



Release of potassium accompanying the dissolution of rice straw phytolith



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HIGHLIGHTS

- Potassium is present in rice stems and leaves.
- Potassium can be immobilized in the mineralized silica (phytolith).
- We found that potassium co-exists with organic matter in phytolith structure.
- Desilification of the phytolith is a main factor regulating potassium release.
- Pretreatment of the rice straw at 600 °C is optimal in providing available potassium.

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ABSTRACT

In rice, Si assimilated from the soil solution is deposited in inter- and intracellular spaces throughout the leaf and stems to form silicified structures (so-called phytoliths). Because K is also present in significant concentrations in rice stems and leaves, the question arises if K is immobilized in the mineralized silica during the precipitation of Si. This work determined whether desilification of the phytolith is a factor regulating K release by implementing batch experiments. Solubility of Si and K of the rice straw heated at different temperatures were examined to identify effect of pretreatment.

Analyses of phytoliths using SEM–EDX and X-ray tomographic microscopy in conjunction with the results from batch experiments revealed that K might co-exist with occluded organic matter inside the phytolith structure. In the kinetic experiments, corresponding increases of K and Si concentrations in the supernatants were observed which suggested that desilification of the phytolith is a main factor regulating K release. The extent of heat pretreatment of the rice straw is of significant importance with respect to dissolution of the phytolith by affecting organic removal and surface modification. At temperatures lower than 600 °C, corresponding increases of the soluble Si and K with heating temperature have been obviously observed. In contrast, the solubility of Si and K gradually decreased at temperatures above 600 °C. This work provides insights into factors that control release of K and Si from phytolith and a practical recommendation for practices of burning rice straw that may maximize subsequent release of Si and K for crops.

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1. Introduction

Silicon (Si) and potassium (K) are two of the most abundant nutrient elements in rice (Tiwari et al., 1992; Epstein, 1999). These

two elements are taken up from the soil solution and transported to leaf, stem and other parts of rice. While K provides the appropriate ionic environment for metabolic processes in the cytosol and, as such, functions as a regulator of various processes including growth regulation (Leigh and Wyn Jones, 1984), Si is known to accumulate as siliceous phytolith by deposition in inter- and intracellular spaces throughout the leaf and stem of rice

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(Parr and Sullivan, 2005). Several studies have reported that, during the precipitation of Si to form phytolith, organic molecules can be trapped within the silica (Kelly et al., 1991; Elbaum et al., 2003; Piperno and Stothert, 2003). This suggests that inorganic compounds, e.g., K in the rice xylem/phloem sap, can also be occluded in the silica body (so-called PhytOK). It is hypothesized that the PhytOK can be released and contribute to the K-pool of the soil once desilification of the phytolith occurs.

It is generally accepted that the desilification of silica in aqueous solutions occurs via hydrolysis of Si–O–Si bonds of the SiO₂ crystal structure. Water itself is a strong promoter of hydrolysis via orientation of the electronegative water oxygens towards the Si atom, leading to a transfer of electron density to the Si–O–Si bond, thereby increasing its length and eventually breaking it (Dove and Crerar, 1990). Recently, it has been reported by Nguyen et al. (2013) that dissolution of Si is controlled by aqueous solution chemistry and pretreatment of the rice straw. However, additional data on co-releases of Si and other occluded elements such as K is currently lacking. Our current studies of the release of K accompanying phytolith dissolution in aqueous solutions helps address this knowledge gap.

On-site burning after harvesting is the primary method of handling rice straw to return nutrients to the soils. In recent decades, burning of rice straw has been predominant because it is a cost-effective method of straw disposal, doesn't interfere with soil preparation, and helps to reduce pest and populations of pathogens resident in the straw biomass (Dobermann and Witt, 2000). The mode of pretreatment of rice straw and burning at different temperatures might result in various degrees of dehydroxylation of biogenic silica and organic matter contents. In this study, we identify co-release of Si and K using batch experiments with rice straw phytolith ash samples obtained from different ashing temperatures. A further treatment by H₂O₂ to remove organic matter, which is often mentioned as a method for phytolith preparation (Parr et al., 2001), will be used to relate losses of K and Si that accompany the removal of organic matter. SEM–EDX spectra reflecting the presence of various elements, and X-ray tomographic microscopy providing 3D-segmentation and visualization of the solid phase were also used in this study to evaluate distribution of K in the rice straw phytoliths. Improved knowledge of the mechanisms and release processes of K from phytolith will open different management options and is of importance for the continued efficient cultivation of rice.

2. Materials and methods

2.1. Sample production

Rice straw was collected from a paddy field in the rice-growing area of the central part of the Red River Delta (105°44'17"E, 20°59'57"N) directly after harvesting. The rice straw was air-dried, ground in a blade grinder, and passed through a 1.0 mm sieve. The rice straw had 73.6 g kg⁻¹ Si examined by an X-ray fluorescence analyzer (XRF-1800 Shimadzu, Japan), 387 g kg⁻¹ C, and 13.2 g kg⁻¹ N as determined by an Elementar Vario EL (Hanau, Germany) elemental analyzer with a respective C:N ratio of 29. Dry ashing of rice straw was performed by heating finely-ground, air-dried, rice straw in an oven at a target temperature (between 300 °C and 1000 °C) for 2 h. To avoid strong exothermic reactions during dry-ashing the weight of sample was limited to 5 g. For kinetic experiments we selected two samples treated at 400 °C and 800 °C.

2.2. Methods

For determination of Si and K distribution and dissolution from phytoliths, soluble salts from the ashes were removed by washing

with deionized water for five minutes followed by centrifugation and decantation. The procedure was repeated five times, and samples were finally freeze-dried. To identify distribution of elements in phytolith using SEM–EDX, the samples were examined using a FEI Quanta 400 FEG ESEM (FEI Company, Hillsboro, OR, USA) at 20 kV under low vacuum conditions using a gaseous secondary electron detector. The stage was at room temperature with a chamber pressure of 80 Pa. Surface point elemental analysis was performed while the samples were being viewed in the SEM using a Princeton Gamma-Tech energy dispersive spectrometer. The visualization was performed by X-ray tomographic microscopy using the 3D segmentation and visualization software YaDiV (Frieze et al., 2013) to provide a three-dimensional image of the principal arrangement of silicified structures and organic matter for a vascular bundle in a dried stem of a rice plant. The samples were analyzed at the synchrotron light source (SLS) of the Paul-Scherrer-Institute in Villigen, Switzerland. The TOMCAT (TOmographic Microscopy and Coherent rAdiology experimenTs) beamline receives photons from a 2.9 T superbending magnet with a critical energy of 11.1 keV, producing a monochromatic beam. A sample is fixed on a centering and rotation stage in front of a microscope, detecting the monochromatic X-ray beam.

In all experiments, 200 mg of sample was mixed with 200 mL of solution in 250 mL polypropylene tubes. Suspensions were gently shaken by hand directly after mixing and allowed to stand for 24 h at room temperature. Some of the batch experiments were extended up to 6 d with sampling at 24 h intervals. The experiments were terminated by filtration of the suspension through a 0.45 μm pore-size cellulose acetate filter. Si and K in solution was determined in triplicate using the molybdate blue method (Mortlock and Froelich, 1989) with a Spectrophotometer UV–Vis (L-VIS-400, Labnics Company, Fremont, CA, USA) and flame photometer (PFP7-Jenway, OSA, UK), respectively.

In more detail, we performed the following experiments:

Experiment 1: We analyzed the dissolution kinetics for the 400 °C-, 800 °C-, 400 °C/H₂O₂-, and 800 °C/H₂O₂-treated rice straw samples by monitoring Si and K releases into solution. The dissolution experiments lasted 6 d and sampling was carried out every 24 h.

Experiment 2: To evaluate the effect of heat-pretreatment of rice straw, the rice straw samples treated at 300, 400, 500, 600, 700, 800, 900 and 1000 °C were mixed with deionized water and kept at room temperature for 24 h.

Experiment 3: To examine whether K sorbs on siloxane surfaces of the phytolith, we used 400 °C- and 800 °C-treated rice straw samples. Ca²⁺ and NH₄⁺ prepared in solutions with a concentration range of 10–100 mmol_c L⁻¹ from pure analyzed chloride salts were used as exchangers. Suspensions were terminated after 24 h.

3. Results

3.1. Sample properties

The different ashing temperatures of straw changed organic-C content of the straw to different extents (Table 1). The organic-C was most completely removed by heating at 800 °C, whereas only less than 12% of total organic-C was removed at 400 °C. With a subsequent treatment with H₂O₂, remaining organic-C contents were 2.85% and 0.48% for the ashes treated at 400 °C and 800 °C, respectively. The total Si and K contents were 20.7% and 9.4%, respectively, after treatment at 400 °C, and 32.0% and 10.3% following 800 °C treatment. The ashes had an alkaline reaction (pH 10–11). Soluble K of the 400 °C- and 800 °C-treated samples in a 1:10

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