leaching effect on water-insoluble Cl in fly ash can be significantly improved by leaching the fly ash with lactic acid FL and sludge FL of food waste, which shows that it is an economical and feasible technology for collaborative disposal of food waste and dechlorination of fly ash.

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