Charles University, Faculty of Science
Institute of Geochemistry, Mineralogy and Mineral Resources

Ph.D. study program: Applied Geology

Summary of the Ph.D. Thesis

WILDFIRES IN POLLUTED AREAS: MINERALOGICAL TRANSFORMATIONS AND REMOBILIZATION OF METAL(LOID)S

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1 Introduction

Since the wildfire frequency is rising, understanding the wildfires' effects on the environment is becoming of great importance nowadays. However, investigations of wildfire effects in sites polluted by anthropogenic activities, combined with advanced mineralogical and phase transformations studies and overall mass-balance calculations, seem to be almost non-existent in the scientific literature. Additionally, at many places in the vicinity of active and abandoned mines and non-ferrous metal smelters, soils exhibit extremely high concentrations of metals and metalloids, commonly in the range of thousands to tens of thousands mg/kg (see Ettler, 2016 for a compilation). This research is focused on African mining/smelting sites, where, according to fire-monitoring systems, wildfires are quite frequent during dry periods of the year when African ecosystems are more vulnerable to fire. For this reason, environments near mines and smelters in Africa represent convenient geochemical laboratories for studying the wildfire impact with a special emphasis on the potential contaminant remobilization. Therefore, we decided to investigate the potential wildfire effects on contaminants in highly polluted environmental samples (soils, biomass) near several active and abandoned mining and smelting sites in sub-Saharan Africa.

2 Aims

1. Adopt and optimize the automated mineralogy technique (autoSEM) for highly complex mining- and smelter-polluted topsoils to decipher the metal(loid)s partitioning within the individual phases, necessary for further interpretations of wildfire simulations.

2. Design and test the experimental wildfire simulating setup with continuous air delivery enabling to sample the aerosol, ashes and outcoming gases.

3. Conduct the burning experiments on soils and biomass samples in the temperature ranges relevant for wildfires in semi-arid areas to estimate the remobilization potential of the key metal(loid)s (As, Cd, Cu, Hg, Pb, Zn).

3 Materials and methods

For this investigation, experimental topsoil samples mainly from the Tsumeb mining/smelting area in northern Namibia were used. In July 2013, the experimental biomass-rich topsoils from Tsumeb were sampled in a polluted hotspot adjacent to the smelter and waste disposal sites on the lee side of the prevailing winds. The experimental topsoils were developed under the most common vegetation cover in the area: A – Acacia spp., AM – Acacia spp. and the marula tree (Sclerocarya birrea) and G – grass (Aristida stipitata).

Biomass samples from several other abandoned smelting areas in sub-Saharan Africa were used to decipher the potential remobilization of the metal(loid)s by the biomass burning during wildfires in grasslands. The grass samples were collected in Selebi-Phikwe in Botswana (Ni-Cu mine and smelter), Luanshya in the Zambian Copperbelt (Cu mine and smelter), and Kabwe in central Zambia (Pb-Zn mine and smelter). Additional grass samples and topsoils were collected in the larger Tsumeb area to determine the spatial distribution of the Hg and its potential fluxes during the wildfire-driven.

For the interpretation of wildfire-simulation experiments, the exact knowledge of the metal(loid) concentrations in the unburned samples is of key importance. Therefore, combustion-free digestion in a

closed system was suggested and tested. The digests obtained by this method were analyzed by a combination of inductively coupled plasma (ICP) optical emission spectrometry (OES) and mass spectrometry (MS). The total Hg content was determined using conventional cold vapor atomic absorption spectroscopy CV-AAS.

The experimental samples were examined using optical microscopy. Concurrently, the selected biomass parts from each sample were adjusted on a carbon tape for further SEM/EDS investigations. Moreover, the heavy mineral fractions of each soil or biomass sample were obtained. The aliquot parts of both original samples and heavy mineral fractions were milled and used for the bulk chemical analyses and X-ray diffraction analysis (XRD). The aliquots of heavy mineral fractions were prepared as polished sections and were analyzed by scanning electron microscopy (SEM/EDS) and electron microprobe (EPMA) afterwards. The selected biomass fragments were also examined and analyzed using SEM/EDS to determine the shapes, sizes and chemical compositions of the particles adhering to the plant surfaces. Additionally, the soil heavy mineral fractions were studied using automated mineralogy (TIMA, autoSEM).

The Hg thermo-desorption (TD) experiments were conducted on the experimental samples using an AMA 254 CV-AAS (75-670 °C). The experiments were conducted under an O_2 gas flow to better simulate the wildfire conditions. Additionally, the

experimental samples were also studied using thermogravimetry (TG; SDT 650, TA Instruments) to determine the temperature-related mass loss (25–1000 °C).

To simulate the potential wildfire effects on the topsoils under laboratory conditions, we introduced two novel experimental set-ups. **A single-step combustion experiment** (Figure 1a) was designed to simulate the wildfire conditions (250 – 850 °C) and consisted of a precise temperature setting, a continuous delivery of fresh air coupled with both a smoke outcome and the possibility of sampling the residual ash and emitted aerosols. The burning experiments were conducted under synthetic air to closely simulate wildfire conditions and to ensure the repeatability of the experiment. For the selected runs, a PTFE filter assembly was placed at the outlet of the burning chamber to trap the aerosol particles for the additional mineralogical investigations. The final part of the setup consisted of a glass pipette for bubbling the outlet gas in the trapping solution. For the metal(loid) mass balance calculations, the chemical compositions of the original soil and the obtained ash were used. Moreover, several mineralogical methods were used to investigate the ashes (XRD, FEG-SEM) and the aerosol particles trapped by the filtering unit (FEG-SEM).

The set-up with a continuous temperature increase and the online ICP-OES detection (Figure 2b) consisted of the combustion

unit as in'the already mentioned set-up (without the filter assembly) directly connected to the quartz injector of the ICP-OES plasma torch. The sample was gradually heated from 25 °C up to 750 °C. The combustion products were directly introduced into the ICP-OES plasma employing an Ar stream.

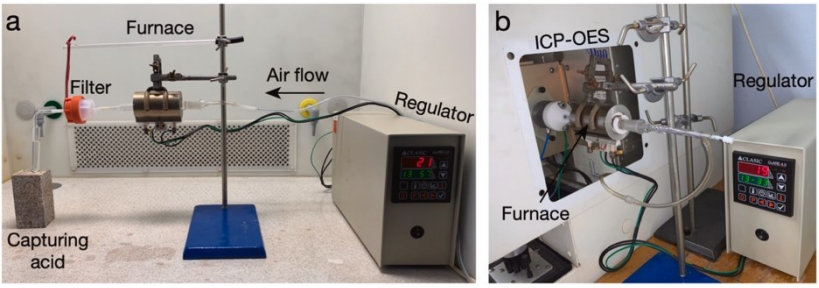

Figure 1. Laboratory wildfire simulation experimental set-ups. a) Single-step combustion set-up; b) Set-up with a continuous temperature increase and online detection.

4 Results and discussion

4.1 Solid speciation of metal(loid)s in soils

We found that As was mainly bound to the apatite group minerals, slag glass and metal arsenates. Copper was predominantly hosted by the sulfides/sulfosalts and the Cu-bearing secondary carbonates. The deportment of Pb was relatively complex: slag glass, Fe and Mn (oxyhydr)oxides, metal arsenates/vanadates and cerussite were the most important carriers for Pb. Zinc was mainly bound to the slag glass, Fe (oxyhydr)oxides, smithsonite and sphalerite. In the case of Cd and Sb, the deportment could not be properly quantified due to their low concentrations in the experimental samples and spectral overlaps with some major elements (Sb vs. Ca, Cd vs. K and Ca in the studied soils). However, based on the test autoSEM measurements, we concluded that the Cd was mainly bound in arsenates, sulfides, slag glass and carbonates.

4.2 Hg emissions during combustion

All the experimental samples exhibited the same TD peak at ~340 °C, which corresponded to more than 91 % of total Hg remobilized. When compared to the TD curves of the technological samples and reference compounds, it can be hypothesized that the Hg in the experimental samples occurs as a mixture of Hg bound to the organic matter and metacinnabar (black HgS),

which presumably originates from the flue gas stream in the smelter (Kim et al., 2004). Based on the acquired Hg distribution maps, we were able to estimate the worst-case scenario of the potential amount of Hg remobilized during the wildfires near the Tsumeb smelter (vegetation: 0.07 g/ha, topsoil: 16.45 g/ha). However, the remobilizable Hg in the polluted hotspots is much higher and corresponds to 0.5 g Hg/ha for the vegetation and 229 g Hg/ha for the topsoil. Thus, our spatial distribution data in the contaminated surroundings of the Tsumeb smelter indicate that ~300 kg Hg can be remobilized by a wildfire from the whole area with a surface of 184 km^2.

4.3 Wildfire driven metal(loid) emissions from the topsoil and biomass

Single-step combustion experiments combined with the experiments performed using the set-up with the online ICP-OES detection indicated that the metals (Cd, Cu, Pb and Zn) were dominantly concentrated in the ash residue at temperatures <550 °C. If exposed to higher temperatures, the metals were partially released (above 550–600 °C; Figure 2). Under this temperature exposure, the aerosol slag-like particles enriched in the metals were emitted. The presence of such particles trapped on filters using the filtration assembly of the single-step combustion setup was confirmed by the subsequent analyses (EDS/SEM).

Arsenic behavior during wildfires is substantially more complicated, due to the complexity of As solid-state speciation in the samples. The main peaks of As emissions were observed at the temperatures of 275 °C, 370–410 °C and 580 °C (Figure 2). Combining the experimental results and the data from the literature (e.g., Johnston et al., 2018, 2019), we hypothesized that As is successively released from As-bearing hydrous ferric oxides at temperature ~275 °C. The multiple peaks of As emissions in the temperature range of 370–410 °C likely correspond to the complex decomposition of the various As-containing phases (arsenolite, arsenates and As-bearing apatite) attached to the organic matter fragments present in the soil litter. A sharp As emission at 580 °C most likely corresponds to the decomposition of enargite (Cu_3AsS_4) (Safarzadeh and Miller, 2016), which is also supported by the concurrent release of sulfur (~600 °C).

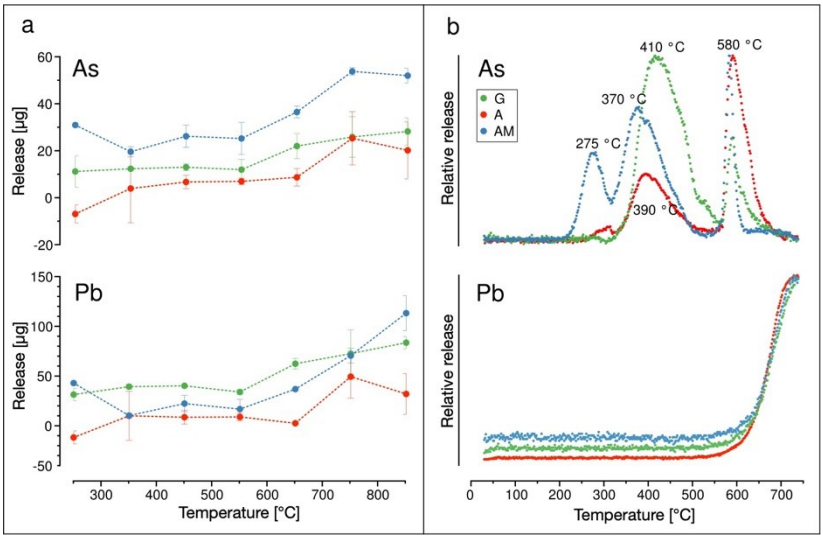

Figure 2. a) Amounts of the As and Pb released from the heated experimental topsoils (in µg) as a function of the temperature as obtained by the single-step combustion set-up. The trend lines were drawn to guide the eyes. The positive values correspond to the release, and the negative values and their standard deviations correspond to the accumulation of a given element; b) Relative release of As and Pb as obtained by the wildfire-simulating set-up with a continuous temperature increase and online measurement. The experimental topsoil samples were collected under the acacia trees (A), acacia and marula trees (AM) and in a grassland (G).

Wildfire-simulating experiments on grass/biomass samples have not been sufficiently interpreted yet. In some cases, the mass-balance calculations were not clear and indicated the inexplicable metal(loid)s enrichment of the ash. We are aware of the need for further additional data processing, nevertheless, the obtained dataset indicates that, during biomass combustion, metals are most probably concentrating in the ash residue.

5 Conclusions

Contamination hotspots near metal mines and smelters in areas exposed to frequent wildfires (e.g., Australia, the Americas, Siberia, southern Africa) may be a significant source of metal(loid) remobilization (Mihaljevič et al., 2011; Ettler et al., 2014; Abraham et al., 2018). Determination of the contaminants' emissions during wildfires and transformations of the burned materials at polluted sites may be complicated due to the high complexity of the metal(loid) speciation. We tried to overcome these difficulties by firstly analyzing the samples using TIMA (autoSEM). Even though this technique is conventionally used in the mining industry, after optimization, it helped to interpret the modal phase distribution as well as the contaminant partitioning and textural relations. Nonetheless, we still face many difficulties. Coupling the sample complexity and variability within a small distance at the given locality significantly complicates the overall estimations of the metal(loid)s' fate. Moreover, the occurrence of wildfires in savannas is often patchy, so the prediction of potential local-to-regional remobilizations and fluxes of metal(loid)s is difficult. It is important to state that the heavy mineral fraction of the topsoils accounted for 4.31 ± 1.81 % of the total mass. However, when the amounts of metal(loid)s bound to the heavy mineral fractions were calculated, they corresponded to 24.3 ± 8.44 % of the total content in the topsoil (median: 21.5 %), which is a relatively low amount when compared with the total metal(loid) contents in the samples. This suggests that

the metal(loid)s are also bound to a less dense soil fraction, such as metal(loid)-bearing phases embedded/locked in larger crystals of quartz, calcite and other geogenic minerals. Additionally, contaminant-bearing particles might be attached to the plant surfaces (e.g., leaf cuticle) in the form of (nano)particles (Kříbek et al., 2014, 2016, 2018; Uzu et al., 2009) or absorbed by the plants' roots (Kříbek et al., 2014; Mihaljevič et al., 2015; Uzu et al., 2009). As a result, autoSEM is a valid tool for mineralogical/geochemical analyses of contaminated sites, but must be supported with other analytical techniques.

To conclude, the presented lab-scale experimental set-up offers several advantages of single wildfire-simulations. These include (i) the possibility of sampling the ashes, (ii) trapping the aerosols and outcoming gases, and (iii) the possible "online" detection of the emitted contaminants as a function of temperature. Also, it overcomes the limitations of wildfire simulations previously performed in muffle furnaces, because it enables a continuous airflow throughout the whole setup and better simulates the natural conditions (see also the limitations mentioned by Bryant et al., 2005). Nevertheless, this experimental approach cannot currently serve as a tool for estimating large-scale emission scenarios, but might be beneficial for local studies and understanding the individual processes driving the wildfire-induced remobilizations of metal(loid)s.

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