Effect of Ca(OH)$_2$ on the Release Characteristics of HCl during Sludge Combustion

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Cite This: ACS Omega 2020, 5, 27197−27203

ABSTRACT: With the addition of Ca(OH)$_2$, the effects of combustion temperature, moisture, sludge particle size, and chlorine-containing additives on the removal of HCl during sludge combustion were studied. The experimental results showed that combustion temperature and moisture content promoted the formation of HCl and Ca(OH)$_2$ played a key role in the formation of HCl during sludge combustion. Under the best conditions of a sludge particle size of 380−250 μm, moisture content of 5%, temperature of 850 °C, and Ca(OH)$_2$/sludge weight ratio of 3/10, the HCl capture efficiency was 79.81%. In addition, the effect of PVC on the production of HCl was greater than that of NaCl, probably because the lattice energy of NaCl was much higher, indicating that inorganic chlorine was not the main source of HCl. Ca(OH)$_2$ can effectively inhibit the formation of HCl, which had practical guiding significance for the formation of HCl during the sludge combustion, especially the sludge containing chlorine.

1. INTRODUCTION

Sewage sludge can come from the domestic sewage treatment process,¹,² and the average annual growth rate of China’s total sludge production was 13% during 2007−2013 years.³ Because of the large amount of energy required, the cost of sludge treatment accounts for approximately 50% of the total operating cost of the sewage treatment plant.⁴,⁵ The disposal and recycling of sludge have attracted much attention because of its serious risk of negative ecological and environmental impacts.⁶−⁸ There are many disposal methods for sludge disposal,⁹−¹¹ and combustion is gaining more and more attention because of its unique advantages.¹²,¹³ Although sludge combustion has many advantages, the problem of flue gas pollution caused by combustion has always been a concern, especially dioxins (PCDD/Fs) and chlorobenzenes (CBs, dioxin precursors). The discharge of toxic pollutants is a bottleneck that restricts the popularization and application of sludge combustion and disposal technologies.

HCl can generate Cl$_2$ through the Deacon reaction during the waste combustion process, which provides chlorine sources for dioxin-like organic pollutants and forms secondary pollution.¹⁴ Therefore, it is important to understand the distribution of chlorine in the sludge combustion process. At present, the use of calcium-based and aluminum-based adsorbents for combustion dechlorination has become an economical and practical method of pollution control. Domestic and foreign scholars have conducted extensive research on the emission and removal of HCl gas during the injection combustion process, but there are relatively few studies on sludge combustion. Weinell et al. found that CaO had the strongest ability to remove HCl in a fixed-bed reactor between 500 and 600 °C.¹⁵ Zhang et al. studied various emission characteristics during the burning of bituminous coal and pickling sludge and found that the conversion rate of fuel Cl to HCl was less than 40% because of the short residence time in the reactor.¹⁶ Estelle et al. found that combustion temperature was the main factor affecting HCl emissions.¹⁷ Wang et al. found that the chlorine content and air flow rate of the sample during the combustion process had a relatively little effect on the distribution of chlorine.¹⁸ Zhang et al. found that about 99% of Cl could be captured when proper emission control was implemented during the waste combustion.¹⁹ During the combustion process, chlorine is mainly released into the flue gas in the forms of HCl, Cl$_2$, NaCl, and so on and finally captured in the fly ash through physical condensation or chemical reaction.²⁰ Most of the inorganic chlorine in the fuel is finally captured in the slag, and different heat treatment
processes have different effects on the volatile ability of inorganic chlorine, generally gasification process > incineration process > pyrolysis process. Further research found that the amount of HCl released by solid-waste combustion increased with the increase in combustion temperature, residence time, and oxygen concentration, and it is mainly derived from organic chlorine in solid waste (accounting for 72~80%). Organic and inorganic chloride sources release chlorine at high temperatures and then form HCl with the hydrogen ion. During the combustion process, some HCl can be released from the chlorinated hydrocarbons in the organic waste between 200 and 360 °C and it releases completely at 400 °C. The depolymerization and thermal decomposition of plastics are the basic pathways for the formation of HCl. The chlorine-containing components in the inorganic waste will tend to volatilize into the gas phase. In addition to the observed HCl, large amounts of NaCl and KCl are also found in the incinerator.

The study found that the addition of suitable additives during solid-waste combustion can effectively reduce the concentration of dioxins, but its inhibitory effect on HCl is still unclear. Therefore, we explored the inhibitory effect of Ca(OH)2 on the formation of HCl, focusing on the inorganic chlorine in solid waste. During solid-waste combustion can effectively reduce the concentration of dioxins, but its inhibitory effect on HCl is still unclear. Therefore, we explored the inhibitory effect of Ca(OH)2 on the formation of HCl, focusing on the inorganic chlorine in solid waste. The organic chlorine and inorganic chlorine in the sludge were measured by the water washing extraction method. The HCl was determined by ion chromatography (IC, Thermo ICS-1100, United States) to be measured. The chemical composition was tested using an X-ray fluorescence spectrometer (XRF, NaU-NDA 200, USA), and the calculation formula for defining the capture efficiency of HCl is as follows

$$\eta = \left(1 - \frac{\alpha}{\beta}\right) \times 100\%$$

where \(\eta\) is the capture efficiency, \(\alpha\) is the concentration of HCl after adding Ca(OH)2, and \(\beta\) is the concentration of HCl without adding Ca(OH)2.

3. RESULTS AND DISCUSSION

3.1. Physical and Chemical Properties of Sewage Sludge

Tables 1, 2, and 3 are the physical and chemical properties of the sludge. Table 1 shows that the sludge has a lower hydrogen-to-carbon ratio (H/C) and oxygen-to-carbon ratio (O/C), indicating that its thermal efficiency is high. The ignition performance of the fuel is related to the sum of volatiles and fixed carbon. The higher the sum of volatiles and fixed carbon, the better the ignition performance of the given fuel. The sum of the volatile matter and fixed carbon content of sludge is 59.63%, indicating that the ignition performance of the sludge is poor.

3.2. Effect of Added Ca(OH)2 Weight.

The sludge combustion experiments were all carried out in an air atmosphere, the flow rate was controlled at 5 L/min, and the sludge weight was 2 g. Figure 2a,b shows the effect of added Ca(OH)2 weight on the amount of HCl production and HCl capture efficiency. The added amount of Ca(OH)2 was 10, 20, 30, and 40% of the weight of sludge. It can be seen

| Added Ca(OH)2 Weight | HCl Production (wt %) | Capture Efficiency (%) |
|----------------------|-----------------------|------------------------|
| 0                    | 42.3                  | 65.2                   |
| 10                   | 45.7                  | 72.8                   |
| 20                   | 48.2                  | 79.5                   |
| 30                   | 50.5                  | 84.2                   |
| 40                   | 52.8                  | 89.0                   |

Figure 1. Diagram of the experimental setup.

Table 1. Ultimate and Proximate Analysis (wt %)

| Sludge Samples | C     | H     | O     | N     | S     | Moisture | Volatile | Ash    | Fixed Carbon |
|----------------|-------|-------|-------|-------|-------|----------|----------|--------|-------------|
|                | 17.60 | 3.23  | 18.369| 2.88  | 0.54  | 2.81     | 56.83    | 37.56  | 2.80         |

Table 2. Chlorine Content and Calorific Value Analysis

| Sludge Samples | Total Chlorine (wt %) | Organic Chlorine (wt %) | Inorganic Chlorine (wt %) | Calorific Value (J/kg) |
|----------------|-----------------------|-------------------------|--------------------------|------------------------|
|                | 0.110                 | 0.088                   | 0.022                    | 8849                   |
from Figure 2a that as the amount of Ca(OH)₂ increases, the amount of HCl produced gradually decreases. When the amount of Ca(OH)₂ added increases from 10 to 40%, the amount of HCl released decreases from 8.250 to 4.800 mg/m³. With the progression of sulfation, the CaSO₄ layer on the surface of the calcium hydroxide particles became thicker and diffusion into the particle became more difficult. The CaSO₄ layer may prevent the release of chlorine from CaCl₂, resulting in a reduction in the production of HCl.

In addition, the reduction in dechlorination efficiency may be due to the interference of the gap and the competition for active sites between SO₂ and HCl. However, when more Ca(OH)₂ was added, this competitive effect did not seem to exist. Figure 2b shows that the capture efficiency of HCl production increases gradually with the increase in Ca(OH)₂. The capture efficiencies of HCl production are 39.99, 50.48, 62.15, and 67.45% when the Ca(OH)₂ addition is 10, 20, 30, and 40% of the weight of sludge, respectively. At first, the capture efficiency increases fast with the increase in Ca(OH)₂ and then slows down, mainly because the reaction of Ca(OH)₂ with HCl is controlled by chemical reaction at the beginning. As the reaction progresses, the product layer may be gradually formed on the surface of the Ca(OH)₂ particles and then, it is controlled by the chemical reaction and the diffusion of the product layer. When all particle surfaces are covered by the product layer, the reaction is completely controlled by diffusion. The rate of the whole reaction has a close relationship with the diffusion rate of HCl gas in the product layer and a low relationship with the increase in Ca(OH)₂ when the amount of Ca(OH)₂ reaches 30%. Considering the practical engineering application, 30% is selected as the optimal dosage of Ca(OH)₂.

3.3. Effect of Combustion Temperature. Figure 3a,b shows the effect of combustion temperature on the amount of HCl production and capture efficiency. Figure 3a shows that the production amount of HCl increases from 1.303 to 7.531 mg/m³ when the combustion temperature increases from 750 to 950 °C. The capture efficiency of HCl production increases from 18.87% to 35.46% when the combustion temperature increases from 750 to 950 °C. Considering the practical engineering application, 900 °C is selected as the optimal combustion temperature.
mg/m³ when the temperature increases from 750 to 950 °C, indicating that the temperature has a promoting effect on the production of HCl, which is consistent with previous reports that calcium-based additives have a low ability to capture HCl at high temperatures.34−36

It can be seen from Figure 3b that the capture efficiency of HCl gradually decreases with the increase in the temperature with the corresponding capture efficiency of 90.52, 87.51, 79.81, 62.15, and 45.22% at temperatures of 750, 800, 850, 900, and 950 °C, respectively. This is mainly because the temperature increase can accelerate the burning rate of sewage sludge, making the combustion process more thorough and the chlorine release more thorough. The increase in the degree of HCl desorption from CaCl₂ at high temperatures led to the low capacity of the additives,37,38 which may be because high temperature reduces the stability of CaCl₂.39 Therefore, during the chlorination process at 850 °C, the molten phase covers the surface of the absorbent particles so that the melting temperature of the system is higher than that of the pure CaCl₂ and CaCl₂−CaO systems.40 The diffusion resistance of HCl gas molecules in contact with the fresh adsorbent increases gradually because of the increase in the molten product layer and the poor porosity.41 CaClO as an intermediate product of chlorination can effectively shorten the reaction time.42,43 High temperature can not only reduce the reaction rate of CaClO formation but also increase the Gibbs energy of the reaction.41 At the same time, the formation rate of CaClO and CaCl₂ reaches the maximum at a moderate temperature.41 Considering that the combustion temperature in actual projects is between 800 and 900 °C, the temperature of the subsequent sludge combustion experiment was chosen as 850 °C.

3.4. Effect of Sludge Moisture Content. Figure 4a,b shows the effect of moisture content on the amount of HCl production and HCl capture efficiency. Figure 4a shows that as the moisture content increases, the production of HCl shows a gradually increasing trend. When the moisture content of the sludge increases from 5 to 50%, the amount of HCl increases from 2.776 to 5.091 mg/m³. It can be seen from Figure 4b that with the increase in the sludge moisture content, the HCl capture efficiency gradually decreases from 79.81 to 62.87%, indicating that moisture content promotes the production of
HCl. This is mainly because the participation of water vapor accelerates the chlorine production reaction and transfer rate. First, the participation of water vapor promotes the release of chlorine in high-molecular organics, which is attributed to the certain solubility of water vapor in the sludge. Second, water vapor participates in the substitution reaction of chlorine in the polymer organic structure and can also produce $-\text{OH}$ at a certain temperature. Furthermore, $-\text{OH}$ replaced $-\text{Cl}$ bound in the sludge polymer organic matter in the reaction, and the substituted $-\text{Cl}$ combined with $-\text{H}$ to form HCl in the gas phase. In addition, water vapor increases the number of pores and the specific surface area of the reaction in the polymer organic reaction, thereby increasing the release rate of chlorine. As a hygroscopic salt, $\text{CaCl}_2$ may react differently with water to form hydrates. The specific reactions are as follows (eqs 1−3):44

$$\text{CaCl}_2(s) + \text{H}_2\text{O} \rightarrow \text{CaCl}_2\cdot\text{H}_2\text{O}(s) \quad (1)$$

$$\text{CaCl}_2(s) + 2\text{H}_2\text{O} \rightarrow \text{CaCl}_2\cdot2\text{H}_2\text{O}(s) \quad (2)$$

$$\text{CaCl}_2\cdot2\text{H}_2\text{O}(s) \xrightarrow{\text{H}_2\text{O}(g)} \text{CaCl}_2(\text{aq}) \quad (3)$$

Therefore, sludge with a water content of 5% was selected for subsequent combustion experiments.

3.5. Effect of Sludge Particle Size. Figure 5a,b shows the effect of particle size on the amount of HCl production and HCl capture efficiency. Figure 5a shows that as the particle size of the sludge gradually decreases, the amount of HCl production decreases first and then increases. When the particle sizes are 1700−250 μm, the production amounts of HCl are reduced from 10.075 to 2.776 mg/m³. When the particle sizes are 180−150 μm, the production amounts of HCl increase to 4.294 mg/m³. It can be seen from Figure 5b that with the gradual decrease in the sludge particle size, the HCl capture efficiencies increase first and then decrease, reaching a maximum capture efficiency of 79.81%, when the particle sizes are 380−250 μm.

This is mainly because the coarse sludge causes insufficient contacts between the sludge and Ca(OH)$_2$ and the contact area is small, so that the inhibitory effect of Ca(OH)$_2$ on HCl is not obvious. The smaller the particle size, the less the resistance for volatiles to escape from the sludge, which shortens the time for volatiles to escape. Its secondary reaction outside the particles increases the distribution rate of chlorine in the gas phase.

3.6. Effect of Chlorine-Containing Additive Types. Figure 6 shows the effect of additive types on the amount of HCl production. The addition of NaCl and PVC was 5, 10, 15, and 20% of the weight of the sludge. It can be seen that as the amount of NaCl added increases, the range of HCl production varies from 3.197 to 4.108 mg/m³. High temperature promotes partial decomposition of NaCl, but the degree of decomposition is not large. PVC has a greater influence on the production of HCl than NaCl, indicating that inorganic chlorine is not the main source of HCl.

The reason for the low production of HCl from the combustion of inorganic chlorides may be that NaCl has a high lattice energy (786 kJ/mol), while the binding energy of PVC is 326 kJ/mol.14 The low chloride-ion release rate increases the formation potential of heavy metal chlorides, thereby reducing the formation of HCl. In addition, in the sludge added with NaCl, the impurities react to produce low-melting crystals that affects the combustion efficiency, thereby hindering the precipitation of HCl.

3.7. BET Results. Table 4 shows the BET results of Ca(OH)$_2$ before and after combustion. It can be seen that a small specific surface area and a larger pore size led to a small pore volume of Ca(OH)$_2$. The mesoporous Ca(OH)$_2$ before and after combustion are all located at pore sizes of 20−50 nm. After combustion, the specific surface area of Ca(OH)$_2$ decreases, while the pore volume and pore size increase. The addition of Ca(OH)$_2$ during sludge combustion can effectively reduce the amount of HCl production. On one hand, Ca(OH)$_2$ has adsorption capacity; on the other hand, calcium-based compounds such as Ca(OH)$_2$ and CaO are the good acid gas absorbent. The added Ca(OH)$_2$ may be converted into CaCO$_3$ or CaO, and then, a chemical reaction will occur between the converted absorbent and the HCl produced during the sludge combustion, which reduces the amount of HCl production.

4. CONCLUSIONS

With the addition of Ca(OH)$_2$, the influence of combustion temperature, moisture, sludge particle size, and chlorine-containing additives on the HCl capture efficiency was emphatically explored. Results show the following:

1. The addition of Ca(OH)$_2$ can inhibit HCl during the combustion of sludge. When the amount of Ca(OH)$_2$ added is 40% of the mass of sludge, the amount of HCl produced is reduced to 4.800 mg/m³ and the capture efficiency reaches 67.45%.

2. Temperature and moisture content can promote the production of HCl. When the temperature is 750 °C, the capture efficiency is 90.52%. When the sludge moisture content is 5%, the HCl capture efficiency is 79.81%.

Table 4. BET Results of Ca(OH)$_2$ before and after Combustion

| samples | surface area (m$^2$/g) | pore volume (cm$^3$/g) | pore size (nm) |
|---------|-----------------------|-----------------------|----------------|
| Ca(OH)$_2$ before being incinerated at 850 °C | 5.8159 | 0.004579 | 24.0304 |
| after being incinerated | 1.8481 | 0.009340 | 40.7373 |

Figure 6. Effect of additive types. [Ca(OH)$_2$ = 30%], [temperature = 850 °C], [moisture content = 5%], and [particle size = 380−250 μm].
(3) With the gradual decrease in the sludge particle size, the amount of HCl production decreases first and then increases and the capture efficiency of HCl increases first and then decreases. When the particle sizes are 380–250 μm, the production of HCl reaches the lowest value of 2.776 mg/m³ and the highest capture efficiency is 79.81%.

(4) The effect of PVC on the production of HCl is greater than that of NaCl, indicating that inorganic chlorine is not the main source of HCl.

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Notes
The authors declare no competing financial interest.

Acknowledgments
This work was supported by the National Natural Science Foundation of China (NSFC-51778264), the Project of Science and Technology Program of Guangdong Province (2018B020208002), the youth Top-notch Talent Special Support Program of Guangdong Province (2016T0932576), the Outstanding Young Scientific and Technological Talent Support Program of South China Institute of Environmental Sciences, the National Natural Science Foundation of China (NSFC-41663002), and the Jiangxi Natural Science Foundation Project (20161BAB203080; 20192BAB213027).

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