

Thermal separation and leaching of valuable elements from waste-derived ashes

Daniel Lindberg*, Emil Vainio, Patrik Yrjas

Åbo Akademi University, Johan Gadolin Process Chemistry Centre, Piispankatu 8, FI-20500 Åbo/Turku, Finland

*Corresponding author. Tel.: + 358 (0)2 215 4452; fax: +358 2 215 4962

Email addresses: Daniel.lindberg@abo.fi

Abstract

Recovery of trace elements from different industrial waste streams is important in realizing the goals of the circular economy. Ash streams from waste combustion can contain high levels of both toxic and valuable trace elements. These elements can be separated during the combustion process based on volatilization and condensation, as well as being separated in post-combustion processes, through thermal or leaching treatment.

In this study, an overview is given of the thermal methods for recovery of valuable elements from ash fractions derived from waste incineration. In addition, a case study is presented of the behaviour of ash-forming elements in combustion of MSW and demolition wood, with special focus on the elements Co, Cu, and Sb.

In conclusion, it is shown that thermodynamic modeling of high temperature processes can be a useful tool to predict ash behavior both during combustion and in the post combustion treatment of the ash.

Introduction

Different types of ash fractions and various types of APC (air pollution control) residues are formed in thermal conversion of waste-derived fuels. The solid residues from WtE (Waste-to-Energy) plants may contain harmful components, such as toxic metal compounds and organic micropollutants, which may lead to problems in the utilization or safe disposal of the material. Various treatment methods and processes can be used to reduce the leachability of harmful residue constituents, destroy toxic organic compounds (organic micropollutants), reduce residue volume, and produce material suitable for utilization.

Van der Sloot et al. [1], Sabbas et al. [2], Quina et al. [3], Whiticar and Ralph [4], and Zacco et al. [5] have recently reviewed different approaches in the management of the ash and solid APC residues from waste combustion. The two main approaches are safe disposal and recycling or reuse of the ash. In order to safely dispose of or reuse/recycle the ash, separate treatment processes are often required.

The main treatment methods for ash and APC residues can be divided into three groups: 1) separation process (physical or chemical); 2) stabilization and solidification; 3) thermal treatment. Table 1 gives an overview of the principles and methods for treatment of MSW (municipal solid waste) ash, based on the reviews of the International Ash Working Group [6], van der Sloot et al. [1], Sabbas et al. [2], and Whiticar and Ralph [4].

Lindberg et al. [7] recently reviewed different thermal treatment methods of MSW ash residues. The main categories for thermal treatment are vitrification, fusion/melting, and sintering. The thermal methods are mainly differentiated based on the characteristics of the process product, rather than the process itself. In vitrification, a glassy phase is produced, whereas in fusion processes, a crystalline or heterogeneous product is formed by melting the ash or residue. In sintering, the residues are heated to achieve a reconfiguration of solid materials.

In addition, thermal separation processes may also be combined with the above-mentioned processes to vaporize harmful trace elements from the bulk material. Thermal treatment processes are among the best methods to destroy harmful organic compounds, such as dioxins and furans, present in the solid residues.

Table 1. Overview of principles and methods of treatment of ash residues from WtE plants [1, 2, 4, 6].

Treatment Principle	Processes/methods and Unit Operations
Chemical and physical separation	Washing Chemical precipitation Crystallization/evaporation Ion exchange Density and particle size based separation Distillation Electrolysis Electrokinetic separation Magnetic separation Eddy-current separation Chemical extraction/mobilization

	Adsorption
Stabilization and/or solidification	Solidification/stabilization with hydraulic binders and pore-filling additives
	Chemical stabilisation
	Ageing/weathering
Thermal treatment	Sintering
	Vitrification
	Melting
	Vaporization/condensation

Most of the thermal methods are processes that are separate from the thermal conversion of the fuel. One exception is the gasification-melting process or the direct melting process [8-10]. MSW is gasified in a fluidized bed reactor and the syngas is used as the heat source for the ash melting. The gasification-melting process may lead to better separation of different elements compared to traditional MSW incineration, with elements distributed between a silicate slag, the fly ash in the process and a metallic phase [10]. Pb, Zn, and Cl tend to form fly ash, whereas Fe and Cu form a metallic phase. Other common ash-forming elements are distributed in both fly ash and the silicate slag.

Another in-situ process for elemental separation into different fractions is the use of hot cyclones in the combustion unit. During combustion or gasification, heavy metals such as Cd and Pb volatilize. The volatilized metals condense when the gas is cooled and will be found in the fly ash or filter ash. The use of a hot cyclone prior to the heat exchanger could make it possible to separate the heavy metals from the fly ash. In a hot cyclone separation concept, the fly ash is collected in a hot cyclone, where volatilized heavy metals continue with the flue gas stream and is collected as filter ash. In an ideal case, the cyclone ash is heavy-metal free, and can be used directly as a product, while the filter ash is highly enriched in heavy metals and may be more easily handled due to lower volumes of hazardous ash.

Ljung and Nordin [11] studied the fate of different elements during combustion of a fast-growing energy tree (*Salix* spp.) and hot cyclone separation in theory using chemical equilibrium model calculations. Calculation results indicated that hot cyclone separation might be possible for Cd, Pb and Cu due to their relatively low volatilization temperatures. According to the calculations Ni, Zn and V would be difficult to separate with the hot cyclone due to their high volatilization temperatures. The calculations were in good agreement with results from previous field studies.

Obernberger and Biedermann [12] compared a combustion plant (Lofer) and a plant with integrated fractionated heavy metal separation (Straßwalchen). The temperature in the combustion zones was varied to check the influence on the heavy metal concentrations. A high-temperature cyclone was placed behind the furnace in Straßwalchen. The temperature in the cyclone varied between 950 and 1050°C. The Cd concentrations in the bottom ashes were about 20 times lower in the new plant compared to the old. Cd levels in combustion zone fly ash and fly ash from the high-temperature cyclone were also lower for the new plant. Cd concentrations in hot-precipitated fly ash showed clear temperature dependence. The Cd concentrations decreased with increasing temperature of ash precipitation. Zn concentrations in the bottom ash were 5 times lower in the ash from the new plant compared to the old. Zn concentration in the combustion zone fly ash were lower than in hot-precipitated fly ashes produced in the old plant but considerably higher in the fly ash precipitated in the high-temperature cyclone. Zn showed no temperature dependence in the investigated range in either plant. The amounts of fly ashes produced were lower in the new plant and the amount of condensation sludge higher. Coarse fly ash particles could be separated from in the combustion zone at high temperatures with a hot cyclone catching the large scale particles between the furnace and the boiler of the plant. The temperature in the hot cyclone has to be above 900°C to be efficient for heavy metal fractionation.

According to calculations Cd, Pb and Cu could be removed by hot cyclone separation from the fly ash [11]. Due to higher volatilization temperatures Ni and Zn would be difficult to separate from the fly ash. According to Obernberger et al. [12, 13] the cyclone temperature has to be over 900°C for efficient separation.

Objective and approach

The objective of the study is to investigate the ash chemistry in combustion of waste-derived fuels (SRF) with emphasis on the separation of ash-forming elements in different ash streams during the thermal conversion of the fuel. The main division between different ash element streams is between elements that stay in a condensed form during the main conversion process and elements that form volatile species at temperatures prevailing at the conversion temperatures and air/fuel ratios. Predictions of this separation is done by using thermodynamic modeling based on Gibbs Energy Minimization. Thermodynamic modeling predicts the amount and composition of stable phases and components at chemical equilibrium at a specified temperature, total pressure and overall

elemental composition. The approach does not require any prior knowledge of specific chemical reactions or initial speciation of elements. In the general approach, chemical kinetics cannot be considered. However, Kangas et al. [14, 15] have utilized a so-called constrained Gibbs Free Energy Method, where kinetic limitations can be considered for certain reactions. This is however not included in the present study.

Thermodynamic equilibrium calculations

Thermodynamic equilibrium calculations were made to predict the speciation of the ash-forming elements as a function of temperature for both oxidizing and reducing conditions. The advanced thermodynamic modeling was performed using the software package Factsage, version 6.4[16]. A tailor-made thermodynamic database was used for the calculations. The data for the gas phase and the stoichiometric solid phases of the elements C–H–O–N–S–Cl–Na–K–Zn–Pb–Ca–Mg–Fe–Al–Si–P–Ti–Co–Cr–Cu–Mn–Sb–Sn were taken from the FACT Pure substance database in the Factsage software. It was assumed that N₂ was the only stable nitrogen compound as the formation of NO_x compounds in biomass combustion is strongly dependent on kinetics and N-speciation in the fuel. A multicomponent molten salt phase including NaCl–KCl–Na₂SO₄–K₂SO₄–Na₂CO₃–K₂CO₃ was also included together with corresponding solid solutions [17]. A molten silicate/slag phase is available in the thermodynamic databases in Factsage but was excluded due to calculational limitations. All condensed silicate phases are therefore considered as stoichiometric phases in the current calculations.

The main fuel studied in the present report is SRF (solid recovered fuel). SRF is mainly the non-recoverable paper, plastics and textiles fractions from MSW that has been shredded and dehydrated for incineration.. The chemical composition of the fuel is given in table 2. It was based on the chemical composition of a SRF fuel that has been burned in an BFB (Bubbling Fluidized Bed) plant in Anjalankoski, Finland operated by Stora Enso.

Table 2. Fuel composition of SRF used as input for the calculations.

Ultimate analysis (dry solids)	C	wt-% (d.s.)	51.0	
	H	wt-% (d.s.)	6.5	
	S	wt-% (d.s.)	0.2	
	O	wt-% (d.s.)	37.4	
	N	wt-% (d.s.)	0.7	
	Cl	wt-% (d.s.)	0.258	
	F	wt-% (d.s.)	0.003	
	Br	wt-% (d.s.)	0.001	
	Element concentrations in the dry substance	Al	g/kg	3.324
		Si	g/kg	8.936
Ti		g/kg	1.067	
Na		g/kg	1.772	
Mg		g/kg	1.421	
K		g/kg	1.612	
Ca		g/kg	17.884	
Fe		g/kg	1.596	
P		g/kg	0.559	
Heavy metals concentrations in the dry substance		Sb	mg/kg	3.58
	As	mg/kg	0.73	
	Cd	mg/kg	0.24	
	Cr	mg/kg	50.16	
	Co	mg/kg	0.44	
	Cu	mg/kg	24.44	
	Pb	mg/kg	42.63	
	Mn	mg/kg	378.38	
	Ni	mg/kg	9.91	
	Sn	mg/kg	15.91	
	V	mg/kg	3.30	
	Zn	mg/kg	159.44	

The fuel compositions taken from table 2 were used as input for the calculations. The calculations were made as a function of temperature and air-to-fuel ratio (λ). The air-to-fuel ratio was varied between 0.4 and 1.2 with steps of 0.01 and the temperature was varied between 400 and 1300 °C with steps of 10 °C. Additional calculations with either double amounts of Cl & S or completely Cl- and S-free fuels have been calculated, but the detailed results are not shown here.

Results

The predicted main ash phases and the minor gas components of ash forming elements (>1 ppm) are shown in Figure 1 as a function of temperature for reducing and oxidizing conditions ($\lambda=0.7$ and $\lambda=1.2$). Ca-Al- and Ca-Mg-silicates dominate the condensed phases, whereas HCl, KCl, and SO₂ (oxidizing conditions) or H₂S (reducing conditions) are the main alkali, sulfur or chlorine species.

The elements Co, Cu, and Sb are of special interest in the present study for assessing the viability to recycle these elements from the ash [18-20]. It was concluded that these elements often occur in the waste ashes at levels higher

compared to other sources, and therefore may have a potential to be recovered from the ash. In the present study, where solid recovered fuel was studied, Cu has the highest concentration in the fuel and Co has the lowest. It is of interest to predict if these elements can be enriched in the flue gas so that they may be captured in filter ash instead of fly ash or bottom ash.

Cobalt is predicted to be present as $\text{Co}(s, g)$, $\text{Co}_9\text{S}_8(s)$, $\text{CoCl}_2(g)$ at reducing conditions and $\text{Co}_3\text{O}_4(s)$, $\text{CoCr}_2\text{O}_4(s)$, $\text{CoCl}_2(g)$ at oxidizing conditions. Copper is predicted to present as $\text{Cu}(s, l, g)$, $\text{Cu}_2\text{S}(s)$, $\text{Cu}_5\text{FeS}_4(s)$, $\text{CuCl}(g)$ at reducing conditions, and $\text{CuO}(s)$, $\text{CuFeO}_2(s)$, $\text{CuCl}(g)$ at oxidizing conditions. Antimony is predicted to be present as $\text{SbO}(g)$, and $\text{Sb}_2\text{S}_3(g)$ at reducing conditions and $\text{SbO}(g)$, and $\text{Sb}_2\text{O}_5(s)$ at oxidizing conditions. At reducing conditions, no condensed antimony compounds were predicted to be stable at temperatures above 400 °C.

The fraction of volatile Co, Cu, and Sb as a function temperature and air-to-fuel ratio is shown in Figure 2 . In addition, the volatile fraction of the important ash-forming elements Pb, Zn, K, and Na, as well as Sn, are shown. It can be seen that Co is volatilized mainly at reducing conditions and at temperatures above 1100 °C. If the chlorine content in the fuel is doubled, the volatilization of cobalt becomes considerable at temperatures above 700 °C. Copper is volatilized at 600-700 °C at oxidizing conditions except if there is no Cl or S present, where temperatures above 1100 °C are needed for copper to volatilize. At reducing conditions, copper is volatilized at temperatures above 1000 °C. Antimony is predicted to be in a condensed phase only at temperatures below 500 °C and only at oxidizing conditions. Sn and Zn are mainly in the condensed phase at oxidizing conditions up to 1000 °C, whereas Pb is volatilized at oxidizing conditions even down to around 500 °C. K is the main volatilized alkali element, occurring as $\text{KCl}(g)$, $\text{KOH}(g)$ or $\text{KOH}(g)$, and Na occurs as condensed $\text{NaAlSiO}_4(s)$ up to high temperatures.

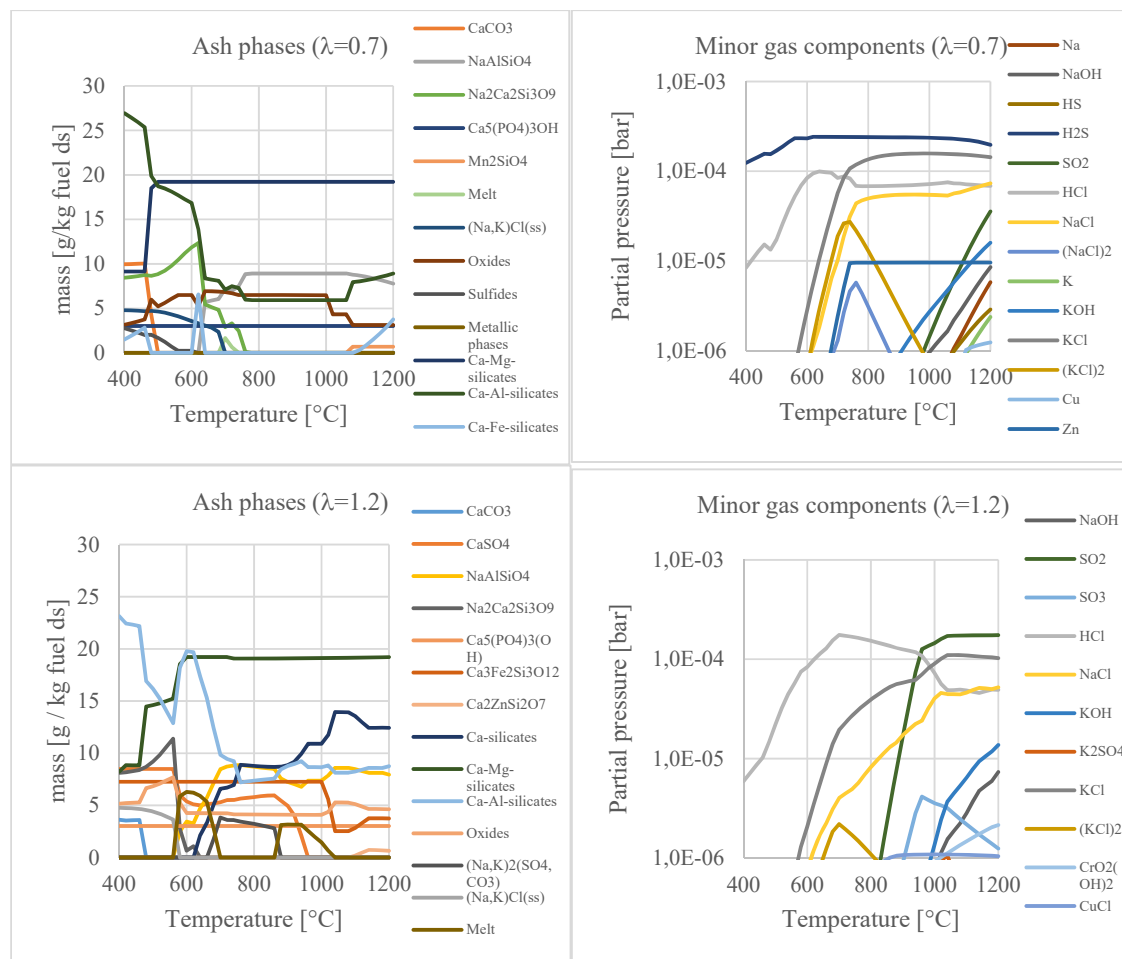


Figure 1. Predicted condensed phases (left), and minor (right) gas components as a function of temperature for SRF conversion at $\lambda=0.7$ (top) and $\lambda=1.2$ (bottom).

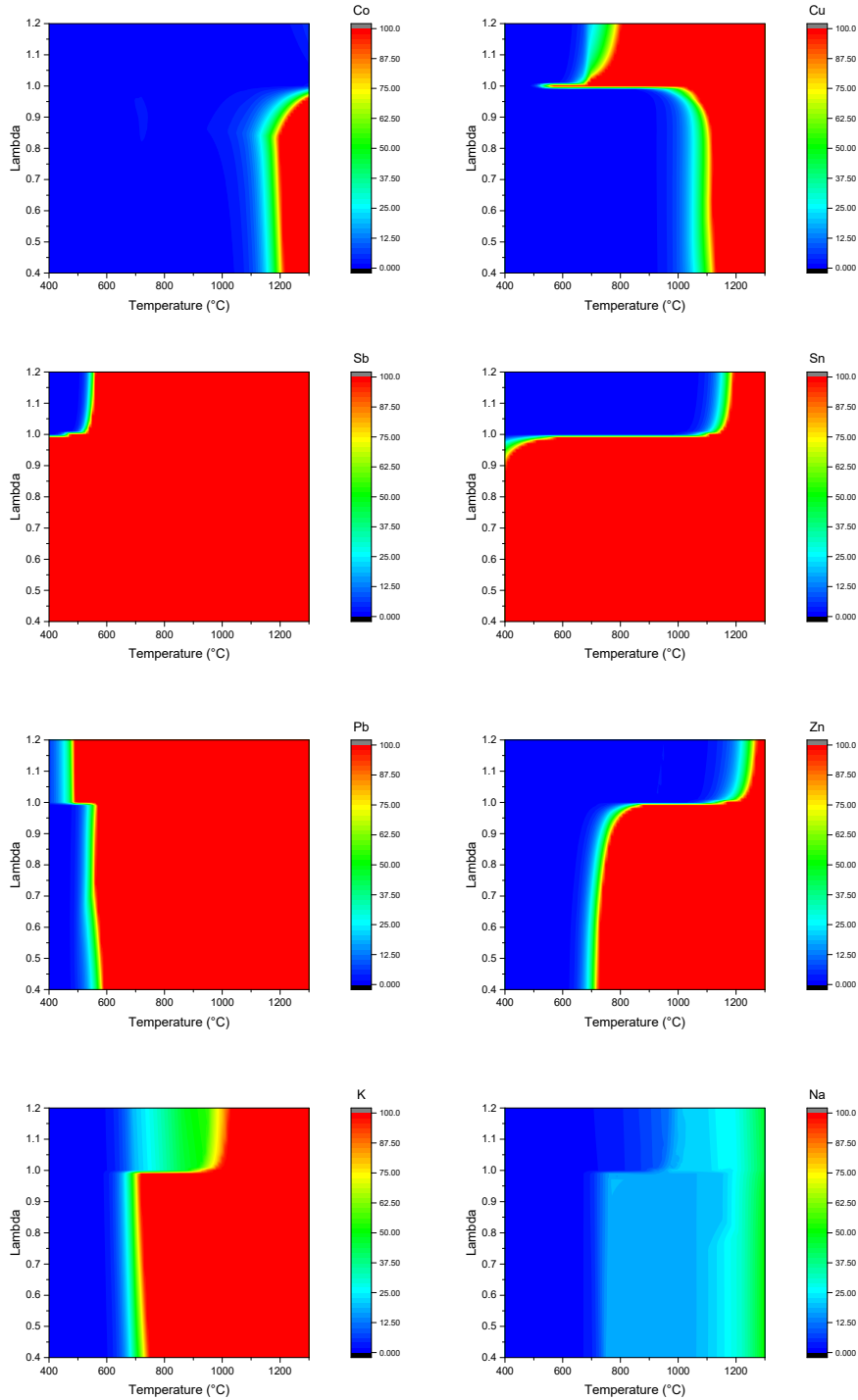


Figure 2. Fraction of Co, Cu, Sb, Sn, Pb, Zn, K, and Na in the gas phase as a function of temperature and air-to-fuel ratio (lambda) for the normal case.

Enrichment of Co, Cu, Sb in gas phase

The enrichment of Co, Cu, and Sb in relation to the other ash-forming elements were calculated for the four different compositional input conditions. The enrichment was calculated as follows:

$$\text{Enrichment factor of element } i \text{ in gas} = \frac{m(i, \text{gas})/m(\text{total metals, gas})}{m(i, \text{fuel})/m(\text{total metals, fuel})}$$

In addition to the metals, phosphorus was also included, whereas the halogens and sulfur were excluded. The calculated enrichment factors for Co, Cu, and Sb are shown in Figure 3 at air/fuel ratios of 0.7 and 1.2. An enrichment factor <1 means that the element is preferentially stable in the condensed phases, whereas values >1 means an enrichment in the gas phase. It can be seen that cobalt is depleted in the gas phase except for high temperatures at reducing conditions and at around 600 °C at oxidizing conditions. Copper becomes enriched in the gas phase above 900 °C at reducing conditions and above 600 °C at oxidizing conditions. Antimony is predicted to be strongly enriched in the gas phase, especially at lower temperatures. There is a thousandfold enrichment of Sb at both reducing and oxidizing conditions below 600 °C. At higher temperatures, the level of volatilized ash-forming elements, such as K, Na, Pb, start to become significant.

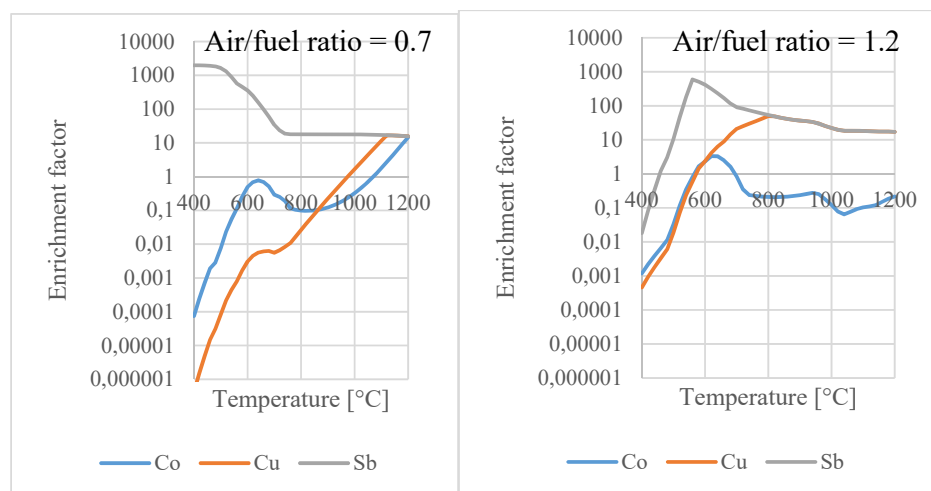


Figure 3. Enrichment factors of Co, Cu, and Sb as a function of temperature for air/fuel ratios of 0.7 (left) and 1.2 (right) for the base case.

Cobalt is mainly depleted from the gas phase and is therefore expected to be found in the bottom ash of different combustion units. The bottom ash fraction is typically considerably larger than fly ash or filter ash fractions, which makes it unlikely of finding high levels of cobalt in any ash fraction.

Copper enrichment in the gas phase is predicted to be the highest at oxidizing conditions at around 600-900 °C. Antimony is predicted to exist mainly for the studied conditions, with the exception of low temperatures at oxidizing conditions. The highest enrichment is predicted to be mainly at temperatures below 600 °C at reducing conditions.

Discussion and conclusions

In a previous report [19], thermodynamic predictions of the speciation of Co, Cu, and Sb in MSW ash showed that the elements mainly occur as different oxides, such as Co_3O_4 , CuO , Sb_2O_5 . Experimentally determined speciation of Cu and Sb suggest similar results. Additional Cu compounds were also observed, such as CuSO_4 and $\text{Cu}_3(\text{PO}_4)_2$. Firing MSW fuels containing high concentrations of halogens may lead to formation of metal halides. We have performed leaching tests in a continuous flow reactor [18, 19, 21] and analyzed the metals that were leached from different waste-derived ashes online by inductively coupled plasma optical emission spectrometry (ICP-OES). It was shown that Co, Cu and Sb are not leached out from the ash in H_2O to any notable extent, except for Sb if the leaching is continuous for 24 h (about 40% for CFB boiler ash from waste wood). If the leaching agent is changed from H_2O to 5% HNO_3 solution, total leaching of Co, Cu, and Sb will reach roughly 40-70% of the original content. If the initial H_2O leaching stage is longer (24 h), larger fractions of Co, Cu and Sb will dissolve compared to shorter H_2O leaching stages (30 min). It was shown that Co, Cu and Sb can be leached out from the ash in acids. However, more experiments are needed to optimize the leaching processes.

In the present study, the speciation and volatilization of Co, Cu, and Sb in thermal conversion of SRF at various temperatures and air/fuel ratios were studied using thermodynamic equilibrium modeling. The main goal was to determine if there are conditions in the combustion unit where it is possible to separate and enrich these elements in relation to the bulk ash composition. Based on the predicted results, cobalt occurs mainly in the condensed phases and is not feasible for enrichment in fly ash or filter ash. Copper is enriched in the gas phase at especially oxidizing conditions if chlorine levels in the fuel is high. Controlled conditions in relation to local temperature of

the flue gas and air staging are possible methods to form fly ash or filter ash enriched in copper compounds. In general, concentration of copper is considerably higher than antimony and cobalt in waste-derived fuels. Antimony is predicted to be mainly in the flue gas except at oxidizing conditions below 600 °C, and it is therefore possible to enrich antimony in filter ash if a hot cyclone is utilized to separate condensed fly ash particles from the flue gas stream that contains the volatilized ash forming elements.

The effect of sulfur and chlorine on the ash chemistry was not very pronounced in the present study. Higher chlorine levels increased volatility of alkali species, Pb, Zn, and Co, but Cu and Sb were not affected to any large extent. Increase of sulfur levels had only minor effects. Complete removal of sulfur and chlorine decreased the volatility of Pb and the alkali metals at lower temperatures.

As a final example to study the possible overall chemistry of ash phases and flue gas in SRF combustion, a case where the condensed fly ash/bottom ash composition as well as the possible compounds condensating from the flue gas was calculated. The calculated condensed phases at 900 °C show that Cu and Sb are predicted to be in the gas phase, whereas the Co concentration in the condensed phases (bottom ash/condensed fly ash) is only 3 ppm. If the corresponding flue gas is cooled to around 200 °C or lower, the Co concentration in the condensate is only 1 ppm, but the Sb concentration is 1630 ppm and the Cu concentration is 11130 ppm (Fig 4).

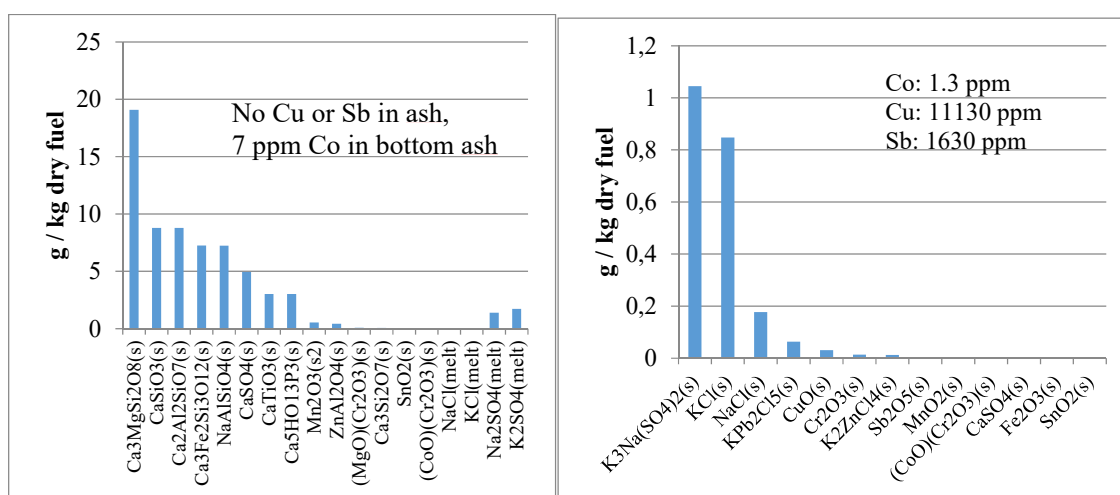


Figure 4. Left graph shows the predicted stable condensed phases at air/fuel ratio=1.2 and 900 °C; right graph shows predicted stable condensed phases that will form if the flue gas is cooled from 900°C to 200 °C.

This suggests that Cu and Sb concentrations in filter ash condensates are considerably higher than the levels in the bulk fuel. However, uncertainties in the calculations stemming from missing or uncertain thermodynamic data, or applicability of the thermodynamic equilibrium assumption for the predictions, are issues that need to be studied, and the predicted results need to be verified with experimental data.

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