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A simplified distillation method for determining reduced sulfur species

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In order to assess the risks of sulfidic soil materials (e.g. marine sediments, peat, dredge materials, mine tailings) on the environment and infrastructure, it is crucial to quantify the different sulfur species that are the major culprits for acidification and metal leaching. Predominately these are reduced species such as metastable iron sulfides (∼FeS) and pyrite (FeS2) that, when subjected to oxidising conditions, produce sulfuric acid which acidifies the surrounding and enhances chemical weathering. The WRB international standard for soil classification states that hypersulfidic material (i.e. soil material capable of severe acidification as a result of the oxidation of inorganic sulfidic compounds) have a diagnostic criterion of $\geq$ 0.01% inorganic sulfidic S per dry mass. Recent studies have also brought to attention coarse-grained (d50% grain size $\geq$ 63 μm) soil material with low sulfidic S that have an acidifying effect on the local environment due to their low buffering capacity, as well as hypersulfidic peat which can produce significant amounts of acidity if excavated. There is therefore a need for a simplified and robust sulfur speciation method, with a determined limit of quantitation of $\geq$ 0.01% sulfidic S to assess these kinds of materials.

In this study, which is part of the TUNNISTUS-project funded by the European Regional Development Fund 2017-2020, the aim is to develop laboratory methods that are easy and safe to use and that rapidly enables reliable assessment of the acidifying potential of acid sulfate soils. The developed methods will be important tools for environmental authorities in order to improve decision making regarding land use on e.g. peat production areas, farmlands and in infrastructure developments. Sulfur speciation methods traditionally require expensive, fragile and cumbersome glassware, which limit the possibility for multiple simultaneous analyses and sets a high demand on the operator. Here we present a simplified distillation apparatus constructed from inexpensive 50 ml polypropylene centrifuge tubes. Gas-tight centrifuge tube caps are fitted with acid resistive tubing plugged with ports and luer-lock stopcocks, which allow delivery of reagents to the reaction chamber, and transport of liberated H2S gas to the collection chamber using N2 gas as a transport medium. The centrifuge tubes are readily sterilized and can sustain over boiling point temperatures, thus enabling shorter reaction times compared to cold purge-and-trap methods. The miniaturised setup permits a range of sample sizes (0.5 – 5 g) and a modified analytical procedure enables sequential distillation of reduced sulfur species from a single sample. Multiple experiments for determining reduced sulfur species in hypersulfidic fine- and coarse-grained material, hypersulfidic peat material and a laboratory graded pyrite standard show good precision and low limits of detection and quantitation, thus enabling more precise risk assessments for a broad range of sulfidic materials.