

Behavior of Cu, Pb, and Zn in Ash during the Endothermic Burning of Mixed Industrial Wastes

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ABSTRACT

The behaviors of Cu, Pb, and Zn during the endothermic burning of heterogeneous wastes were investigated using a variety of operational parameters, i.e., the mixed waste ratio, burning temperature, and burning time, to obtain fundamental knowledge to generate an optimal burning operation and recycling strategy for bottom ash. Changing these parameters had no impact on the Cu content of the ash, whereas the Pb content depended on the burning temperature and the mixed ratio, and the Zn content was affected by all three parameters. It was found in this study that the optimal burning conditions were a temperature of 1100°C, a time of 15 minutes, and either the current waste conditions or waste conditions with double the waste plastic and wood content.

Keywords: Heavy Metals, Endothermic Burning, Portioning Behavior, Industrial Waste, Ash

1. Introduction and Methods

Japan has two categories of waste: general waste mainly from residential areas, and industrial waste. Industrial waste accounts for almost 90% of the waste generated, reaching about 400 million tons annually. For treatment of both types of waste, Japan primarily uses incineration. In fact, over 80% of the generated general waste, approximately 40 million tons annually, is incinerated. A variety of metal elements are present in the resultant incineration ashes, especially in the fly ash or fly and bottom mixed ash; therefore, intermediate treatment methods to reduce their environmental impact are nationally designated. The disposal of ashes treated by either solidification in cement or melting fusion into landfills and the recycling of those ashes as commercial materials have a serious impact on health and economics due to the spread of toxic materials (*i.e.*, heavy metals) into the environment and the waste of non-recycled metal resources [1,2]. Burning is also one of the nationally designated intermediate treatment options for ash recycling (**Figure 1**). This method is commonly used by private companies that generate a large amount of ash to avoid cost-prohibitive ash disposal in landfill sites. Since ash burning is an endothermic process, fuel is necessary to generate the heat required. Normally a synthesized fuel or refused paper &

plastic fuel (RPF) is used instead of kerosene to reduce the operating costs.

Alternatively, the ash is burned with other combustible waste such as plastic and wood, which results in the mixed burning of ash, plastics, and paper or wood. As mentioned earlier, ash contains metal elements and the effect of heterogeneous burning conditions on the behavior of those metal elements has not yet been investigated. The purpose of this study is to investigate the behaviors of Cu, Pb, and Zn, as representative elements with negative health and environmental impacts, under heterogeneous waste burning with varying burning temperature, burning time, and mixed waste ratio. Understanding the behaviors of metals in heterogeneous conditions is essential in order to determine the appropriate conditions for burning operations and to optimize a recycling strategy for the bottom ash from endothermic burning.

2. Materials and Methods

2.1. Waste Samples

The following six samples were used as samples for burning: automobile shredder residue (ASR) fly and bottom ashes, fly and bottom ashes from the incineration of waste plastics and woods, waste plastic residue from RPF

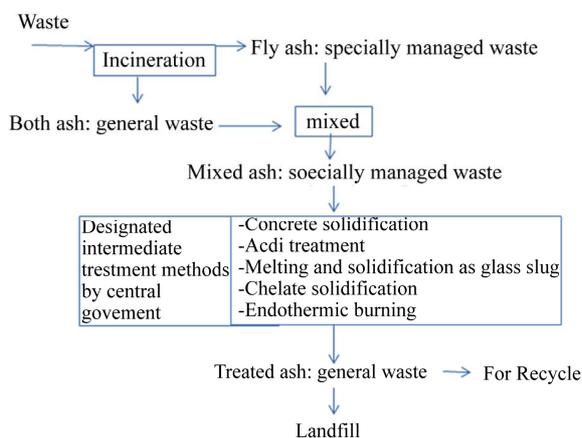


Figure 1. Designated intermediate treatment methods for fly ash or fly and bottom ash, as determined by the central Japanese government.

production, and the benthic sludge of rivers. The ASR fly and bottom ashes were obtained from the incineration of ASR by a rotary kiln.

2.2. Methods for the Analysis of the Total Content of Elements in the Waste Samples

The conditions of the boiling extraction method for element extraction [3], which is the standard Japanese method, were examined to determine the optimal conditions for element extraction from the ash samples after incineration of the mixed waste. The selected conditions are shown in **Table 1**.

An inductively coupled plasma–mass spectrometer (ICP-MS) was used for elemental analysis, and hydrochloric acid (conc. 35% - 37%, Kanto Kagaku), nitric acid (60% - 61%, Kanto Kagaku) and deionized water were used as solvents. The standard method for boiling extraction was as follows: The ash sample (1 ± 0.01 g, wet base) was placed in a beaker and mixed with hydrochloric acid (1:4 HCl:H₂O) and nitric acid (1 + 1 HNO₃:H₂O) and boiled for 30 minutes. After cooling, the solution was transferred to a 100 mL volumetric flask, which was accurately filled with deionized water. Next, the solution was filtered using a 1 μ m cellulose acetate filter (Advantec). A 10 mL aliquot was diluted to 50 mL in a volumetric flask using deionized water. The solution was then analyzed by ICP-MS (HP4500, Yokokawa).

2.3. Quantitative Analysis of Cu, Pb, and Zn Content

Cu, Pb, and Zn heavy metals, which are present in relatively high concentrations and cause considerable harm to the environment and human health, were selected for analysis using an atomic absorption spectrophotometer (AAS; A-2000, Hitachi). The samples were pre-treated

Table 1. Pretreatment condition.

Altered conditions	Details
Standard (SD)	Boiling extraction using nitric acid and hydrochloric acid
Altered condition 1 (AC1)	Double concentration of hydrochloric acid of SD
Altered condition 2 (AC2)	Double concentration of nitric acid of SD
Altered condition 3 (AC3)	Double concentrations of hydrochloric and nitric acids of SD
Altered condition 4 (AC4)	Double boiling time of SD
Altered condition 5 (AC5)	Double boiling time and concentration of hydrochloric acid of SD

with AC1, which uses double the concentration of hydrochloric acid as compared to the standard (see **Table 1**). For degradation of the waste samples, two methods were tested: degradation at 600°C combustion or drying at 105°C. For the first method, the waste samples were burned at 600°C for 60 minutes and the ash was collected for total content analysis for the metals. For the second method, the sample was prepared by drying it at 105°C without burning and then analyzed for the total content of the metals. The ash or dry samples (2 - 5 g) were transferred into a flask and hydrochloric (60 mL) and nitric (30 mL) acids were added, and the solution was heated until it reduced to about 5 mL. After cooling, 20 mL of hydrochloric acid (1 + 5 HCl:H₂O) was added to the solution, which was further heated for 5 - 6 minutes. After cooling again, the solution was diluted to 100 mL in a volumetric flask using deionized water. The diluted solution was filtered through a 1 μ m glass fiber filter and the filtrate was analyzed using an AAS.

2.4. Quantitative Analysis of Cu, Pb, and Zn in Ash

The content of heavy metals, *i.e.*, Cu, Pb, and Zn, in ashes burned under different conditions was analyzed via the following procedure. The AC1 pretreatment, which uses a double concentration of hydrochloric acid as compared to the standard, was used. The parameters that were investigated in this study were waste sample mixed ratio, burning temperature, and burning time. Different waste samples types were prepared with varying mixed ratios, as summarized in **Table 2**. Burning temperatures were either 900°C, 1000°C, or 1100°C and burning times were either 15, 30, or 60 minutes in an electric furnace (KDF S80, Eyela). All possible combinations of parameters were performed and the sample descriptions are shown in **Table 3**.

Ash samples (2 - 5 g) were transferred into a flask, hydrochloric (60 mL) and nitric (30 mL) acids were added, and the solution was heated until it was reduced to about

Table 2. Mixed status of burning samples for designing the experimental preparation (%).

Sample type		Type A	Type B	Type C
		Current condition of waste	Double of ASR content	Double of waste plastic & wood content
ASR	bottom ash (BA)	11.22	22.44	11.22
	fly ash (FA)	6.12	12.24	6.12
Waste plastic & wood	bottom ash (BA)	2.04	2.04	4.08
	fly ash (FA)	1.02	1.02	2.04
Waste plastic residue from RPF production (RPF-R)		67.34	50.02	64.36
Benthic sludge of rivers (BSR)		12.24	12.24	12.24

Table 3. Combination of experimental conditions and sample IDs.

Burning temperature (°C)	Burning time (minutes)	Sample type in Table 2	Sample ID
900	15	A	915A
		B	915B
		C	915C
	30	A	930A
		B	930B
		C	930C
	60	A	960A
		B	960B
		C	960C
1000	15	A	1015A
		B	1015B
		C	1015C
	30	A	1030A
		B	1030B
		C	1030C
	60	A	1060A
		B	1060B
		C	1060C
1100	15	A	1115A
		B	1115B
		C	1115C
	30	A	1130A
		B	1130B
		C	1130C
	60	A	1160A
		B	1160B
		C	1160C

5 mL. After cooling, 20 mL of hydrochloric acid (1 + 5 HCl:H₂O) was added to the solution, which was further heated for 5 - 6 minutes. After cooling again, the solution was diluted to 100 mL using deionized water in a volumetric flask. The diluted solution was filtered through a 1 µm glass fiber filter and the filtrate was analyzed using an AAS (A-2000, Hitachi).

3. Results

3.1. Effect of the Chemical Pretreatment on the Content Analysis

Figures 2(a)-(f) show the efficacy of each of the pretreatment methods and the amount of each selected element. The heavy metals were almost undetectable in the samples of waste plastic residue from RPF production (RPF-R) and the benthic river sludge (BSR) using SD and AC3 methods. The AC1 method resulted in the most balanced detection of the Cu, Zn and Pb metals and other elements, and is therefore considered to be the optimal pretreatment method for analyzing heavy metals contained in these samples. The amount of Cu, Zn, and Pb in each sample after pretreatment using the AC1 method is shown in Figure 3. The ASR bottom ash sample (ASR-BA) contained the largest amount of Cu, Zn, and Pb.

3.2. Effect of Sample Preparation on the Cu, Pb, and Zn Content

To optimize the detection of the selected heavy metals in the samples, two methods of preparing the samples, *i.e.*, burning at 600°C or drying at 105°C, were investigated. Regardless of the waste sample types and the species of heavy metal, the metal content of the burned samples was always larger than that of the dried samples (Figures 4(a)-(i)). Therefore, it is evident that burning the samples at 600°C is a better method for degrading the waste samples for optimal detection of the heavy metals. The reason for the lower detection of the metals in the dried samples might be the remnant presence of organic materials. The initial reaction of the organic materials with hydrochloric acid and altering the pH and oxidation-reduction potential (ORP) did not alter the environment around the heavy metals enough to accelerate the metal extraction from the samples.

3.3. Effect of Varying the Burning Conditions on the Cu, Pb, and Zn Content

Figures 5(a)-(c), 6(a)-(c) and 7(a)-(c) show the behaviors of Zn, Pb and Cu, respectively, under different burning conditions, where Figures 5-7(a), Figures 5-7(b), and Figures 5-7(c) display the effects of varying the temperature, burning time and sample type, respectively.

4. Discussion

4.1. Optimal Chemical Pretreatment

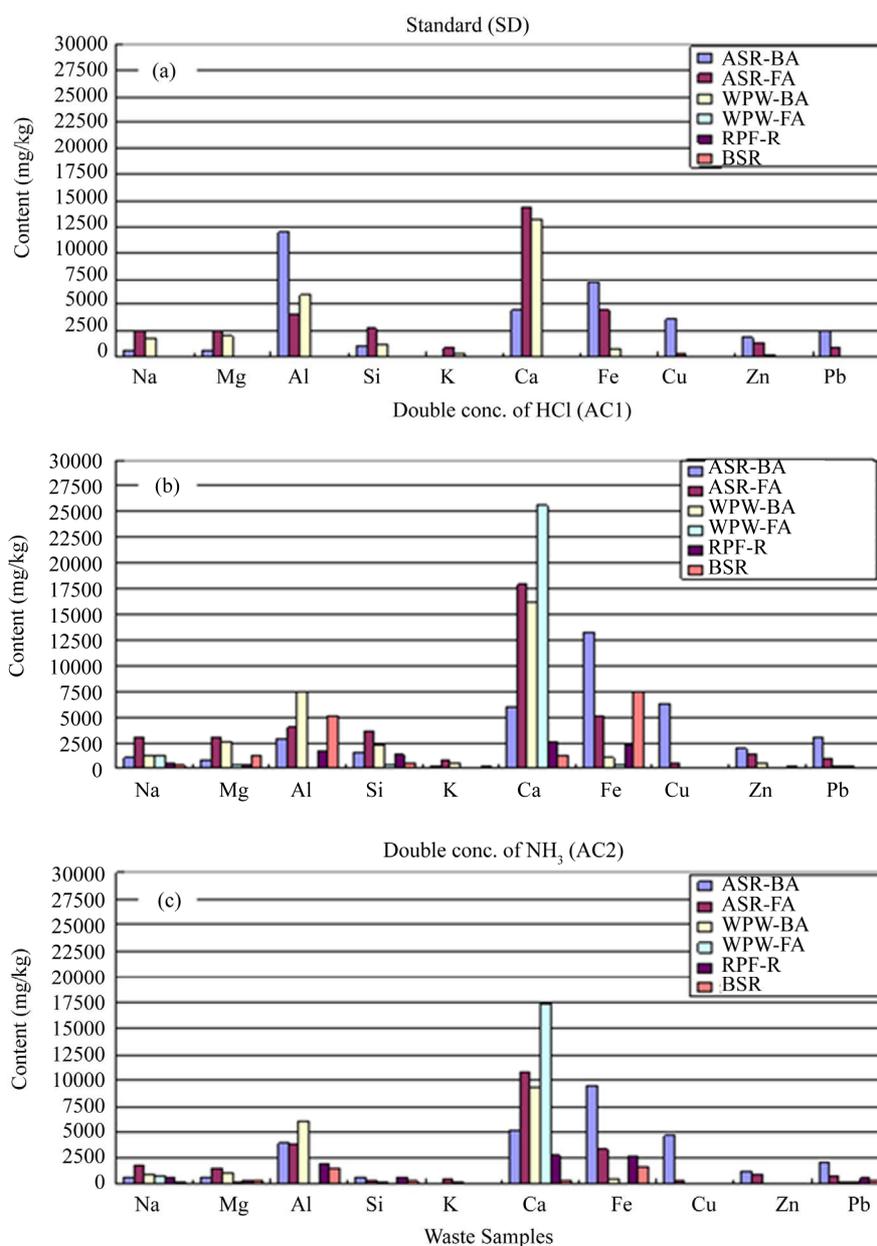
The metal content of the samples were assessed employing the 6 different pretreatment methods described in Table 1 and Figure 2. High amounts of all metals were detected using both the AC1 and AC4 pretreatment methods, although the metal levels detected after the AC4

pretreatment were higher. However, to maximize time efficiency, AC1 was used as the pretreatment for the further analyses, as the boiling time was 30 and 60 minutes for AC1 and AC4, respectively. Using the SD method, the elements shown in **Figure 2** could not be detected in the WPW-FA, RPF-R, and BSR waste samples. These results indicate that SD, which is especially useful for heavy metal analysis in sewage sludge, was not suitable for the waste samples focused on here. Since an accurate measurement of the total heavy metal content in solid samples is almost impossible using the acid solution extraction method with ICP, it is more accurate to refer to the results as the “maximum extracted element content”

instead of the “total content of heavy metals.” Al, Ca, and Fe were detected in high levels in all of the waste samples. The amount of the Zn, Pb and Cu detected in each sample in **Figure 3** was shown in **Figure 4(a)**, **Figure 4(d)**, and **Figure 4(g)**, respectively, and was significantly dependent on the heat treatment. Therefore, it was concluded that heat treatment is extremely important for analyzing the elemental content of waste samples.

4.2. Optimal Thermal Pretreatment

Figures 4(a)-(i) show the results of the thermal destruction portion of the pretreatment. For all cases, the total metal content was higher in the samples treated at 600°C



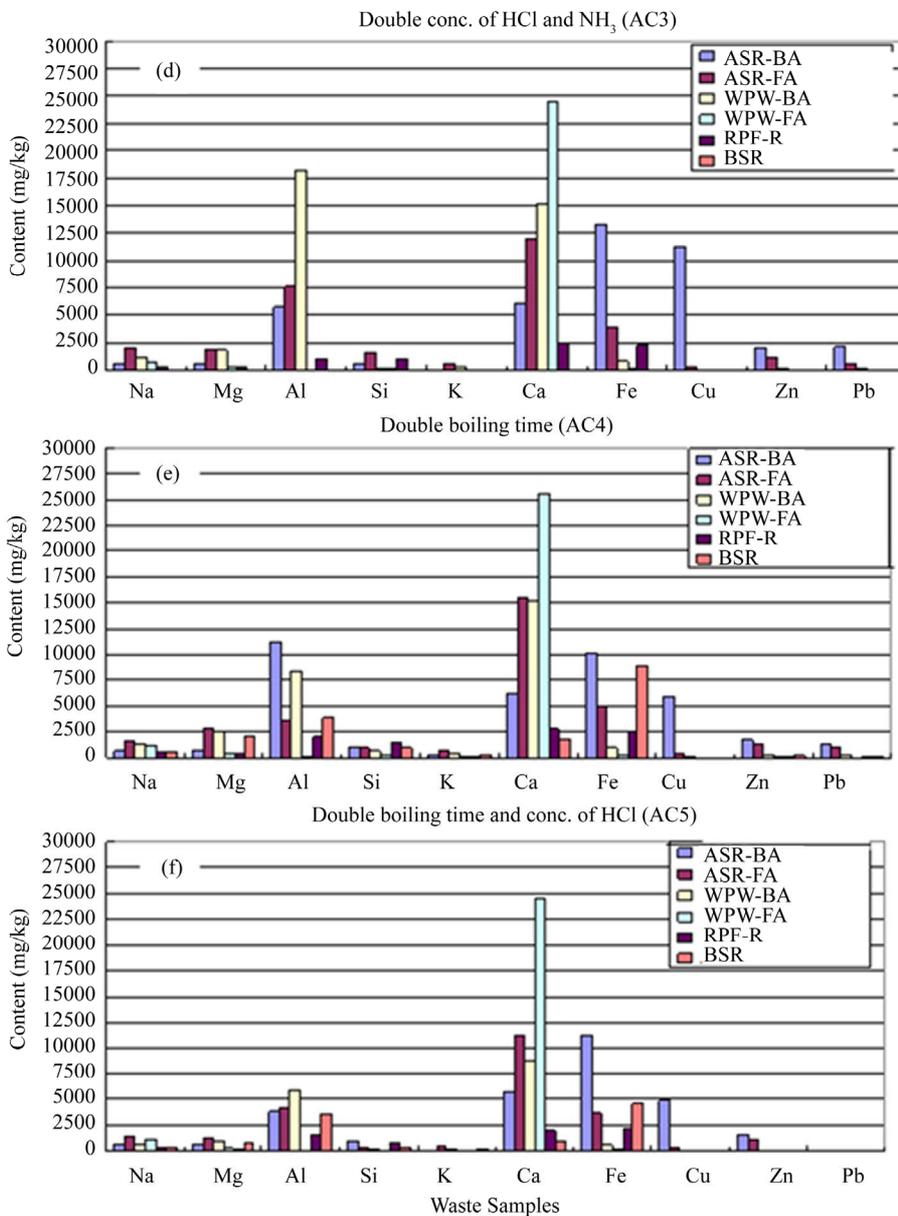


Figure 2. Element contents in samples treated by (a) Standard method, (b) Double concentration of HCl (Altered condition 1), (c) Double concentration of NH₃ (Altered condition 2), (d) Double concentrations of HCl and NH₃ (Altered condition 3), (e) Double boiling time (Altered condition 4), and (f) Double boiling time and concentration of HCl (Altered condition 5).

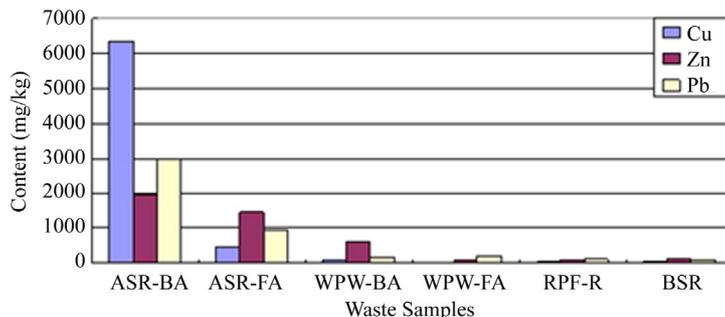


Figure 3. Cu, Zn, and Pb contents in samples pretreated by Double concentration of HCl (Altered condition 1).

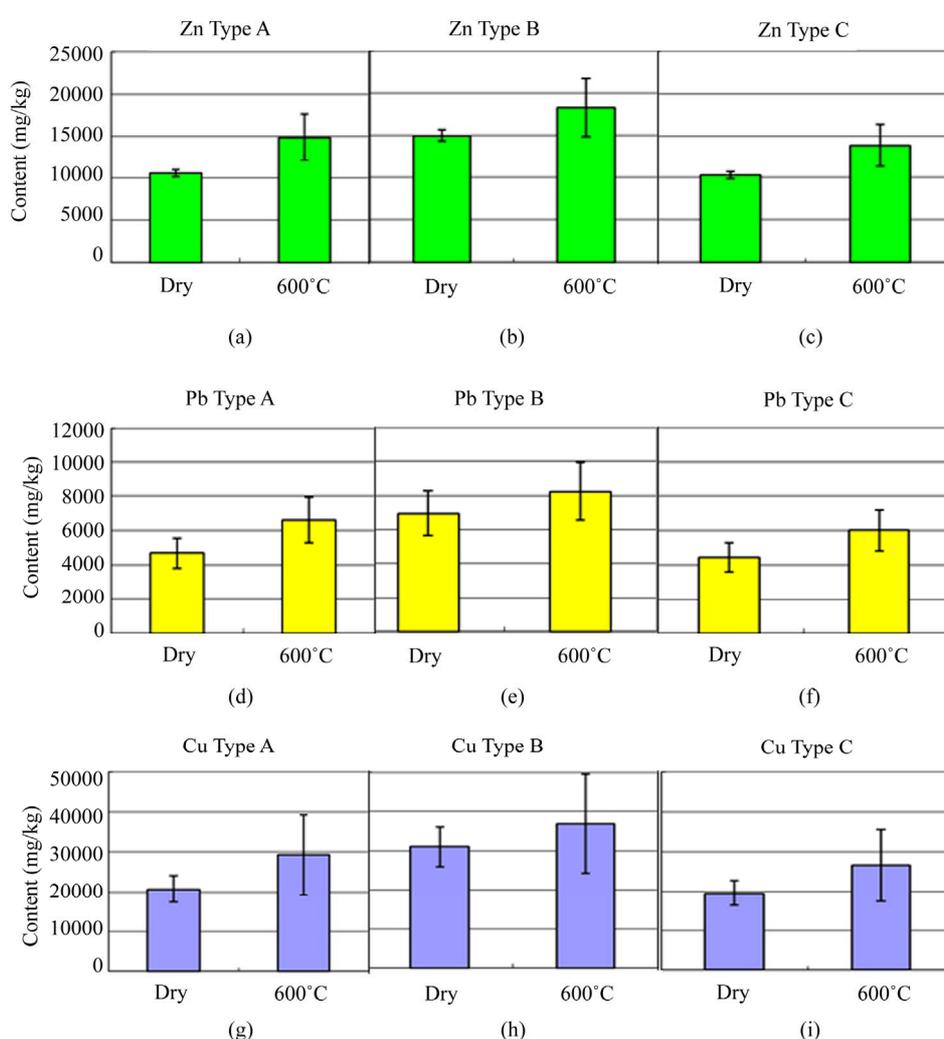


Figure 4. Comparison of pretreatment (thermal treatment) on element content: (a)-(c):Type A-C on Zn, (d)-(f):Type A-C on Pb, and (g)-(i):Type A-C on Cu.

than those that were dried at 105°C. As it was established that thermal pretreatment at 600°C is better than at 105°C, the effect of thermal pretreatment at 900°C, 1000°C, and 1100°C was also investigated. Since the time of burning or drying was one hour for the results in **Figure 4**, a comparison of **Figure 4** and 960A–C, 1060A–C, and 1100A–C for the detection of Cu, Pb, and Zn (**Figures 5–7**) was carried out. The amounts of the elements detected after the 600°C thermal pretreatment was highest in almost all cases (**Figures 8–10**). There was the only one exception: the amount of Pb detected after 960B pretreatment was higher than that after 600°C thermal pretreatment. Therefore, it can be concluded that the 600°C thermal pretreatment was the best overall method to obtain the maximum extraction content of elements from waste samples.

4.3. Behaviors of Cu, Pb, and Zn in Ash

Figures 5–7 show the behaviors of Zn, Pb, and Cu, re-

spectively, under different operating conditions; the results are summarized in **Table 4**.

The speciation of Cu, Pb, and Zn during municipal solid waste combustion was described as follows [4,5]: Cu, CuCl, CuH, CuO, CuS, Cu₂, (CuCl)₃, CuCl, CuO, CuO–Al₂O₃, CuO–Fe₂O₃, CuS–FeS, CuSO₄, Cu₂O–Al₂O₃, Cu₂–Fe₂O₃, Cu₂S, Cu₅FeS₄, Pb, PbCl, PbCl₂, PbO, PbS, Pb₂, PbCl₂, PbO–SiO₂, 2Pb–SiO₂, PbS, PbSO₄, Pb₂B₂O₄, Pb₃O₄, Zn, ZnCl₂, ZnS, ZnO, ZnO–SO₂, ZnS, ZnSO₄, 2ZnO–SiO₂, Zn₂SiO₄, ZnFe₂O₄, ZnCr₂O₄, ZnAl₂O₄, and ZnO–2ZnSO₄. The metallic states (*i.e.*, Zn, Pb, and Cu), mono-oxidized species (*i.e.*, ZnO, PbO, and CuO), and chlorinated species (*i.e.*, ZnCl₂, PbCl₂, and CuCl₂) were chosen as the representative compounds in this study; their melting and boiling points are listed in **Figure 11**.

According to Verhulst *et al.* [6], ZnCl₂(solid (s)) converts to ZnO(s) at less than 300°C and forms ZnCl₂

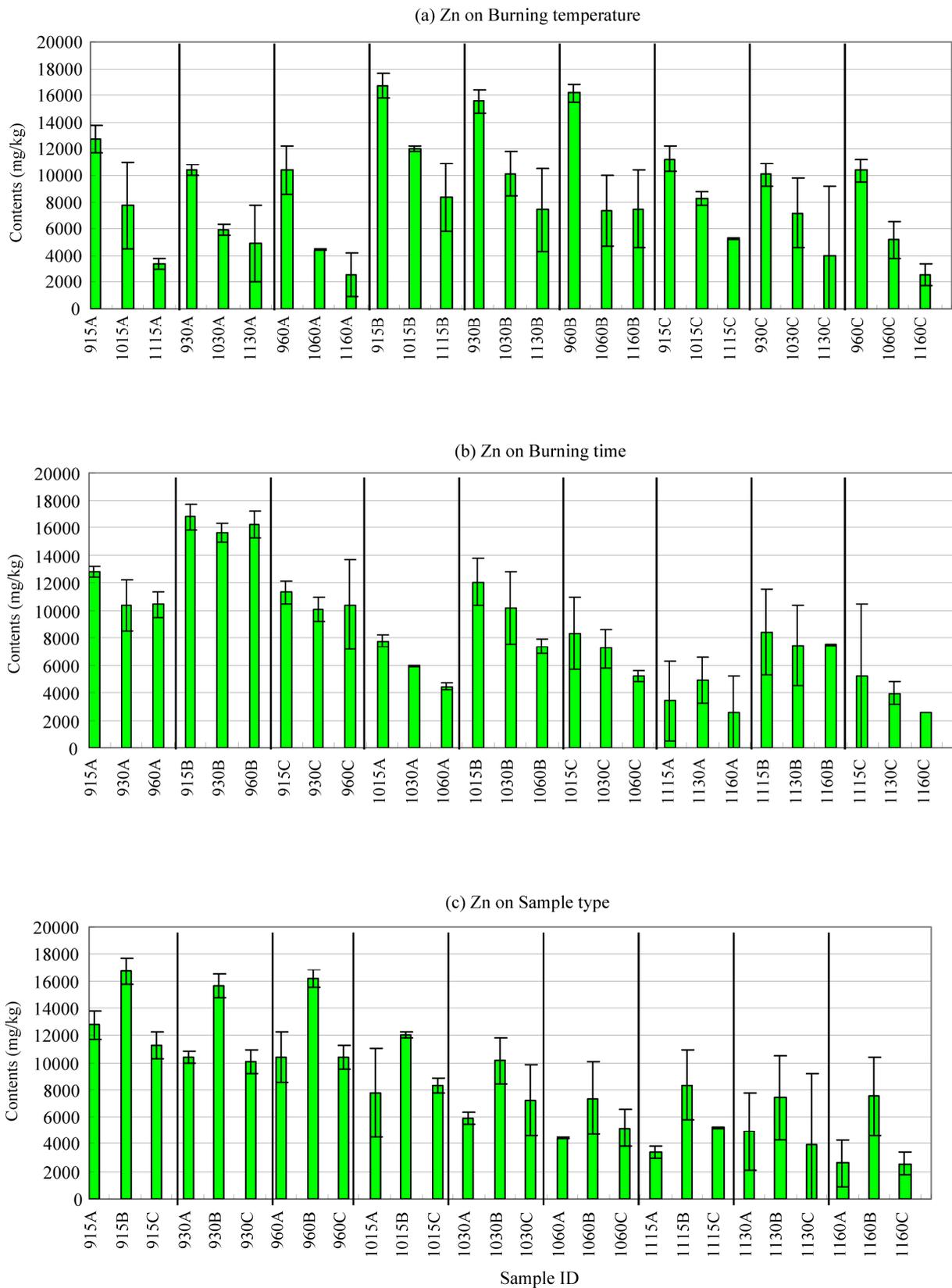


Figure 5. The behavior of Zn depending on the (a) burning temperature, (b) burning time, and (c) sample types.

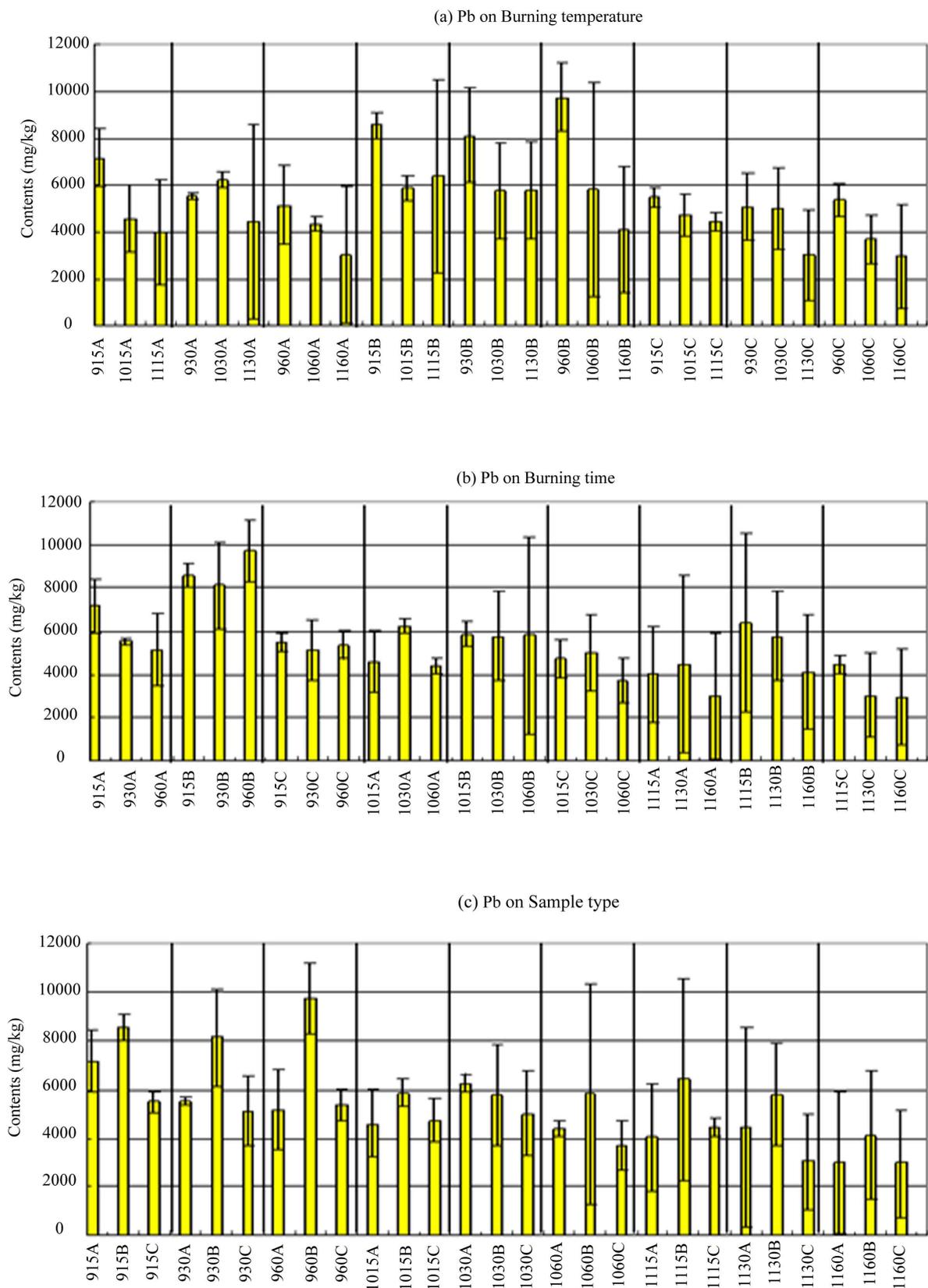


Figure 6. The behavior of Pb depending on the (a) burning temperature, (b) burning time, and (c) sample types.

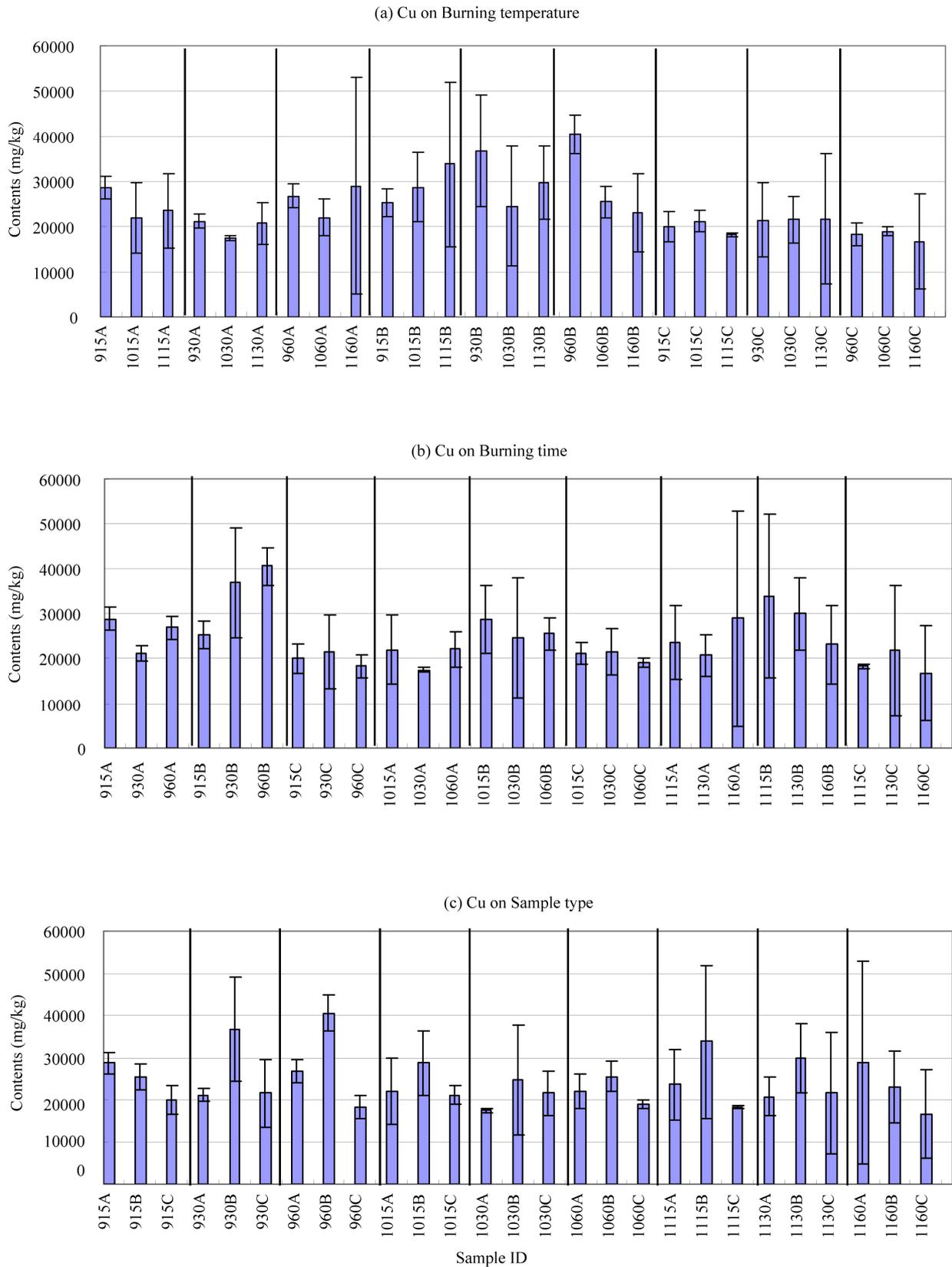


Figure 7. The behavior of Cu depending on the (a) burning temperature, (b) burning time, and (c) sample types.

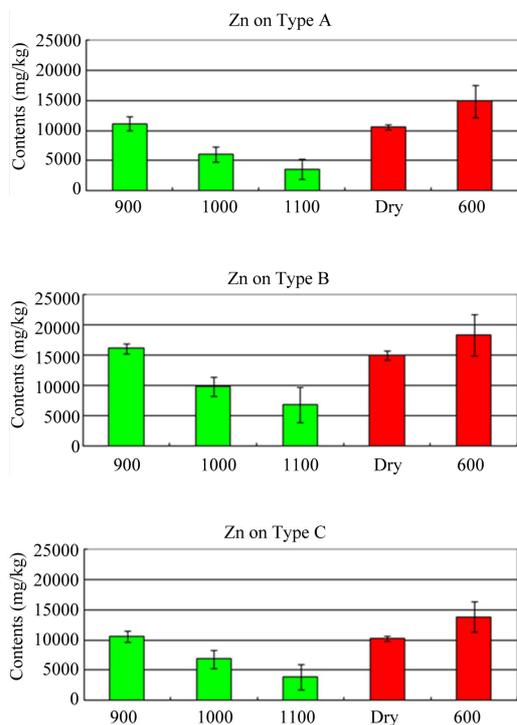


Figure 8. Content of Zn detected after burning at different temperatures.

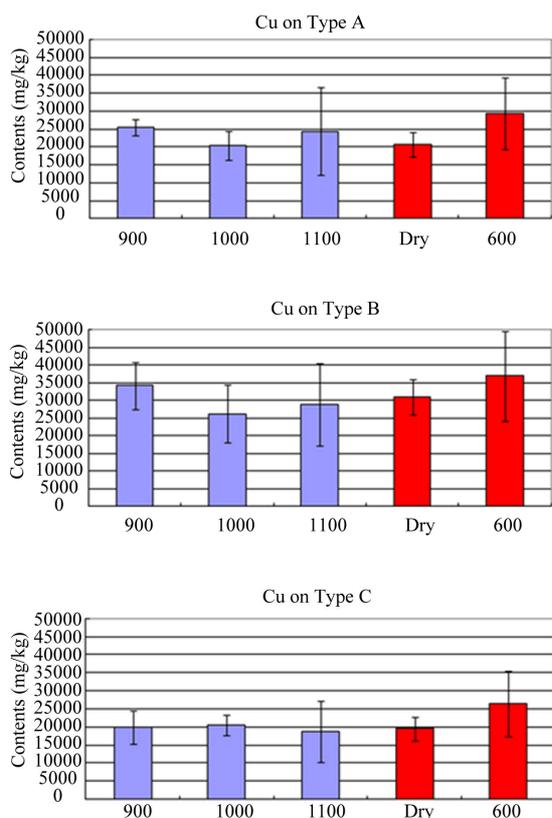


Figure 9. The content of Cu detected after burning at different temperatures.

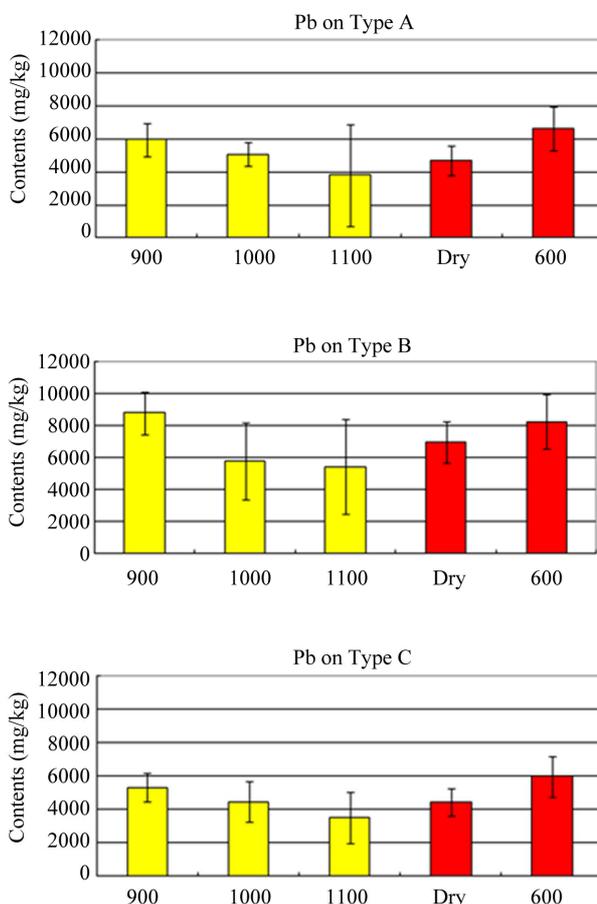


Figure 10. The content of Pb detected after burning at different temperatures.

Table 4. Summary of Zn, Pb, and Cu behaviors.

Influence by	Zn	Pb	Cu
Burning temperature	Its detection became small as temperature goes up. -No difference at 900°C	High detection at 900°C	No significant difference
Burning time	-Its detection became smaller as time becomes longer. -Mixed ratio B always shows the highest.	-No significant difference	No significant difference
Mixed ratio	-No significant difference between mixed ratios A and C.	Mixed ratio B seemed high	No significant difference

(gas (g)), whereas $PbCl_2(g)$ starts to volatilize around 300°C and $PbO(g)$ and $PbCl(g)$ are predominant above 800°C. In the case of Cu, $CuCl_2$ is converted to CuO around 700°C and $CuCl(g)$ is predominant around 900°C after $Cu_3Cl_3(g)$ is formed. Generally, the presence of Cl greatly influences the behavior of metals; the volatility of heavy metals increases when they are chlorinated due to their decreased boiling point.

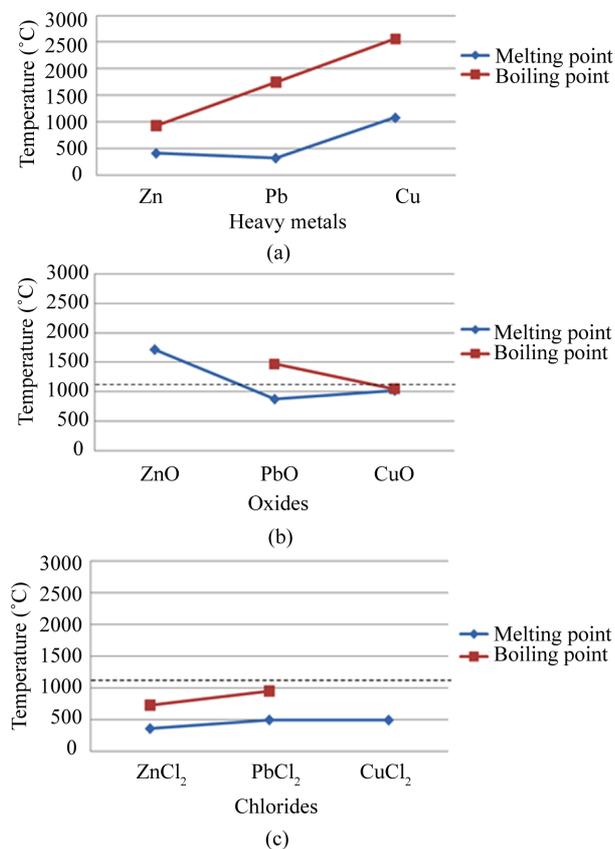


Figure 11. Melting and boiling properties of (a) metal, (b) metal oxide, and (c) metal chloride species. The broken line indicates 1100°C, which was the highest burning temperature in this study. The temperature of ZnO is the sublimation point.

The effect of Cl on heavy metals was discussed by many researchers [7-10]. According to most studies, Cl exerts a stronger influence on Zn than on Cu or Pb; however, Pb was the most volatile chloride according to a study by Trouvé *et al.* (1998). Also, Pb and Zn were more likely than Cu to be transferred into the combustion gas by forming compounds with chlorine [11]. All reports agreed that chlorinated Cu was the most stable. In the current study, the chlorine concentrations in the samples were not analyzed because, in reality, the chlorine concentration in waste cannot be controlled. In addition to the chlorine content, the combustion temperature also greatly influences metal partitioning and speciation [12]. Heavy metal partitioning behaviors are also greatly affected by the presence of alkaline metals such as Na and K and moisture in the waste [13]. According to Wang *et al.* (1999), the presence of Na and K increased the partitioning of heavy metals into fly ash. All of the analyzed samples contained Na and K (Figure 2), but the behavior of Cu was not influenced by the changes in the mixed ratio. In opposition to the trend reported by Wang *et al.*

(1999), Pb and Zn showed the highest contents in the ash under the mixed ratio B, which had the highest Na and K concentration. Therefore, it can be said that the influence of Na and K was not supported by this study.

The behavior of Cu behavior has also been shown to be affected by the presence of Ca; the detection level of Cu in the gas phase drastically increased in the presence of limestone with a Ca/S ratio of 1.3 [14]. However, the increase in the detection level disappeared when the ratio doubled to 2.5. From Figure 2, it is evident that the ASR-FA, WPW-BA, and WPW-FA samples contain a relatively large amount of Ca, which could influence Cu detection in the ash analysis. It has also been reported that the presence of organic chloride species decreases the capture of Cu by limestone, while the presence of inorganic chlorides increases it [15]. The influence of Ca on Cu was not directly tested in this experiment. However, according to Table 2, there was no evident impact of Ca on Cu as the Cu detection levels did not differ significantly upon changes in the mixed ratio (Table 4) although Type C did contain double the amount of WPW-BA and WPW-FA, which contained a large amount of Ca (Figure 2). From the results shown in Table 4, it was determined that Cu was the most stable among the three metals as it demonstrated no significant difference in the Cu levels upon changing the burning temperature, burning time, and waste mixed ratio, which is in agreement with the results of Williams [16] and Trouvé *et al.* (1998). Zn, on the other hand, was the most unstable metal among the three: the content of Zn in the ash decreased with increasing temperature and was also influenced by the burning time at 1000°C and 1100°C. Changes to the mixed ratio resulted in a higher Zn content in the ash from sample type B. In contrast, the amount of Pb detected was not influenced by the burning time (Table 4). Weight percentages of 89% - 96% of Cu, 58% - 94% of Pb, and 37% - 86% of Zn remained in the bottom ash [4]. These ranges are consistent with the results of this study.

4.4. Comparison to Environmental Criteria

After burning, ash either goes to a landfill site or to recycling after clearing the criteria for the heavy metal extraction test; lower heavy metal content in the ash is better for both purposes. According to the results, the samples with the lowest content of heavy metals were 1115A and 1115C. The content of Zn, Pb, and Cu was $3,384 \pm 434$ mg/kg, $4,013 \pm 2228$ mg/kg, and $23,570 \pm 8,210$ mg/kg, respectively, for 1115A, and 5219 ± 58 mg/kg, 4455 ± 414 mg/kg, and $18,271 \pm 434$ mg/kg, respectively, for 1115C. The heavy metal content in samples 1115A and 1115C were compared to standards related to environmental issues such as landfill, sea dumping, and composting (Table 5). The experimental values were 7.5 to 11.6, 261 to 337, and 803 to 891 times larger than the

Table 5. Environmental values.

Heavy metals	Landfill standard (Japan) (mg/l)	Sea dumping (Japan) (mg/kg)	Soil (Japan) (mg/kg)	Compost standard (mg/kg)					This study (Mean) (mg/kg)	
				Japan	India [†]	USEPA [†]	Canada [†]	Germany [†]	1115A	1115C
Zn	-	450	-	-	1000	2800	500	400	3,384	5,219
Cu	-	70	125	-	300	1500	60	100	23,570	18,271
Pb	0.3	5	0.01*	-	100	300	150	150	4,013	4,455

note[†]:Reference [17].

sea dumping standards for Zn, Cu, and Pb, respectively. When compared with the United States Environmental Protection Agency (USEPA) compost standards, which are relatively high, the experimental values were approximately 1.2 to 1.9, 12 to 16, and 13 to 15 times higher for Zn, Cu, and Pb, respectively. Therefore, although the content of heavy metals in ash is greatly reduced by tuning the burning temperature and time for Zn and Pb, it is still far higher than that allowed by any of the standards described here.

5. Conclusions

The behaviors of Cu, Pb, and Zn under the endothermic burning of heterogeneous wastes were investigated by changing the operational parameters, *i.e.*, the mixed waste ratio, burning temperature, and burning time, to obtain fundamental knowledge to generate an appropriate burning operation and recycling strategy for bottom ash. Changing these parameters yielded no significant effect on the Cu content of the ash, whereas the Pb content was influenced by the burning temperature and mixed ratio, and the Zn content was influenced by all three parameters. The burning conditions not only influence the partitioning behavior of metals in thermal treatment reactors, such as incinerators, but also the characteristics of the metals in the ash. Therefore, it is important to understand these effects to plan an effective recycling strategy for incineration ash. In this study, the optimal operation conditions were 1115A and 1115C, which correspond to a burning temperature of 1100°C, a burning time of 15 minutes, and either the current waste conditions or the waste condition with double the waste plastic & wood content. It is also important that managers in charge of thermal treatment reactors conduct their own investigation into the partitioning behavior of heavy metals at their plants based on the academic results that have been reported in order to optimize the operations for their reactors.

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