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ANTIMONY AND VANADIUM IN INCINERATION BOTTOM ASH – LEACHING BEHAVIOR AND CONCLUSIONS FOR TREATMENT PROCESSES

Franz-Georg Simon *, Christian Vogel and Ute Kalbe

Division 4.3 Contaminant Transfer and Environmental Technologies, Bundesanstalt für Materialforschung und -prüfung (BAM), 12200 Berlin, Germany

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ABSTRACT

Due to its large mineral fraction, incineration bottom ash (IBA) from municipal solid waste incineration is an interesting raw material that can be used for road construction or to produce secondary building materials. However, leaching chloride, sulfate, and potentially harmful heavy metals may cause problems in using IBA in civil engineering. Investigating leaching behavior is crucial for the assessment of the environmental compatibility of IBA applications. Various test procedures are available for that purpose. In the present study, a long-term leaching test of a wet-mechanically treated IBA was performed in a lysimeter for almost six years. While concentrations of chloride, sulfate and the majority of the heavy metals started to decrease rapidly with progressive liquid-to-solid ratio (L/S), antimony (Sb) and vanadium (V) behaved differently. At the beginning of the lysimeter test, the Sb and V concentrations were low, but after approximately one year of operation at an L/S ratio of around 0.8 L/kg, a steady increase was observed. It was shown that this increase is the result of low Ca concentrations due to the formation of $CaCO_3$. With the data, the solubility products from Ca-antimonate and Ca-vanadate were calculated. The unusual leaching behavior of Sb and V should be kept in mind when considering field scenarios and evaluating the impact on the environment.

1. INTRODUCTION

Around 26 million metric tons of combustible waste were incinerated per year in Germany in 100 thermal waste treatment plants (66 of which are municipal solid waste incineration plants) (BDE et al., 2018). According to information from the German Association of Thermal Waste Treatment (Interessengemeinschaft Thermische Abfallbehandlung, ITAD) (ITAD, 2019), around 5.7 million tons of incineration bottom ash (IBA) from municipal solid waste incinerators were generated in Germany in 2017. In the course of processing bottom ash, 382,000 tons of iron (Fe) and 95,000 tons of non-ferrous metals (NFe) were separated annually in accordance with state of the art techniques (Verein Deutscher Ingenieure, 2014). Due to the mostly applied wet extraction out of the furnace chamber, these metals are often integrated in a heterogeneous and unstable matrix (Šyc et al., 2020). In general, the recovery of elemental metals is still a challenge in terms of recovery rate and purity.

The vast majority of IBA is made up of the mineral fraction (90%), in which the metals with a high affinity for oxygen (calcium, aluminum, magnesium, etc.) are bound in oxidic form in the bottom ash and cannot be recovered economically in elemental form. In terms of their composition, however, these oxides are similar to raw materials that can be used to produce secondary building materials (Bayuseno & Schmahl, 2010; Bunge, 2018). Large quantities of the mineral fraction are reused for sub-base material in road construction (Di Gianfilippo et al., 2018; Hyks & Šyc, 2019). A three-month ageing period has been established as the common practice for further treatment of bottom ash before reuse applications. In the course of this ageing, the pH value of bottom ash decreases, and contaminants are immobilized by processes like carbonation, hydration, and oxidation. In particular, the leaching of heavy metals is reduced to environmentally acceptable levels as required for disposal of IBA on landfills (Simon et al., 1995). However, leaching chloride, sulfate, and potentially harmful heavy metals may cause problems in using IBA in civil engineering. The legal requirements for using IBA in Europe are described in detail elsewhere (Blasenbauer et al., 2020). Some countries have set limit values for the total content measured after acidic digestion and/or limit values for elu-

* Corresponding author:
Franz-Georg Simon
email: franz-georg.simon@bam.de

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ate concentrations. To assess leaching behavior, a number of test methods are statutory, so that for a comparison, the limit values for concentrations in mg/L have to be converted to leached content E in mg/kg by multiplying by the respective liquid-to-solid ratio (L/S in L/kg), commonly 2 L/kg or 10 L/kg.

Leaching tests can be performed as batch tests (e.g. according DIN EN 12457-2 (EN 12457-2, 2002-09) with an L/S of 10 L/kg) or as column tests (e.g. according to DIN 19528 (DIN 19528, 2009-01) up to an L/S ratio of 4 L/kg for basic characterization or an L/S ratio of 2 L/kg for the column test in the compliance test option in Germany). In batch tests, the concentrations in the eluate are analyzed after a fixed agitation period (usually 24 h), whereas in column tests the eluate is generated continuously over the complete test duration. Concentrations can be determined for individual L/S ratios. DIN 19528 prescribes the L/S ratios 0.3, 1.0, 2.0, and 4.0 L/kg. Rather than in batch tests, the dynamic behavior of leaching can be assessed by column tests (Grathwohl & Susset, 2009). However, column tests are more time-consuming. Information on long-term leaching behavior can be gained from lysimeter experiments, which take even more time (Krüger et al., 2012). However, lysimeter tests are better for simulating field scenarios and long-term leaching behavior than laboratory column tests, particularly due to the unsaturated conditions and realistic contact time with the leachant (Krüger et al., 2012; López Meza et al., 2010).

Previously, it has been shown that the elements antimony (Sb) and vanadium (V) exhibit unusual leaching behavior in leaching tests (Cornelis et al., 2006, 2012). Total contents of IBA are reported with an average of 73 mg/kg (range from 18-250 mg/kg) for Sb and 41 mg/kg (19-248 mg/kg) for V, respectively (Hjelmar et al., 2013). The source for Sb is very likely plastic in municipal solid waste, in which it is used as a flame-retardant agent or catalyst (Okkenhaug et al., 2015). In addition, Sb is a critical element, because released toxic Sb(V) from secondary construction materials can sorb onto inorganic oxides and organic matter in soils (Diquattro et al., 2021) which may affect environmental health by ecotoxic effects and bioaccessibility (Bagherifam et al., 2021). Therefore, to analyze the long-term leaching behavior of Sb and V we performed a lysimeter experiment with treated IBA which was performed for almost 6 years up to an L/S ratio of 4.1 L/kg.

2. MATERIALS AND METHODS

The IBA material for the experiments was sampled on November 5, 2013 at a municipal solid waste incineration (MSWI) bottom ash treatment plant in Germany. Fe and NFe metals had already been removed by standard methods (magnetic and eddy current separation). A wet-mechanical process step was implemented in the plant to remove the finest fraction below 0.25 mm, amounting to approximately 10% (Holm & Simon, 2017). This finest fraction contained around 60% of the initial sulfate (Kalbe & Simon, 2020) and is usually landfilled. Two mineral fractions (0.25-4 mm and 4-60 mm) were generated by different sieving steps. The mineral fractions from 0.25-4 mm and from 4-60 mm were sampled, air-dried, and homogenized separately. Material larger than 45 mm was removed from the 4-60 mm fraction by sieving. Representative subsamples of both fractions were generated by partitioning the sample and then a mixed sample at a ratio of 40:60 (0.25-4 mm:4-45 mm) was assembled to the final 0.25/45 mm test material employed for the leaching experiments reported here. This condition was expected as the status on an intermittent storage site before reuse of such material.

Two laboratory scale lysimeters (30 cm in diameter) were operated with approximately 60 kg IBA each from March 2014 until February 2020 to investigate the long-term leaching behavior of a 0.25/45-mm mineral material obtained by using a wet processing technology to treating IBA directly after incineration. The resulting bulk density of the sample in the lysimeters was 1.46 g/cm³; the corresponding porosity was 45%; and one pore volume amounted to 18.4 L. Artificial rainwater was used as leachant (pH ≈ 6, constituents NO_3^{2-}, Cl^-, SO_4^{2-}, Na^+, K^+, Ca^{2+}, Mg^{2+}) (Sanusi et al., 1996). The lysimeters were irrigated in proportion to an average annual precipitation rate of 600 mm/a, leading to a liquid to solid ratio (L/S) of about 0.7 L/kg per year of operation. The L/S ratios of the two lysimeter experiments finally reached after almost six years were 4.17 (lysimeter 1) and 4.09 (lysimeter 2) L/kg, respectively. The lysimeters were then dismantled and the remaining material analyzed for their main constituents.

Eluates were aliquoted after sampling. For chemical analysis, one aliquot was preserved using concentrated nitric acid (DIN EN ISO 5667-3, 2013-03) to measure cations, and one aliquot remained untreated for anion analysis. The eluates were stored at 4° C until measurement. pH values were measured with a Schott CG 841 pH-meter equipped with a WTW SenTix 41 pH electrode (DIN ISO 10390, 2005-12), the electric conductivity with a WTW LF 437 microprocessor conductivity meter (DIN ISO 11265, 1997-06), and turbidity with a HACH 2100N turbidity meter (DIN EN ISO 7027, 2016-11).

Cations (Al, As, Ba, Ca, Cd, Co, Cr, Cu, Fe, Hg, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Sn, Sr, V, Zn, Se) were quantified using an iCAP 7000 ICP-OES equipped with an ASX-260 Autosampler (Thermo Scientific, Dreieich, Germany) in accordance with (DIN EN ISO 11885, 2009-09). ICP-MS measurements were performed on 10-fold dilutions with an iCAP Q (Thermo Scientific, Dreieich) in accordance with (DIN EN ISO 17294-2, 2017-01) for elements below the detection limit of the ICP-OES. Results for the elements not reported in this work can be found elsewhere (Kalbe & Simon, 2020).

Anions (Cl^-, SO_4^{2-}) were determined with the Dionex IC320 ion chromatograph with the AS 40 Autosampler, ASRS 300 suppressor, AS-9-HC separation column with column oven, and a conductivity detector in accordance with DIN EN ISO 10304 (DIN EN ISO 10304-1, 2009-07). The separation was isocratic with a mixture of carbonate/bicarbonate in aqueous solution (8 mmol/L / 1 mmol/L) as eluent.

3. RESULTS

It is known that the leaching behavior of IBA strongly depends on the pH (Chandler et al., 1997). Leaching tests with unaged IBA result in pH values above 12, due to the $Ca(OH)_2$ content. At high pH values, various heavy metals exist in a soluble form, e.g. $Pb(OH)_4^{2-}$, so that leaching limits even for landfilling may be exceeded (Simon et al., 1995). During the required ageing period of three months, $Ca(OH)_2$ reacts with CO_2 from the air and rain to form $CaCO_3$, leading to lower pH values and usually lower heavy metal release (Schnabel et al., 2021), e.g. Pb forms sparingly soluble $PbCO_3$ or $Pb(OH)_2$. This is in agreement with our study. The observed pH values did not change significantly during the lysimeter tests and were 9.9±0.5. Furthermore, for the applied wet processed IBA material tested in this study, the chloride and sulfate leaching in the lysimeters was reduced (see Fig. 1a). The sulfate release kept almost constant up to an L/S of about 0.7 L/kg, limited by $CaSO_4$ solubility, then dropped to around 500 mg/L (L/S 1.25 to 3.25 L/kg), and finally decreased to approximately 250 mg/L. The chloride concentration dropped quickly, starting at 7300 mg/L, and later (from L/S = 1.5 L/kg) remained at an almost constant level of 50 mg/L, which was significantly higher than the chloride concentration in the artificial rainwater used as leachant. Overall, 2x140 single concentrations c_i measured (with i the individual volume of the collected eluates) for chloride are displayed in Figure 1a. Multiplication by the respective L/S ratio gives the leached content E_i in mg/kg, from which the cumulative release U (with $U = \Sigma E_i$, also in mg/kg) can be calculated. The cumulative release of chloride is displayed in Figure 1b.

A similar behavior is observed for calcium (Ca, see Figure 1c) and most of the heavy metals. In contrast, the behavior of Sb and V differ from the usual decrease of concentrations c_i as a function of L/S ratio. At the beginning of the lysimeter tests, the Sb concentrations were low, but at an L/S of around 0.8 L/kg, a steady increase was observed up to concentrations above 25 µg/L (see Figure 1d). Similarly, V started with values of c_i below 10 µg/L and reached values above 90 µg/l at higher L/S ratios (data not illustrated).

This phenomenon has already been observed by other researchers (Cornelis et al., 2006, 2012; Johnson et al., 1999). Antimony forms sparingly soluble calcium antimonate see equation (1).

$$Ca^{2+} + 2\,Sb(OH)_6^- \rightleftharpoons Ca(Sb(OH)_6)_2 \qquad (1)$$

The solubility product K_L of Sb-antimonate is a constant defined as

$$K_L = [Ca^{2+}]^m [Sb(OH)_6^-]^2 \qquad (2)$$

with m as an exponent for the Ca / Sb ratio.

The solubility product K_L is a constant that describes the solubility behavior of a sparingly soluble compound: if the concentration of one component drops, the concentration of the other component increases accordingly. This means that decreasing Ca concentrations as a result of the ageing process (formation of limestone, $CaCO_3$) lead to increasing Sb concentrations. The fact that there is a connection between the concentrations of Ca and Sb becomes apparent when the concentrations are plotted against each other (Figure 2 left). The same applies for vanadium (see Figure 2 right).

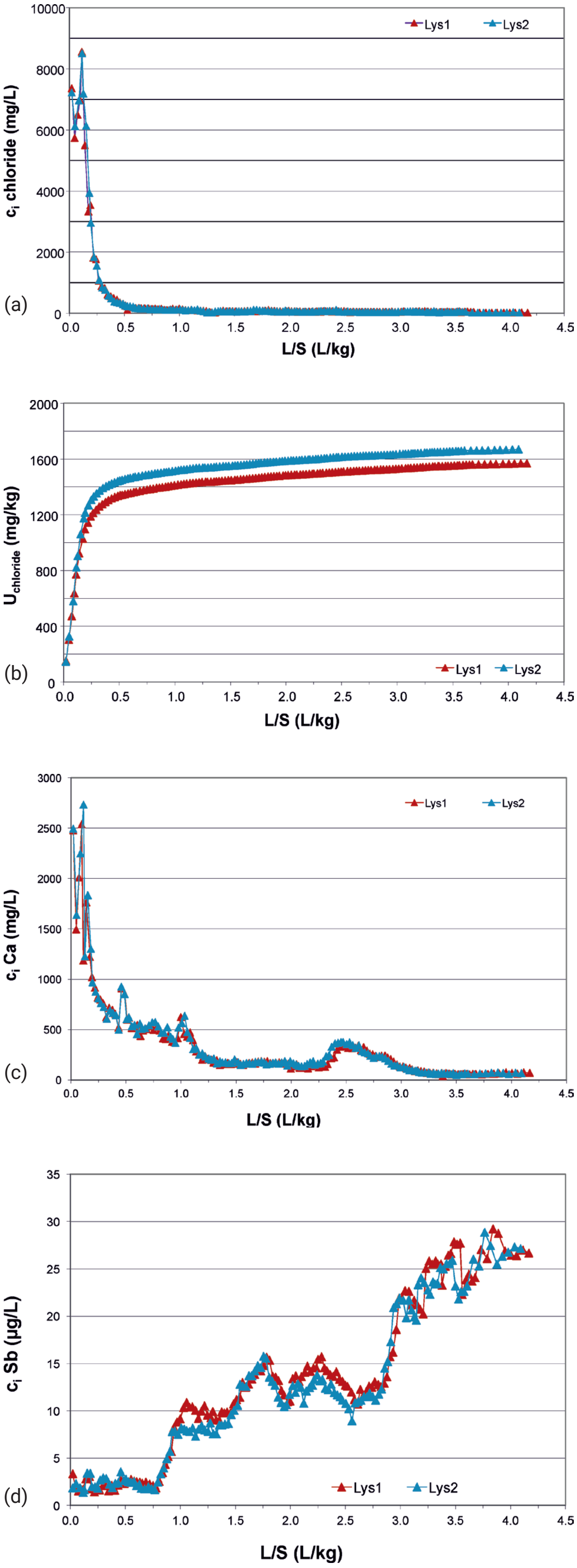

FIGURE 1: Results from the long-term lysimeter experiment as a function of L/S. a: Concentrations c_i of chloride. b: Cumulative release U of chloride, c: Concentrations c_i of calcium, d: Concentrations c_i of antimony.

$$Ca^{2+} + V_2O_6^{2-} \rightleftharpoons CaV_2O_6 \qquad (3)$$

$$K_L = [Ca^{2+}][V_2O_6^{2-}] \qquad (4)$$

The experimental data (circles in Figure 2) were fitted with the solver module in MS Excel (EA, evolutionary algorithm). The best match was achieved in the case of Sb with K_L = 8.9x10^{-19} mol$^{3.7}$ L$^{-3.7}$ and m = 1.7. This leads to $Ca(Sb(OH)_6)_2 x Ca_{0.7}(OH)_{1.4}$ as a chemical formula for Ca antimonate in IBA. Fitting the results for V gave K_L = 1.9x10^{-9} mol^2 L^{-2}.

The standard leaching tests mobilize usually only small amounts of the complete reservoir of certain substances in IBA. Exceptions are substances with high solubility, such as alkaline metal compounds and chloride. Even the solubility of $CaSO_4$ at 2 g/L is high enough that a substantial amount is leached. During the whole duration of the experiment, 1600 mg/kg of chloride and 3000 mg/kg of sulfate were cumulatively released from the IBA in the lysimeters. However, it has to be considered that sulfate and chloride were constituents of the artificial rain (Sanusi et al., 1996) that was used as leachant. Nevertheless, after dismantling the lysimeters, the total content of sulfate and chloride found in the remaining material dropped from 13,400 to 10,400 and from 2,840 to 390 mg/kg, respectively.

4. DISCUSSION

As stated above, IBA is an inhomogeneous mixture of mineral phases and metals not suitable for recycling without processing. The concentrations especially of trace metals can vary by one order of magnitude. However, the described leaching mechanisms, e.g. pH dependence of Pb leaching or Sb mobilization as function of Ca concentration, are largely independent of waste composition, type of furnace or location of the incinerator (Hjelmar et al., 2013). The findings here are therefore relevant not only for the IBA from the sampled plant in Germany but also in general for IBA.

The separation of elemental metals is simple and can be carried out using magnets (Fe metals) and eddy current separators (NFe metals). In Germany, an average of 6.6% Fe and 2.0% non-ferrous metals are extracted from IBA (Gleis & Simon, 2016). The potential would be even greater (Kuchta & Enzner, 2015) if the separation of NFe metals was extended to small grain sizes. Recovering the mineral fraction, which accounts for about 90%, is much more difficult. Applications exist primarily as an unbound base layer in road construction, see for example (Hyks & Hjelmar, 2018).

Numerous contaminants could be minimized with improved processing technologies. Wet treatment means that easily soluble substances such as chlorides are transferred to the washing water. The simultaneous separation of a fine fraction <0.25 mm reduces the sulfate content, mainly calcium sulfate, by more than 60% (Holm & Simon, 2017; Kalbe & Simon, 2020). Broken glass can be separated using sensor-based methods (Šyc et al., 2018). Heavy metal compounds have a higher specific density than other minerals in the bottom ash, so that a reduction of the content of heavy metal compounds is possible by density sorting (Holm et al., 2018).

According to the final draft of the German Ordinance on Secondary Building Materials (Bundesrat, 2020), new regulations are planned for reusing IBA. Accordingly, two quality classes are going to be introduced for IBA (abbreviated HMVA-1 and HMVA-2). To use the mineral fractions of IBA e.g. in road construction, at least class HMVA-2 must be achieved with maximum concentrations in the eluate of L/S = 2 L/kg, as displayed in Table 1.

To compare the results of the experiment (Exp. Data, Table 1) with the limit values (HVMA-1 and HVMA-2, Table 1), the data for cumulative release U must be divided by the respective L/S ratio. The calculated concentrations (see Table 1, right column) comply with the limit values for HMVA-2; however, the concentration of Mo is close to the limit. It is known that Mo is leached as oxyanion MoO_4^{2-} already at low L/S ratios and preferably at pH values above 8 (Dijkstra et al., 2006; van der Sloot et al., 2001). Mobilization of Mo in standard leaching tests is between 1 and 5%, which is much higher than for other heavy metals (e.g. Cu < 0.01%) (Kalbe & Simon, 2020).

5. CONCLUSIONS

Unlike most leached substances, which decrease as a function of the L/S ratio, the leaching values for Sb and

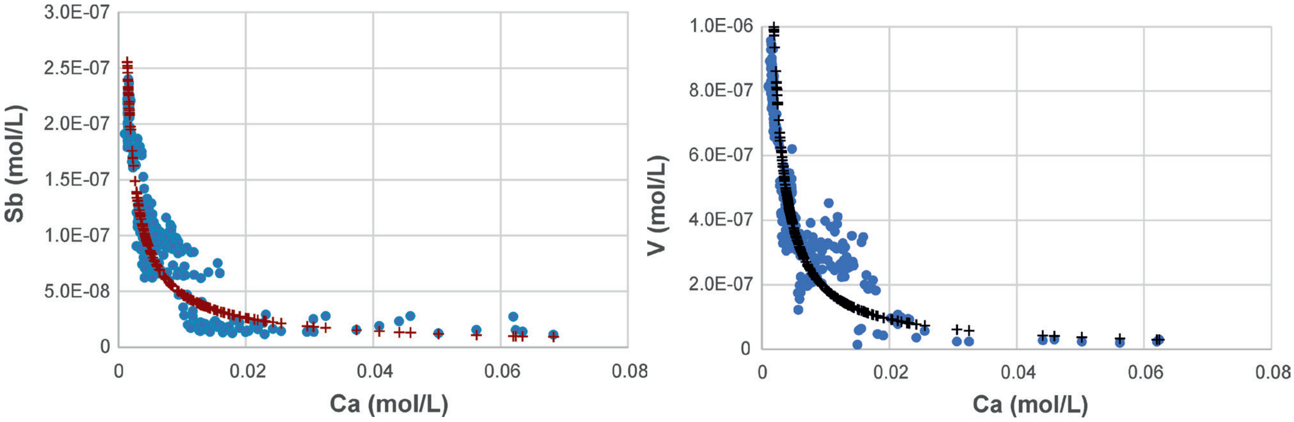

FIGURE 2: Concentration of Sb (left) and V (right) in the lysimeter experiment as a function of Ca concentration (circles). The crosses are the result of a fit with MS Excel.

TABLE 1: Requirements for IBA according to the German Ordinance on Secondary Building Materials (leaching test DIN 19528, L/S = 2 L/kg; classes HMVA-1 and HMVA-2) compared with the calculated concentration from the lysimeter experiment (Exp. Data - average of lysimeter 1 and 2).

Parameter	Dimension	HMVA-1	HMVA-2	Exp. data
pH value		7 - 13	7 - 13	
Electrical conductivity	µS/cm	2,000	12,500	
Chloride	mg/L	160	5,000	768
Sulfate	mg/L	820	3,000	1,110
Antimony	µg/L	10	60	7.1
Chromium (total)	µg/L	150	460 *	25.0
Copper	µg/L	110	1,000 *	152.1
Molybdenum	µg/L	55	400	388.0
Vanadium	µg/L	55	150	31.1

* *for certain applications Cu ≤ 230 µg/L, Cr (total) ≤ 110 µg/L*

V increased over time in the long-term lysimeter experiment. These different behaviors are shown exemplarily in Figure 3 for Ca, Cu, Sb, and V. The concentrations of Ca and Cu drop considerably during the lysimeter experiment, i.e., with the advancing ageing process with increasing L/S in the lysimeter experiment. At an early sampling time (t1) directly after combustion or after processing without ageing, heavy metal concentrations in the leachate may be still too high. However, Sb and V should initially not exceed any limit values for processed IBA. At a later point in time (t2), heavy metals like Cu are still present in the material but are immobilized. At this point, Sb and V may already be released and are approaching the limit values (see Table 1). Our experiment shows that the release of Sb and V from the IBA is not minimized over the time of almost six years. Thus, the long-term use of IBA in e.g. secondary building materials can pose a potential risk for the environment.

The release of Sb and V is dependent on the availability of Ca, which is increasingly precipitated during the ageing process as sparingly soluble $CaCO_3$. On one hand, this effect is desired, because pH values decrease to values below 11, leading to immobilization of most heavy metal compounds. On the other hand, decreasing Ca concentrations lead to the dissolution of Ca-antimonates and Ca-vanadates. Here, the availability of Ca^{2+} is decisive. Mobilized Sb and V can possibly be sorbed on ferrous minerals or form sparingly soluble Fe-antimonates or Fe-vanadates (Van Caneghem et al., 2016). Economically viable technical measures for the targeted depletion of Sb and V have not been reported yet. The limit values for class HMVA-1 are not reachable even for wet-treated IBA as used as test material in the investigation presented here. Chloride concentrations as low as 160 mg/L (see Table 1) might be achievable for separated fractions from IBA, e.g. after dry extraction of bottom ash in the waste incineration plant (Bürgin et al., 1995). Here, a more inert coarse fraction above 2 or 4 mm can be easily separated by sieving (Simon & Andersson, 1995; Šyc et al., 2020). However, wet treatment alone already provides a significant improvement of quality regarding the environmental compatibility in the use scenario.

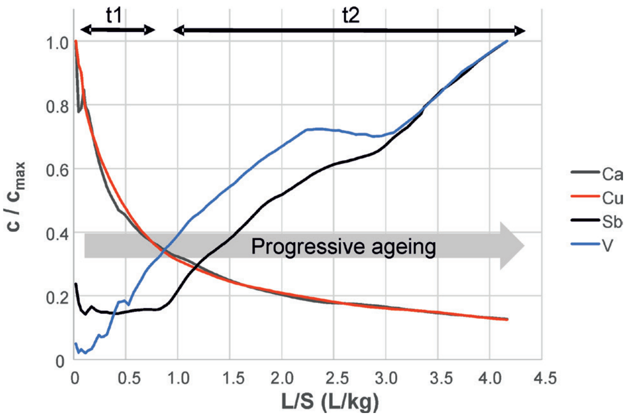

FIGURE 3: Concentrations of Ca, Cu, Sb, and V as a function of L/S. Assessment at the end of period t1 would allow use of the treated IBA, whereas in period t2 the concentration of Sb and V might exceed the limit values.

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