

Separation of heavy metal compounds from solid waste incineration bottom ash by tabling

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S 1.1 Methods and analytical details

The mixing and dividing of sample A was performed with riffle boxes according to the following procedure: i.) separation of the two buckets into four subsamples using a sheet metal cross, ii.) dividing of the four subsamples into eight and then 16 samples, iii.) recombination by cross-riffing back into four samples, iv.) dividing of the samples down to subsamples.

For grain density determination, a He-pycnometer from Micromeritics (AccuPyc 1330) was utilized. Sample masses of 5 to 15 g were used to fill the cell volume of 12 cm³ during double determinations with 1.4 bar He pressure. The values of true density for calculating porosities were measured analogously, using previously milled samples (described below). Bulk densities were determined using a funnel equipped with a slide valve and a 500 mL fixed volume cup.

The sulfate and chloride contents of process water samples were analyzed by RFIC-EG (reagent-free ion chromatography with eluent generation) using a Dionex Integrion from Thermo Fisher Scientific. For analysis 5 µL sample solution (filtered, 45 µm) was introduced at 30 °C to an IonPac AS20 (2x250mm) column with Ag20 (2x50mm) pre-column from the same company. Eluent generation (CR-TC 600) was done by increasing KOH concentration from 10 to 35 mM and quantification was performed by conductivity detection.

Samples were milled prior to elemental analysis, by using a vibration mill from Retsch (MM2000) with one ceramic grinding ball (d=20 mm) for 4 min at 70 rpm. From each fraction three samples of 10 g were analyzed and averaged for sufficiently high accuracy. Milled ash samples were filled into cuvettes with 24 mm inner diameter (Spectro) and sealed beneath using 4 µm polypropylene foil from Fluxana (TF-240). Analysis was performed on a Xepos Benchtop XRF (X-ray fluorescence) spectrometer from Spectro Analytical Instruments, with 35 kV and 0.3 mA for X-Ray generation and TurboQuant-Powders method for analysis.

Dried (24 h, 80 °C) bottom ash was screened on a AS 200 control sieving machine from Retsch (4 min at 1.5 mm amplitude) with test sieves of 2 mm, 1 mm, 630 µm, 200 µm, 100 µm and 63 µm mesh size from the same manufacturer. Obtained sieve fractions were weighed and analyzed regarding elemental composition and grain density.

For evaluation of the remaining organic content, loss on ignition was determined by calcination of 15 g pre-dried (105 °C) sample. Thermal treatment conditions were 2 to 3 h at 550 °C until constant weight. Samples were stored in a desiccator during the cool-down period.

The content of carbonate was determined by conversion into CO₂, using 5 g milled sample and 10 mL HCl_{aq} (10% v/v). The resulting pressure after 5 min was measured and compared to a previous calibration with CaCO₃ (500 mg). Used device was constructed by the company Klosa and applied 500 mL glass bottles as reaction vessel.

Conductivity and pH value were examined using 5 mL solid sample shaken for 2 h in 25 mL ultrapure water. After 10 min sedimentation and subsequent decantation, pH value (pH 526, WTW) and conductivity (LF 537, WTW) were measured. The moisture contents of bottom ash were determined by drying 1 g sample at 105 °C until constant weight. Threefold measurements were performed with a Satorius MA-40 device.

ATR-IR (attenuated total reflection infrared) spectroscopy was done with a 2000 FT-IR device from PerkinElmer equipped with a DuraSamplIR 2 from SensIR Technologies. X-Ray diffraction patterns were collected on a XRD 3000 PTS from Seifert using a wavelength of 1.79 Å in a θ range of 10 to 120°. Optical microscope images were collected with a VHX 1000 from Keyence, using the objectives VH-Z20R for magnifications of 20-200 and VH-Z250R for magnifications of 250-2500. For SEM (scanning electron microscope) images with EDX (energy dispersive X-Ray) spectroscopy mapping, a Gemini1530VP microscope from Zeiss was used with an EDX-XFlash detector 5030 from Bruker. The sample was fixed to a carbon pad and subsequently sputtered with carbon prior to measurement at 20 kV in high vacuum.

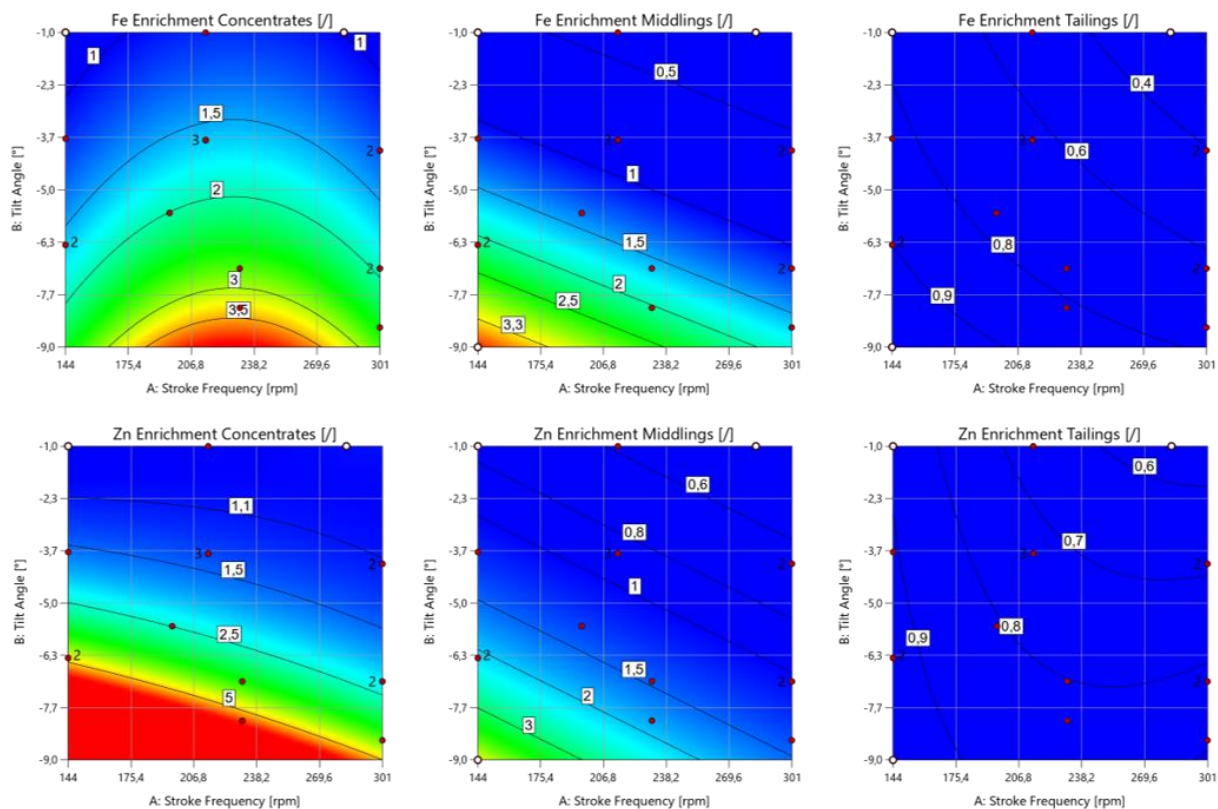


Fig. S1 Contour plots for the iron and zinc enrichment of sample A; color gradient ranges: top 1-4, bottom 1-6

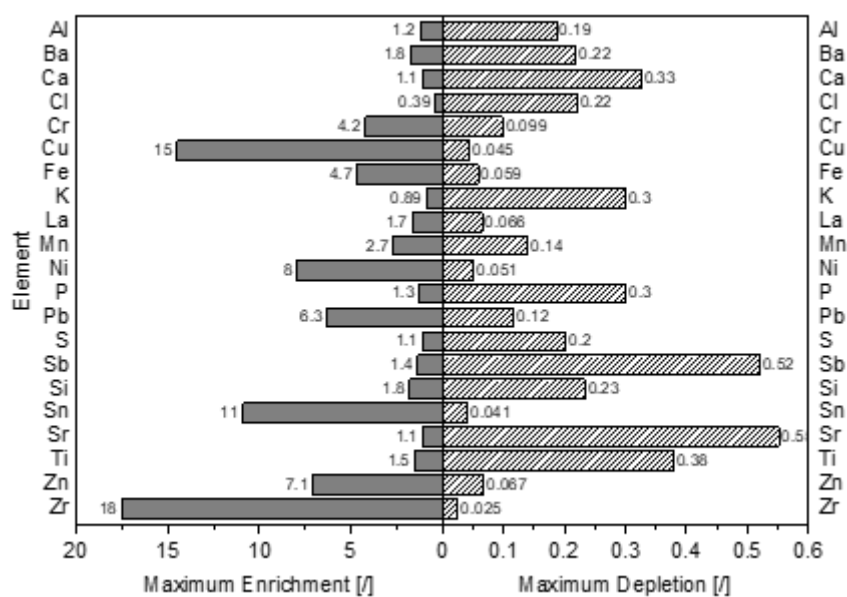


Fig. S2 Maxima of achieved element enrichment and depletion for sample A

Table S1 Absolute maxima of achieved enrichments, together with the respective element concentrations for sample A; shown values may correspond to different individual experiments

	Element	Initial content [wt.%]	Maximum content [wt.%]	Enrichment []
Concentrates	Co	0.006	0.014	2.26
	Si	9.254	16.560	1.79
	Ba	0.263	0.463	1.76
	Ti	0.780	1.207	1.55
	P	0.483	0.626	1.30
Middlings	Cd	0.000	0.011	24.97
	Zr	0.018	0.311	17.54
	Cu	0.259	3.760	14.52
	Sn	0.014	0.157	10.89
	Ni	0.021	0.167	7.98
	W	0.004	0.032	7.58
	Zn	0.609	4.311	7.08
	Pb	0.075	0.472	6.30
	V	0.005	0.027	5.53
	Fe	5.625	26.343	4.68
	Cr	0.056	0.239	4.23
	Mn	0.145	0.392	2.70
	Sb	0.010	0.013	1.36
	Al	4.956	6.066	1.22
	S	2.329	2.551	1.10
	Ca	15.021	16.327	1.09
	K	0.524	0.469	0.89
Tailings	La	0.011	0.018	1.65
	Sr	0.037	0.040	1.08
	Cl	1.084	0.426	0.39