Continuous leaching and analysis and high temperature thermodynamic calculations to predict separation of valuable elements from biomass and waste ash streams

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Abstract

Recovery of precious trace elements from different industrial waste or side streams is important in realizing the goals of the circular economy. Various ash streams from thermal conversion processes of solid fuels (e.g. coal, biomass, municipal solid waste) can contain high levels of toxic trace elements that need to be immobilized or collected before the ashes can be further utilized or safely disposed of. Valuable elements can be separated during the combustion process based on the volatilization and subsequent condensation of their compounds, as well as being separated in post-combustion processes, through either thermal treatment or leaching treatments with various solvents.

In the present study, the speciation and leaching behavior of the ash forming elements from combustion of MSW and demolition wood, with special focus on Co, Cu, and Sb, were studied using thermodynamic modelling and a novel continuous leaching and analysis method. The leaching tests were done in a flow-through reactor combined with an ICP-OES that enables fast analysis and determination of elements leached and the leaching rates. Different fly ash fractions were collected at different conditions and with different Cl-content in the fuel (demolition wood).

The thermodynamic predictions showed that Sb has the highest potential to be separated and enriched in the fly ash streams for further treatment and recovery. In the leaching tests, only Sb was leached in pure H_2O, whereas Co, Cu and Sb were leachable in a HNO_3 solution. The leached Cu was shown to depend on the cyclone temperature, where the ash was collected from, with an increasing leachability at lower cyclone temperatures.

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

1. 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 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 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

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. It has been 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 [8-10]. In the present study, where ash from demolition wood fuel was studied, Cu had the highest concentration in the ash and Co had the lowest.

2. Methods

2.1 Procedure for continuous leaching and analysis of ashes

A procedure for continuous analysis of elements leached from fly ashes using different leaching agents has been developed previously [8, 11]. This method enables for fast analysis and determination of elements leached using different solvents. A scheme of the procedure is shown in Figure 1. Similar setups have been used before in chemical fractionation studies of fuels [12, 13].

A flow through reactor was used in the procedure, however various reactor types can be used. Using the flow through reactor, the solvent is pumped through a plug of ash and the elements leached are analyzed continuously using ICP-OES. The ash is kept in place with wool buds and teflon filters were used to avoid particles from escaping the reactor. Water was used as the first leaching agent to determine the water soluble elements. 0.50 g of ash was placed in the reactor in the experiments and the flow through the ash was 0.6 ml/min. A total of 22 elements were analyzed every 16 s, those were: Ag, Al, As, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, P, Pb, S, Sb, Se, Si, V, and Zn. After 30 min of leaching with water, a solution of 5% HNO_3 was pumped through the ash for 2 h 10 min. The weight loss during the leaching procedure was determined and the residual ash was analyzed using SEM/EDX. For the weight loss determination, the ash was dried in a furnace at 105°C for 24 h.

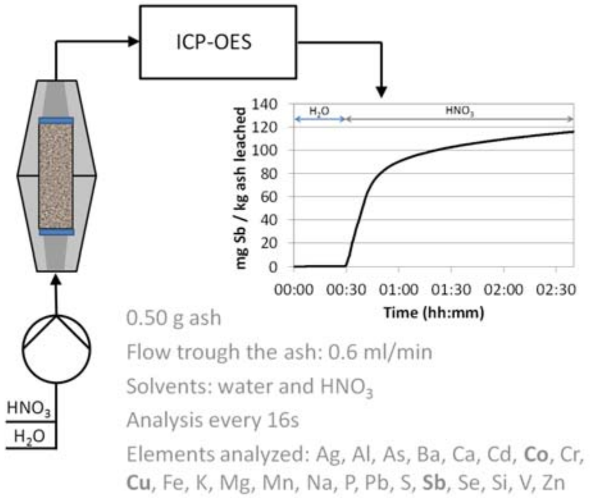

Figure 1. Scheme of the procedure for continuous leaching and analysis of ashes using a flow through reactor and ICP-OES.

2.2 Ashes tested

Five ashes were tested and the Cu, Co, Sb contents are given in Table 1. The ashes originated from test runs made with the 20 kW bubbling fluidized bed reactor at VTT Technical Research Centre of Finland in Jyväskylä, Finland. The reactor setup has previously been described by Aho et al. [14]. Ash T1 was from the co-combustion of demolition wood and plastic cables, and ashes T2-T5 were from combustion of demolition wood. The cyclone temperatures were adjusted for the different cases to: T1 and T2 650°C, T3 600°C, T4 500°C, and T5 450°C. The ashes were first leached with water for 30 min and then with a 5% HNO_3 water solution for 2 h and 10 min. Further, a sixth test was done with the ash designated T5 where it was leached for 24 h with water before the HNO_3 step. This was done to understand how the leaching of the water-soluble elements affects the acid leaching step of Sb, Co, and Co.

Table 1. Cu, Co, and Sb contents in the ashes

Ash	Cu (mg/kg)	Co (mg/kg)	Sb (mg/kg)
CFB T1	990	48	390
CFB T2	1200	51	350
CFB T3	1400	53	400
CFB T4	1500	54	400
CFB T5	1400	52	370

3. Results

3.1 Ashes T1-T5

The leaching behavior of ashes T1-T5 are similar. Some Sb is leached during the first 30 min. of water leaching, however, no Cu or Co was leached. When HNO_3 leaching starts at 30 min., Cu and Co are also leached. The leaching rate is high for the first 15 min. with HNO_3. The cumulative amounts of leached Co, Cu and Sb as a function of time for T1 are shown in Fig. 2. The ashes T2-T5 show similar behavior. The composition of the solid ash before and after the leaching are also shown for the corresponding ash. The fraction of leached Co was between 50 and 56%, and for Cu it varied between 40 and 57%. Sb showed slightly larger variations, with the leached fraction varying between 40 and 74%. The results for each ash is shown in Fig. 4. It was observed that all P, S, and Cl were leached out in the tests.

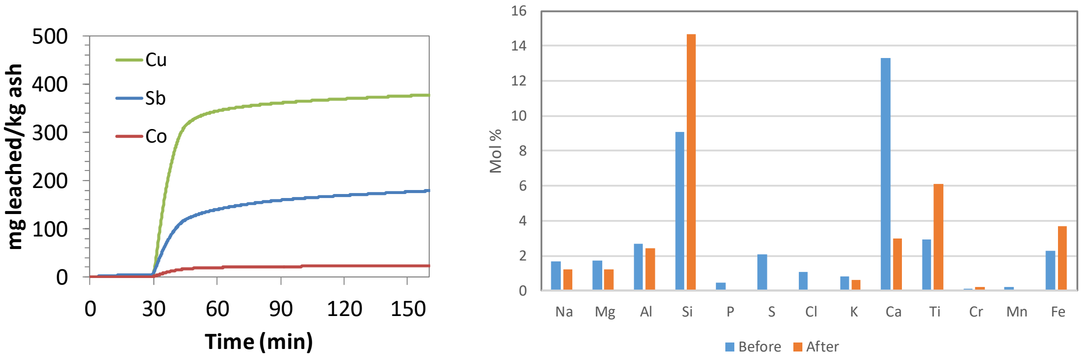

Figure 2. Leaching results of Co, Cu, and Sb of T1 ash in ascending order. The ashes were leached with water for the first 30 min and then with a 5% HNO_3solution. The graph on the left show the cumulative amount of leached Co, Cu, and Sb and the graph on the right show the chemical composition of the original ash and the residual ash after leaching.

3.2 Ash T5 with 24h water leaching before HNO_3

One experiment with a longer, 24 h, water-leaching step was made. During the 24h leaching the elements were not analyzed continuously. 40% of Sb was leached in the water leaching step, however, no Co or Cu was leached out (Figure). Significant amounts of Ca, K, Na, and S were also leached in the water leaching. After the water leaching, the ash was leached with 5% HNO_3. The acid leaching seems to be enhanced by the long water leaching step. The leaching of Co, Cu, and Sb is fast (Fig 4), and the total percentages of Co, Cu, and Sb leached were: 67, 66, and 79%.

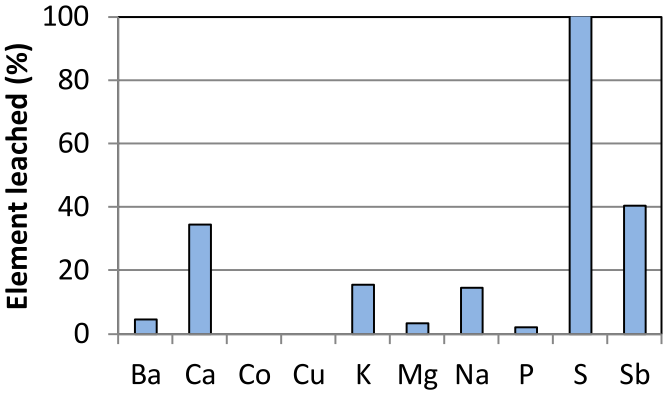

Figure 3. Elements leached during the 24 h water leaching.

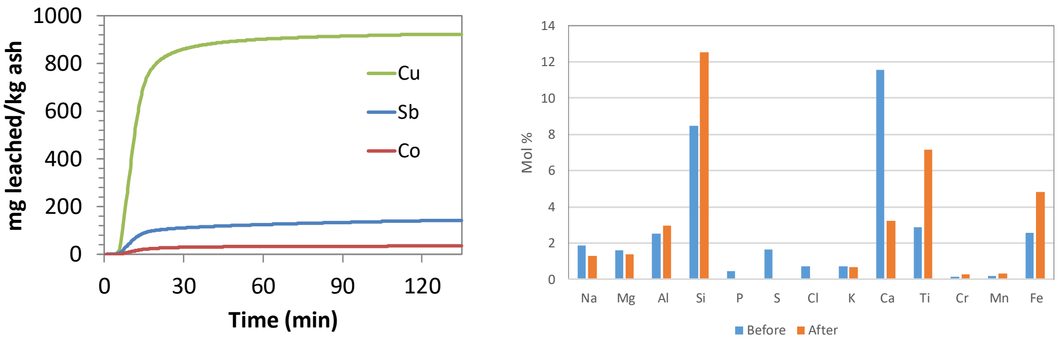

Figure 4. Leaching results with 5% HNO_3-solution of Co, Cu, and Sb of T5 ash. The ash was leached with water for the first 24 h (results in Figure 3) and then with a 5% HNO_3-solution. The graph on the left shows SEM-EDX analysis of T5 ash before and after the leaching procedure.

3.3 *Effect of cyclone temperature on leachability of Cu*

Figure 5 shows the leachabilty of Cu for the different ashes. It seems that Cu is more easily leached for the ashes from the lower cyclone temperature cases.

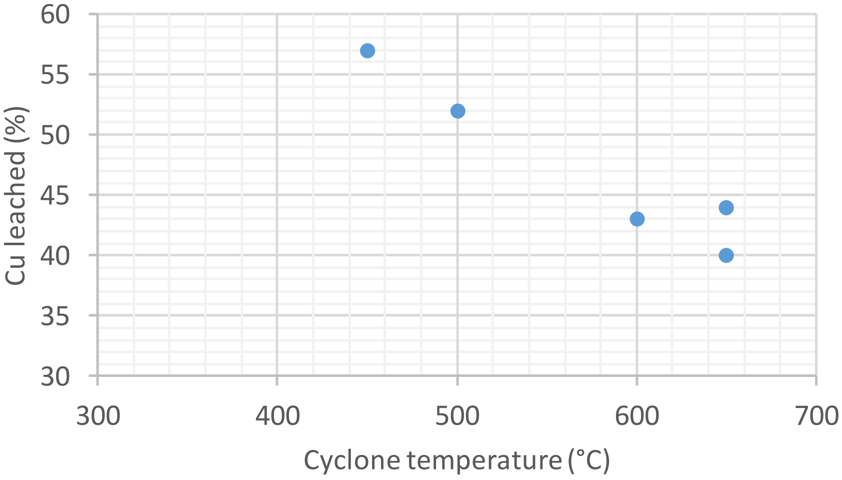

Figure 5 Leaching of Cu as a function of cyclone temperature.

3.4 Speciation of Cu, Co, Sb in ash

The speciation of Cu, Co, and Sb can be determined with various experimental methods, such as X-ray diffraction based on crystallographic structure or SEM/EDXA based on the chemical composition. However, due to the low concentrations of Cu, Co, and Sb in the ash, it is often difficult to identify phases containing these elements. Cu typically occurs in higher concentrations compared to Co and Sb, and is therefore the most likely to identify in a phase.

Talonen [15] studied the speciation of several trace elements in MSW combustion and gasification by using thermodynamic modeling to predict the phase equilibrium and the composition of the condensed phases and the gas phase at temperatures varying between 200 °C and 1600 °C. The study emphasized the differences when using different thermodynamic databases.

Cobalt was predicted to occur mainly as solid $CoSO_4$, Co_3O_4, or CoO and gaseous $CoCl_2$ and $Co(OH)_2$ at oxidizing conditions, whereas different cobalt sulfides or metallic Co are the stable solid phases at reducing conditions.

Copper was predicted to occur mainly as solid $CuSO_4$, CuO or $CuO \cdot CuSO_4$ phase and gaseous CuCl as well as minor amounts of other gaseous copper halides at oxidizing conditions. At reducing conditions, solid Cu_2S or metallic Cu is predicted to be the main stable phases.

Antimony was predicted to be stable mainly as solid Sb_2O_5 at lower temperatures and as various gaseous Sb-O compounds at higher temperatures (SbO, Sb_4O_6) at oxidizing conditions. At reducing conditions, solid Sb_2S_3 is stable upt to around 400 °C, with gaseous Sb, SbCl or SbO are the main stable compounds at higher temperatures.

Lassesson & Steenari [16] have analyzed the speciation of copper in bottom ash, cyclone ash and filter ash from a MSW-fired BFB boiler using synchrotron based X-ray absorption spectrometry. They concluded that the most common copper compounds in the bed ash were metallic copper, Cu_2O, CuO, and mixed oxides, such as $CuCr_2O_4$. The cyclone ash contained a mix of metallic copper, Cu_2O, CuCl, $Cu(OH)_2$, $CuSO_4 \cdot 5H_2O$, and possibly CuO. Copper sulfate was the major species in the filter ash together with hydroxides and chlorides. Lasseson et al. [17] analyzed the speciation of Cu in two different MSWI fly ashes, one rich in Ca and Cl with a Cu-concentration at 5.4 g / kg dry ash (Ash A), and the second fly ash with high levels of Ca, K, Na, S, Si, and Cl, containing 2.5 g Cu / kg dry ash (Ash B). The copper speciation in Ash A was mainly $CuSO_4 \cdot 5H_2O$, with minor amounts of CuClOH, whereas Ash B contained a mainly $Cu_3(PO_4)_2$ and minor amounts of metallic Cu, Cu_2S, and CuO.

Miravet et al. [18] determined that most of the soluble antimony in coal fly ash was in the form of Sb(V), with only minor amounts of Sb(III) occurring in the leachate.

In this study, thermodynamic predictions of the speciation of ash-forming elements have therefore been made to estimate in which form Cu, Co, and Sb can be found in the ash. The predictions were made with the thermodynamic software package Factsage [19] version 6.4 using the FactPS database for thermodynamic data of the phases and compounds that may form. The estimations were made based on the ash composition of the BFB and CFB ashes given in Vainio et al. [8]. The calculations give the stable phases at 25 °C, for both dry ash and for ash with 10 wt% H_2O, simulating slightly hydrated ash. The predicted phase composition is given in table 3.

Table 3. Predicted ash phases for the three different ashes at 25 ℃.

BFB1_dry		BFB1_10% H2O		BFB2_dry		BFB2_10% H2O		CFB ESP_dry		CFB ESP_10% H2O	
Al_2SiO_5	21.7	$KAl_3Si_3O_{10}(OH)_2$	25.1	$Ca_3Al_2Si_3O_{12}$	22.5	$Na_2Ca_3Si_6O_{16}$	16.4	$Ca_3Fe_2Si_3O_{12}$	21.7	$Ca_3Fe_2Si_3O_{12}$	22.3
$Na_2Ca_3Si_6O_{16}$	18.8	$Na_2Ca_3Si_6O_{16}$	18.6	$CaSO_4$	19.3	$CaSO_4(H_2O)_2$	14.7	Ca_2SiO_4	19.7	$Ca(OH)_2$	18.1
$KAlSi_3O_8$	17.3	$CaAl_2Si_2O_7(OH)_2(H_2O)$	18.0	$CaMgSiO_4$	10.0	$Ca_3Al_2Si_3O_{12}$	13.6	$Ca_3MgSi_2O_8$	16.9	$Na_2Ca_3Si_6O_{16}$	14.0
$CaMgSi_2O_6$	10.9	H_4SiO_4	15.3	$Ca_3Fe_2Si_3O_{12}$	9.7	$Ca_3Fe_2Si_3O_{12}$	8.8	$CaSO_4$	11.3	$CaSO_4(H_2O)_2$	10.0
Fe_2O_3	8.1	Fe_2O_3	8.0	$Na_2Ca_2Si_3O_9$	7.4	$CaCl_2(H_2O)_6$	8.4	$CaTiO_3$	7.5	$CaTiO_3$	6.8
$Ca_3(PO_4)_2$	7.6	$Mg_5Al_2Si_3O_{10}(OH)_8$	5.6	$KCaCl_3$	7.3	$Mg_5Al_2Si_3O_{10}(OH)_8$	6.5	$CaMg_2Al_{16}O_{27}$	6.6	$Mg_5Al_2Si_3O_{10}(OH)_8$	6.6
SiO_2	7.3	SiO_2	3.1	$CaTiO_3$	7.1	$CaTiO_3$	6.4	$ZnAl_2O_4$	3.0	$CaCl_2(H_2O)_6$	3.8
$CaSiTiO_5$	2.2	$CaSO_4$	2.0	$ZnAl_2O_4$	6.4	$CaSO_4$	5.9	KCl	2.8	$Al_2O_3(H_2O)$	3.7
$CaSO_4$	2.1	$Ca_{10}(PO_4)_6F_2$	1.2	$Na_2Ca_3Si_6O_{16}$	4.5	$ZnAl_2O_4$	5.8	$Na_2Ca_2Si_3O_9$	2.6	$Ca_3Al_2Si_3O_{12}$	3.3
$Ca_{10}(PO_4)_6F_2$	1.3	Others	2.9	$Ca_{10}(PO_4)_6F_2$	1.8	$Ca(OH)_2$	5.6	NaCl	2.2	$ZnAl_2O_4$	2.7
Others	2.6			Others	3.6	KCl	3.4	$Ca_{10}(PO_4)_6F_2$	1.2	KCl	2.5
						$Ca_{10}(PO_4)_6F_2$	1.6	$(CaO)_2(PbO_2)$	1.2	$CaSO_4$	2.3
						Others	2.7	Fe_2O_3	1.0	$Ca_{10}(PO_4)_6F_2$	1.1
								Others	2.2	Others	2.7
CuO	0.06	$Cu(OH)_2$	0.07	CuO	0.3	$Cu(OH)_2$	0.3	CuO	0.19	$Cu(OH)_2$	0.21
Sb_2O_5	0.004	Sb_2O_5	0.004	Sb_2O_5	0.02	Sb_2O_5	0.02	Sb_2O_5	0.02	Sb_2O_5	0.02
Co_3O_4	0.003	Co_3O_4	0.003	Co_3O_4	0.004	Co_3O_4	0.004	Co_3O_4	0.004	Co_3O_4	0.004

The predictions of the main phases show that various Ca-Al-Fe silicates, as well as $CaSO_4$ and gypsum, are the dominating phases. The main highly soluble phases are KCl, NaCl, $CaCl_2(H_2O)_6$, and $KCaCl_3$.

The predicted speciation of Cu is CuO in completely dry ash, and $Cu(OH)_2$ in humid ash, whereas Sb occurs as Sb_2O_5 in all cases and Cu occurs as Co_3O_4 in all cases.

Similar predictions for the ash speciation at 500 °C give similar results for the bulk ash composition (Table 4). The difference between dry ash and humid ash is minimal due to the instability of hydrated species at these temperatures. The predicted speciation of Cu, Co, and Sb is slightly different compared to the low-temperature case. Sb occurs as Sb_2O_5, whereas Cu is predicted to form either CuO or $CuO{\cdot}Fe_2O_3$ (or $CuFe_2O_4$, cuprospinel), and Co is predicted to form Co_3O_4 or $CoO{\cdot}Cr_2O_3$ (or $CoCr_2O_4$).

Table 4. Predicted ash phases for the three different ashes at 500 ℃.

BFB1_dry		BFB1_10% H2O		BFB2_dry		BFB2_10% H2O		CFB ESP_dry		CFB ESP_10% H2O	
$CaAl_2Si_2O_8$	29.1	$CaAl_2Si_2O_8$	30.2	$Ca_3Al_2Si_3O_{12}$	22.3	$Ca_3Al_2Si_3O_{12}$	22.4	$Ca_3MgSi_2O_8$	21.5	$Ca_3MgSi_2O_8$	21.5
$KAlSi_3O_8$	17.0	$KAlSi_3O_8$	19.3	$CaSO_4$	19.3	$CaSO_4$	19.3	$Ca_2Al_2SiO_7$	18.7	$Ca_2Al_2SiO_7$	18.7
$NaAlSi_3O_8$	16.7	$NaAlSi_3O_8$	18.2	$CaSiO_3$	11.6	$CaSiO_3$	11.6	$Ca_3Si_2O_7$	13.2	$Ca_3Fe_2Si_3O_{12}$	13.2
$CaMgSi_2O_6$	10.5	$CaMgSi_2O_6$	10.8	$Ca_3Fe_2Si_3O_{12}$	9.7	$Ca_3Fe_2Si_3O_{12}$	9.7	$Ca_3Fe_2Si_3O_{12}$	13.1	$Ca_3Si_2O_7$	13.0
Fe_2O_3	8.0	Fe_2O_3	8.7	$CaMgSiO_4$	8.5	$CaMgSiO_4$	8.5	$CaSO_4$	10.3	$CaSO_4$	10.3
$Ca_3(PO_4)_2$	7.6	SiO_2	3.6	$CaTiO_3$	7.1	$CaTiO_3$	7.1	$CaTiO_3$	7.5	$CaTiO_3$	7.5
SiO_2	3.2	$CaSiTiO_5$	2.4	$ZnAl_2O_4$	6.4	$ZnAl_2O_4$	6.4	$ZnFe_2O_4$	3.9	$ZnFe_2O_4$	3.9
$CaSiTiO_5$	2.2	$CaSO_4$	2.1	NaCl	3.6	NaCl	3.6	NaCl	2.9	NaCl	2.9
$CaSO_4$	2.0	$Mg_2Al_4Si_5O_{18}$	1.4	KCl	3.0	KCl	3.1	KCl	1.9	KCl	1.9
$Ca_{10}(PO_4)_6F_2$	1.3	$Ca_{10}(PO_4)_6F_2$	1.4	$CaMgSi_2O_6$	2.2	$CaMgSi_2O_6$	2.1	$Ca_{10}(PO_4)_6F_2$	1.2	$Ca_{10}(PO_4)_6F_2$	1.2
Others	2.2	Others	1.6	$Ca_{10}(PO_4)_6F_2$	1.8	$Ca_{10}(PO_4)_6F_2$	1.8	$(PbO)(Al_2O_3)$	1.1	$(PbO)(Al_2O_3)$	1.1
				$KCaCl_3$	1.5	$KCaCl_3$	1.4	Others	4.1	Others	4.0
				K_2PbCl_4	1.0	K_2PbCl_4	1.0				
				Others	1.6	Others	1.6				
$(CuO)(Fe_2O_3)$	0.17	$(CuO)(Fe_2O_3)$	0.16	CuO	0.29	CuO	0.3	$(CuO)(Fe_2O_3)$	0.6	$(CuO)(Fe_2O_3)$	0.6
Sb_2O_5	0.004	$(CoO)(Cr_2O_3)$	0.01	Sb_2O_5	0.02	Sb_2O_5	0.02	Sb_2O_5	0.02	Sb_2O_5	0.02
Co_3O_4	0.003	Sb_2O_5	0.004	Co_3O_4	0.004	Co_3O_4	0.004	Co_3O_4	0.004	Co_3O_4	0.004

As a summary, Cu, Co, and Sb are predicted to form oxide phases in the studied ashes, or possibly $Cu(OH)_2$ if the ash is hydrated. None of the metals were predicted to form silicates, sulfates or halides.

4. Summary and conclusions

Five ashes were tested (T1-T5) for leaching various ash elements. The ashes were collected from test runs with the VTT BFB reactor. Ash T1 originated from co-combustion of demolition wood and plastic cables, while the ashes T2-T5 came from combustion of demolition wood. The main emphasis was laid on leaching of Co, Cu, and Sb, however, also 19 other elements were simultaneously analyzed every 16 s. Water was used as the first leaching agent to determine the water soluble elements. 0.50 g of ash was used in the experiments and the flow through the ash was 0.6 ml/min. After 30 min of leaching with water, a solution of 5% HNO_3 was pumped through the ash for 2 h 10 min. Only some Sb was leached during the water leaching step. Co and Cu were leached only when HNO_3 was used as solvent. The leaching rate was faster during the first 15 min with acid. The leaching behaviour was similar for all the ashes (Fig 6). T5 was also tested using a 24 h water leaching step before the acid leaching. 40% of Sb was leached out during this step. The acid leaching step seemed to be enhanced by the long water leaching step, with higher Co and Cu leached out compared to the normal leaching procedure. Leaching out the water soluble elements before the acid step may also have a positive effect on recovering the elements from the leachate, due to a less complex solution.

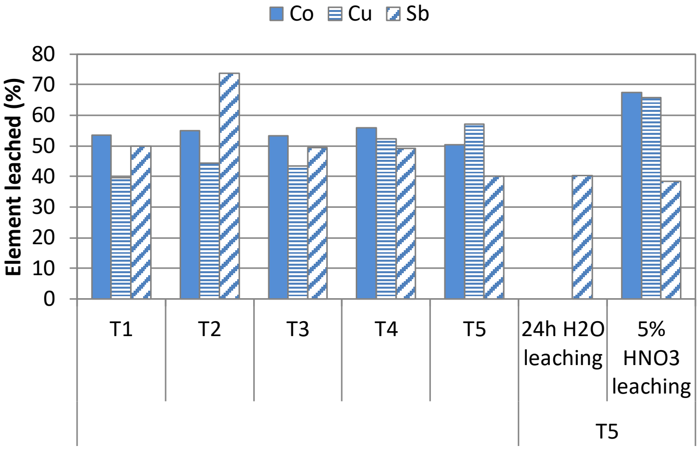

Figure 6. Co, Cu, and Sb leached during the leaching procedure.

A procedure for the continuous analysis of elements leached from fly ashes using different leaching agents has been developed. This method enables fast analysis and determination of elements leached and the leaching rates. A flow through reactor is used in the procedure, however various reactor types can be used. Using the flow through cell, the leaching agents is pumped through a plug of ash and the elements leached are analyzed using ICP-OES. Different solvents can be used in the procedure. Additionally the temperature in the cell can be adjusted. The method enables for rapid testing of different parameters with the on-line analysis of the leached elements. The method can be used to find optimal parameters for leaching various elements.

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