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<td>Characteristics of leachate from pyrolysis residue of sewage sludge</td>
</tr>
<tr>
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Characteristics of leachate from pyrolysis residue of sewage sludge

I.H. Hwang*, Y. Ouchi, T. Matsuto

Laboratory of Solid Waste Disposal Engineering
Graduate School of Engineering
Hokkaido University
Kita 13, Nishi 8, Kita-ku
Sapporo 060-8628, Japan

*Corresponding author. Tel./fax: +81-11-706-6830/6831.
E-mail address: ihwang@eng.hokudai.ac.jp (I.H. Hwang).
Abstract

The pyrolysis residue (SP) of sewage sludge (SS) produced at 500 °C was subjected to batch and column leaching tests to investigate the release of its organic and inorganic constituents and metals. For comparison, incineration ash (SI) obtained from a SS incinerator was also tested. Pyrolysis and incineration reduced organic matter of SS from 0.78 kg kg⁻¹-dry SS to 0.16 and 0.01 kg kg⁻¹-dry SS, respectively. Heavy metals remained in SP without being volatilized, although Cd and Pb were transferred into the off-gas during incineration.

In the batch leaching test with the leaching liquid-to-solid mass ratio (L/S) = 10, the pH of the SS, SP, and SI filtrates was 6.3, 7.9, and 11.0, respectively. The total organic carbon concentrations were in the order SS (8771 mg l⁻¹) >> SP (99 mg l⁻¹) > SI (26 mg l⁻¹). The SP and SI filtrates met the landfill standard for the Cd and Pb concentrations (< 0.3 mg l⁻¹). In the column tests, although the SP contained more organic matter than that of SI, its carbon discharge into the leachate under aerobic conditions was similar to that of SI under anaerobic conditions. The leaching of heavy metals, such as Cd, Cr, Pb, and Zn, was also suppressed in SP during the active decomposition of organic matter.

We demonstrated that pyrolysis reduces the potential release of pollutants from sewage sludge in landfill, making it a promising method of treating sewage sludge before landflling.

Keywords: Batch and column leaching tests; Incineration ash; Landfilling; Pyrolysis residue; Sewage sludge
1. Introduction

The thermal treatment of sewage sludge is considered one of the best treatment methods, since dumping in the sea, direct landfilling, and agricultural utilization have been limited or prohibited. The remarkable volume reduction in sewage sludge through incineration is attractive for countries that have difficulty in securing landfill sites. For example, in Japan, over two million tons of sewage sludge (on a dry basis) were generated in 2002 and about 89% of that was incinerated (Sannomiya and Kato, 2004).

However, such large-scale thermal conversion of solid to gas requires extensive flue gas cleaning and safe disposal of toxic heavy metals concentrated in final residue. Regulations related to incineration and landfilling stipulate strict criteria for gas emissions and require detailed information on the composition and leachability of the residues (Lopes et al., 2003). Werther and Ogada (1999) suggested that the capital and operating costs of gas purification and ash disposal were driving forces for seeking alternatives to the mono-combustion of sewage sludge. To this end, sewage sludge pyrolysis can be considered an alternative.

Pyrolysis is the thermal degradation of material in the absence of air or in an oxygen-deficient atmosphere. The three major products of pyrolysis are gas, liquid (oil and tar), and carbonaceous residue (char). Many researchers have investigated the product yield and characteristics of sewage sludge pyrolysis (Kaminsky and Kummer, 1989; Rumphorst and Ringel, 1994; Caballero et al., 1997; Inguanzo et al., 2002; Gascó et al., 2005; Shen and Zhang, 2005; Domínguez et al., 2006; Karayildirim et al., 2006). Conesa et al. (1997) established the pyrolysis kinetic model of sewage sludge, in which three different organic fractions decompose independently.
Compared to incineration, pyrolysis has the following advantages: the amount of flue gas to be cleaned decreases, the generation of acidic gases and dioxins are inhibited under reducing conditions, and the release of heavy metals concentrated in the pyrolysis residue is well suppressed. Kistler et al. (1987) studied the behavior of heavy metals such as Cr, Ni, Cu, Zn, Cd, Pb, and Hg during sewage sludge pyrolysis and found that those metals were highly immobile in the pyrolysis residue due to the well buffered neutral to alkaline properties of the char.

Previously, we characterized chars derived from various solid wastes, including sewage sludge (Hwang et al., 2007). As sewage sludge char produced at 500 °C had a high ash content and low heating value, it was considered landfill material rather than an alternative fuel. Although several studies have examined metal release from the pyrolysis residue of sewage sludge (Kistler et al., 1987; Kaminsky and Kummer, 1989; Rumphorst and Ringel, 1994; Shinogi et al., 2003), they were limited to batch leaching tests or were performed without considering landfill conditions, such as the redox potential and microbial activity.

Therefore, we measured the release of the organic and inorganic constituents and metals from the pyrolysis residue of sewage sludge in column tests under simulated landfill conditions. Bottom ash obtained from a sewage sludge incinerator was tested together to compare the environmental loads of the leachates depending on thermal treatment.
2. Material and methods

2.1. Sample preparation

2.1.1. Sewage sludge and incineration ash

10 kg of dewatered sewage sludge were obtained from a sewage treatment plant with a capacity of 3600 m$^3$ d$^{-1}$ sewage in April 2004. This dewatered sewage sludge is referred to as SS. The moisture content of SS was about 75%. For further experiments, SS was dried at 60 °C until no weight change was observed.

This plant uses an incinerator to treat 550 t d$^{-1}$ dewatered sewage sludge. During the drying process before incineration, calcium hydroxide is added to dewatered sewage to control arsenic release into the leachate. The weight ratio of calcium hydroxide to dewatered sewage sludge is 0.2 based on the dry weight. The incineration temperature ranged from 900 to 1000 °C and the average retention time was 1 h. About 5 kg of incineration ash were sampled from an ash conveyor line, which is referred to as SI.

2.1.2. Pyrolysis residue

250 to 300 g h$^{-1}$ of dry SS was loaded to a laboratory-scale rotary kiln-type reactor (φ 0.15 m × 1.2 m) with using a screw feeder and it was pyrolyzed at 500 °C (Hwang et al., 2007). Nitrogen was used as the carrier gas at a rate of 11 l min$^{-1}$ to maintain an inert atmosphere. The slope angle and rotation rate of the kiln were adjusted to 0.8 ° and 2 rpm, respectively, to maintain a retention time of 1 h. After pyrolysis and cooling, the solid residue was weighed. The pyrolysis residue obtained from dry SS is subsequently referred to as SP.
2.2. Experiments

2.2.1. Organic matter and metal content

SS, SP, and SI were pulverized to homogeneity in a ball mill for 30 min. Ignition loss at 600 °C is used as a kind of index for organic matter and is also nearly equal to the sum of volatile matter and fixed carbon determined by proximate analysis (JISC, 2004). The organic matter of SS and SP was estimated by proximate analysis whereas that of SI was obtained from ignition loss at 600 °C for 3 h. The microwave-assisted acid digestion method was used to decompose the solid matrix before measuring the metal content of the samples (USEPA, 1996).

2.2.2. Batch and column leaching tests

Japanese leaching test no. 13 (JLT-13; Environment Agency of Japan, 1973) was used to measure the leaching concentrations of organic matter and metals from SS, SP, and SI. A 10 g dry sample was immersed in a flask containing 100 ml of distilled water, and it was capped and shaken horizontally for 6 h at 200 rpm. The mixture was filtered using 1 µm pore filter paper, and the filtrate was prepared for measuring pH, total organic carbon (TOC), and metals such as Cd, Cr, Pb, Zn, Cu, Na, K, and Mg.

In addition, three 0.1 × 0.4 m (D × H) transparent acrylic columns were prepared for the column tests: two were packed with 0.9 kg of dry SP and one was filled with 1.5 kg of dry SI. The initial moisture content and effective bed height of the packing material were adjusted to 17% and 0.24 m, respectively. Two separate SP columns and one SI column were kept under aerobic, anaerobic, and anaerobic conditions,
respectively. The upper and lower parts of the aerobic column were open to air, while the anaerobic columns were airtight. To facilitate microbial activity under the given conditions, the supernatant obtained from a mixture of soil and distilled water was seeded into the aerobic column, whereas the supernatant from anaerobically digested sludge was added to the anaerobic column. All three columns were placed in a room at 30 °C.

Every two weeks, 200 ml of distilled water were injected into each column for eighteen weeks. For the anaerobic columns, micro-tubing pumps were used to sprinkle distilled water to prevent air permeation. After 24 h, the leachate was collected at the lower part of the column and weighed with an electric balance. The upper and lower parts of the anaerobic column were purged with nitrogen to drive out any air that may have entered the column during the sampling procedure. The pH, electrical conductivity (EC), oxidation–reduction potential (ORP) of the leachate were measured as quickly as possible. Each leachate was filtered using 1 µm pore filter paper before measuring the TOC, 5 day biochemical oxygen demand (BOD), total nitrogen (TN), ammonia (NH₄), inorganic carbon (IC), chloride, and sulfate ion. To measure metal concentrations (Cd, Cr, Pb, Zn, Cu, K, Na, and Mg), the leachate was filtered through a 0.45 µm pore filter paper and acidified with one to two drops of concentrated nitric acid.

2.2.3. Analytical methods

The pH, EC, and ORP were measured using glass electrode methods. A simultaneous TOC-TN analyzer (TOC-V CPH/CPN; Shimadzu) was used to measure the TOC, TN, and IC concentrations. The BOD and NH₄ concentrations were measured using the methods of the Japan Society for Analytical Chemistry (JSAC, 1994). The
concentrations of Cd, Cr, and Pb were analyzed using graphite furnace atomic absorption spectrometry (AAS; Z-8200; Hitachi), whereas the concentrations of Zn, Cu, and Mg were analyzed using inductively coupled plasma atomic emission spectrometry (ICPS-7500; Shimadzu). Finally, K and Na were measured using flame AAS (Z-8200; Hitachi). Detection limits of metals are as follows: Cd 0.2 µg l⁻¹, Cr 0.4 µg l⁻¹, Pb 2 µg l⁻¹, Zn 1 µg l⁻¹, Cu 1 µg l⁻¹, K 0.1 mg l⁻¹, Na 0.1 mg l⁻¹, and Mg 1 µg l⁻¹.

2.3. Estimating the total amounts of released constituents

The total amounts of organic and inorganic constituents and metals released from 1 kg of dry SS according to pyrolysis and incineration were estimated as follows:

\[ M_i = \frac{\sum_{j=1}^{n} (C \times V_j)}{W \times \lambda}, \]

where \( M_i \) is the total amount of constituent \( i \) released, \( C \) is the concentration of constituent \( i \) in the \( j \)-th sample (mg l⁻¹), \( V_j \) is the leachate volume of the \( j \)-th sample (l), \( W \) is the weight of the packed material (kg-dry), and \( \lambda \) is the ratio of the solid residue to dry SS by weight.

3. Results and discussion

3.1. Weight reduction by pyrolysis and incineration
As illustrated in Fig. 1, the total solids in the SS was reduced to 0.37 and 0.38 kg kg\(^{-1}\) dry SS by pyrolysis and incineration, respectively (\(\lambda_{SP} = 0.37\) and \(\lambda_{SI} = 0.38\)). In addition, the organic matter in the SS (0.78 kg kg\(^{-1}\) dry SS) decreased to 0.16 and 0.01 kg kg\(^{-1}\) dry SS, respectively. Although the organic matter was largely degraded by incineration, \(\lambda_{SI}\) was similar to \(\lambda_{SP}\), owing to the calcium hydroxide added during the drying process before incineration. In general, calcium hydroxide is converted into calcium oxide and water on heating at above 800 °C. Therefore, the weight of calcium oxide in SI was estimated to be 0.15 (0.2 \(\times\) 56/74) kg kg\(^{-1}\) dry SS, as shown in Fig. 1.

3.2. Metal contents of SS, SP, and SI

Table 1 shows the metal contents of SS, SP, and SI. In order to understand the metal balances among SS, SP, and SI, the metal contents of SS were counted from the metal contents of SP and SI. Most metals in the SS remained in the SP. However, some of the Cd, Pb, and Zn in the SI seemed to volatize during incineration. Kistler et al. (1987) reported that Cr, Cu, Zn, and Pb remained in the char during pyrolysis at temperatures up to 705 °C, while the Cd was reduced to Cd\(^{0}\) and volatilized to the off-gas at temperatures above 600 °C. As we conducted the pyrolysis at 500 °C, the Cd remained in the char without volatilization.

3.3. Leaching characteristics during JLT-13

Table 2 shows the pH, TOC, and metal concentrations of SS, SP, and SI filtrates obtained according to JLT-13. The pH of the SS, SP, and SI filtrates was 6.3, 7.9, and
11.0, respectively. The high pH of SI was caused by the large quantity of calcium oxide it contained. The TOC released from SS, SP, and SI shows that pyrolysis and incineration also reduced the soluble organic matter considerably.

Pyrolysis and incineration are thought to suppress heavy metal leaching. The Cd, Pb, and Zn concentrations in the SI filtrate were below the detection limits. Cd, Cr, Pb, and Zn were essentially immobilized in SP. Thus, the SP and SI filtrates met the landfill standard for the Cd and Pb concentrations (< 0.3 mg l⁻¹). The leaching ratios of those metals calculated from the total amount and amount in the leachate were nearly zero, as shown in Table 2. In contrast, alkali metals tended to be released readily compared to heavy metals. The high acid-buffering pH of incineration ash and the surface properties of pyrolysis residue might be the reasons for the immobilization of heavy metals (Kistler et al., 1987; Inguanzo et al., 2002; Hwang et al., 2007).

3.4. Results of the column tests

3.4.1. Gas generation

In the aerobic SP column (SP-A), the gas concentration was checked to determine whether air permeated the column well. Beginning 2 wk after setting up the column, the oxygen concentration averaged 20.4 ± 0.7% (mean ± SD).

Over the course of the experiment, CO₂ 20 mmol kg⁻¹ SP and H₂ 4 mmol kg⁻¹ SP were emitted from the anaerobic SP column (SP-An), whereas no gas was generated from the anaerobic SI column (SI-An) because the microbial activity was hindered by the high pH.
3.4.2. The pH, EC, and ORP

Figure 2 plots the changes in the pH and EC of the leachate versus the cumulative liquid (distilled water) to solid (packed material) weight ratio, i.e. L/S. The pH did not change significantly throughout the experiment (Fig. 2a). The pH of the leachates from SP-A, SP-An, and SI-An was in the range of 7.9-8.5, 7.5-8.1, and 11.8-12.2, respectively, and they were nearly the same as the pH of SP and SI under JLT-13 (Table 2). The slightly lower pH of the SP-An leachate can be explained by the accumulation of organic acid due to anaerobic microbial reactions. Biodegradable organic matter was promptly decomposed into carbon dioxide under aerobic conditions, whereas it was first decomposed into organic acids under anaerobic conditions.

Except for the initial period of unsaturation with L/S = 0-1, the EC values of the SP-A, SP-An, and SI-An leachates continued to decrease with L/S, indicating that the concentrations of dissolved constituents were reduced by the normal washout (Fig. 2b).

The ORP values of SP-A, SP-An, and SI-An were in the range of 210-300 and 190-240, and 40-80 mV (Eh), respectively. Even though both SP-An and SI-An were maintained airtight conditions, the ORP did not show negative values. This is because leachate might not represent the true redox potential of the bed due to the reason that ORP was measured 24 h after the start of water injection under the atmosphere.

3.4.3. TOC, BOD, TN, NH₄-N, IC, Cl⁻, and SO₄²⁻

Figure 3 plots the TOC, BOD, TN, NH₄-N, IC, Cl⁻, and SO₄²⁻ concentrations versus the cumulative L/S ratio.
In SP-A (Fig. 3a), the TOC and BOD decreased rapidly from 459 to 51 mg l\(^{-1}\) and from 445 to 2 mg l\(^{-1}\), respectively, until L/S = 1.1 (8 wk after setting up the column) and then decreased slowly until L/S = 2.2.

In SP-An (Fig. 3b), the decreases in the TOC and BOD concentrations were slightly slower than in SP-A (Fig. 3a) because the organic acid produced by anaerobic degradation was measured as TOC and BOD. The TOC and BOD concentrations decreased from 568 to 109 mg l\(^{-1}\) and from 643 to 11 mg l\(^{-1}\), respectively, until L/S = 1.4. The inorganic carbon concentration increased from 20 to 127 mg l\(^{-1}\) until L/S = 0.4 and then decreased.

As depicted in Fig. 3c, the TOC and BOD concentrations were similar as was their rate of decrease. Most of the carbon compounds in SI are thought to be biodegradable organic matter. Nevertheless, microbial decomposition was hindered or did not occur because of the high pH. Accordingly, any biodegradable organic matter was simply washed out by distilled water without microbial degradation.

The TN and NH\(_4\)-N concentrations in SP-A were three to four times lower than in SP-An (Figs. 3a and 3b). The rapid decreases in the TN and NH\(_4\) concentrations in SP-A might have resulted from assimilation caused by aerobic microorganisms and by ammonia nitrification. In contrast, the initial TN and NH\(_4\)-N concentrations in the SI-An leachate were considerably low (< 10 mg l\(^{-1}\)) because a large amount of nitrogen had already been lost during the sewage sludge incineration. Similar to the release of TOC and BOD, the TN and NH\(_4\) in SI-An were also simply washed out by distilled water and discharged into the leachate (Fig. 3c).

The decrease in inactive compounds, such as chloride, is an index of the washout rate by normal rainfall. The chloride concentrations in the SP columns decreased from
224–278 to 128–145 mg l\(^{-1}\) (Figs. 3a and 3b). Similarly, the chloride concentration in SI-An decreased linearly from 361 to 135 mg l\(^{-1}\) (Fig. 3c).

The pattern of sulfate release from the SP columns differed depending on whether the conditions were aerobic or anaerobic. As shown in Fig. 3a, the sulfate concentration was nearly constant under aerobic conditions because it was decreased by normal washing out, whereas it decreased sharply to below 10 mg l\(^{-1}\) until L/S = 1.4 under anaerobic conditions (Fig. 3b). The abrupt drop in the sulfate concentration in the leachate might be explained by the reduction of sulfate besides washing out. In the SI-An, the sulfate concentration decreased in parallel to that of chloride, indicating that it was released via washout (Fig. 3c).

3.4.4. Heavy metals

Figure 4 plots the Cd, Cr, Pb, and Zn concentrations in leachate versus the cumulative L/S ratio. The Cd concentration was 2 \(\mu\)g l\(^{-1}\) at maximum and decreased continuously in SP-A, whereas it was not detected in SP-An after L/S = 0.4. This is because CdS is precipitated via the reduction of sulfate. As shown in Fig. 3a, the sulfate concentration in SP-An decreased drastically after L/S = 0.4 with the reduction of sulfate: \(\text{SO}_4^{2-} \rightarrow \text{S}^2^-\). In SI-An, no Cd was detected throughout the experiment.

The level of Cr detected was less than 13 \(\mu\)g l\(^{-1}\) in SP columns, and it was slowly washed out by distilled water. The SI-An leachate contained 80–190 \(\mu\)g l\(^{-1}\) Cr, which was six to fifteen times higher compared to the SP columns.

The Pb concentrations were almost below the detection limit (2 \(\mu\)g l\(^{-1}\)) in SP-A and SI-An, except for some fluctuations. The Pb in SP-An was slightly higher than in SP-A, but decreased slowly to < 2 \(\mu\)g l\(^{-1}\).
The Zn concentrations decreased to 100 µg l⁻¹ in both SP columns. Based on the similar leaching patterns of SP-A and SP-An (Figs. 4a and 4b), washout by distilled water controlled the Zn release.

3.5. Total release of carbon, nitrogen, and heavy metals

Table 3 shows the total amounts of carbon, nitrogen, and heavy metals released into the leachate for 18 wk assuming that 1 kg of dry SS is pyrolyzed or incinerated and then their residues are landfilled under aerobic or anaerobic conditions. The release of carbon and nitrogen into the leachate was reduced considerably by pyrolysis and incineration. The TOC release into the leachates of SP-A, SP-An, and SI-An was 62, 133, and 52 mg kg⁻¹ dry SS, respectively; i.e., the order of carbon release was SP-An > SP-A ≈ SI-An. Since the TOC release was in the order SS (87710 mg kg⁻¹ dry SS) >> SP (366 mg kg⁻¹ dry SS) > SI (99 mg kg⁻¹ dry SS) for JLT-13 (see Table 2), the results of column tests indicate that the biodegradable organic matter in SP actively decomposed into carbon dioxide under aerobic conditions. In SP-An, 88 mg C kg⁻¹ dry SS was emitted as carbon dioxide into the air (see section 3.4.1). Accordingly, about 34% of released carbon (TOC + IC + carbon as CO₂) was emitted to the air and 66% was discharged into the leachate. In contrast, in SI-An, all 58 mg C kg⁻¹ dry SS was released to the leachate. Since the SP-A column was open to ambient air, the gas was not measured quantitatively.

The TN release was in the order SP-An (47 mg kg⁻¹ dry SS) > SP-A (17 mg kg⁻¹ dry SS) > SI-An (3 mg kg⁻¹ dry SS). This was because the nitrogen content of SI was markedly smaller than that of SP.
While the release of Cd, Pb, and Zn was in the order SP-A ≥ SP-An > SI-An, Cr was SI-An > SP-A = SP-An. The amount of heavy metals released from SP was larger than that from SI except Cr; however, as shown in Table 3, the difference in the total leaching of heavy metals depending on the pretreatment methods and landfill conditions was very minute.

4. Conclusions

We investigated the pyrolysis residue and incineration ash obtained from sewage sludge as pretreated materials for landfilling based on the concentrations of organic and inorganic constituents and heavy metals released in batch and column leaching tests.

Although the SP contained relatively more organic matter than the SI and held most metals without volatilization during thermal treatment, the releasing of organic matter and heavy metals was considerably reduced in its batch leaching test.

Moreover, in the column leaching tests, the release of organic matter from the SP into the leachate was decreased to the same level of SI, since it was biodegraded under the neutral to weak alkali conditions. In contrast, the decomposition of organic matter in SI was inhibited by the high pH.

The amount of heavy metals released from the SP was greater than from incineration ash; however, the amount leached was very small in terms of ppb. In addition, we found little difference in the amount leached between aerobic and anaerobic conditions. SP showed good ability to retain heavy metals without the addition of extra chemicals such as calcium hydroxide for SI.
The potential release of organic and inorganic constituents and metals from sewage sludge can be reduced by pyrolysis, which could serve as a suitable thermal treatment method for sewage sludge prior to landfilling.

References


Table 1
Metal balance of SS estimated from the metal content of SP and SI

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Units: mg kg<sup>−1</sup> dry

<sup>a</sup> = (Metal content of SP) × 0.37
<sup>b</sup> = (Metal content of SI) × 0.38
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<tr>
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<td>47 (24.0)</td>
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*: dry SS
Units: mg l⁻¹
( ): metal leaching ratio (%) = \([\text{leaching concentration by JLT-10, mg l}^{-1}] ÷ \text{[metal content in Table 1, mg kg}^{-1}] \times 10 \text{(L/S)} \times 100\)
\(-\): not estimated
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<td>$6.6 \times 10^3$</td>
<td>8</td>
<td>30</td>
<td>1</td>
</tr>
<tr>
<td>Cd</td>
<td>$5.6 \times 10^{-2}$</td>
<td>$5 \times 10^{-4}$</td>
<td>$2 \times 10^{-4}$</td>
<td>-</td>
</tr>
<tr>
<td>Cr</td>
<td>$6.8 \times 10^{-1}$</td>
<td>$3 \times 10^{-3}$</td>
<td>$3 \times 10^{-3}$</td>
<td>$4.5 \times 10^{-2}$</td>
</tr>
<tr>
<td>Pb</td>
<td>$5.6 \times 10^{-1}$</td>
<td>$5 \times 10^{-3}$</td>
<td>$2 \times 10^{-3}$</td>
<td>$8 \times 10^{-4}$</td>
</tr>
<tr>
<td>Zn</td>
<td>34</td>
<td>$1.4 \times 10^1$</td>
<td>$1.5 \times 10^1$</td>
<td>$4 \times 10^3$</td>
</tr>
<tr>
<td>Cu</td>
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<td>$4.0 \times 10^{-2}$</td>
<td>$2.5 \times 10^{-2}$</td>
<td>$8 \times 10^{-3}$</td>
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<tr>
<td>K</td>
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<td>187</td>
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<tr>
<td>Na</td>
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<td>25</td>
<td>34</td>
<td>75</td>
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<tr>
<td>Mg</td>
<td>472</td>
<td>16</td>
<td>19</td>
<td>8</td>
</tr>
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Units: mg kg$^{-1}$ dry SS

$^a$: Calculated from JLT-13 (Table 2)
Fig. 1. Weight reductions of SS by pyrolysis and incineration and the composition of SS, SP, and SI.
Fig. 2. Changes in pH and EC values plotted against the cumulative L/S ratio.
Fig. 3. Changes in the TOC, IC, BOD, TN, NH₄-N, Cl⁻, and SO₄²⁻ concentrations of the leachate plotted against the cumulative L/S ratio.
Fig. 4. Changes in the Cd, Cr, Pb, and Zn concentrations of the leachate plotted against the cumulative L/S ratio.