**QUEEN'S
UNIVERSITY
BELFAST**

Characterization and dissolution properties of phytolith occluded phosphorus in rice straw

Trinh, T. K., Nguyen, T. T. H., Nguyen, T. N., Wu, T. Y., Meharg, A. A., & Nguyen, M. N. (2017). Characterization and dissolution properties of phytolith occluded phosphorus in rice straw. *Soil and Tillage Research*, 171, 19-24. <https://doi.org/10.1016/j.still.2017.04.002>, <https://doi.org/10.1016/j.still.2017.04.002>

Published in:
Soil and Tillage Research

Document Version:
Peer reviewed version

Queen's University Belfast - Research Portal:
[Link to publication record in Queen's University Belfast Research Portal](#)

Publisher rights
© 2017 Elsevier Ltd. This manuscript version is made available under the CC-BY-NC-ND 4.0 license <http://creativecommons.org/licenses/by-nc-nd/4.0/>, which permits distribution and reproduction for non-commercial purposes, provided the author and source are cited.

General rights
Copyright for the publications made accessible via the Queen's University Belfast Research Portal is retained by the author(s) and / or other copyright owners and it is a condition of accessing these publications that users recognise and abide by the legal requirements associated with these rights.

Take down policy
The Research Portal is Queen's institutional repository that provides access to Queen's research output. Every effort has been made to ensure that content in the Research Portal does not infringe any person's rights, or applicable UK laws. If you discover content in the Research Portal that you believe breaches copyright or violates any law, please contact openaccess@qub.ac.uk.

Characterization and dissolution properties of phytolith occluded phosphorus in rice straw

Trang K. Trinh¹, Thinh T. H. Nguyen², Tu N. Nguyen³, Ta Yeong Wu⁴,

Andrew A. Meharg⁵, Minh N. Nguyen^{*1, 5}

¹ Faculty of Environmental Science, VNU University of Science, Vietnam National University, Hanoi (VNU), 334 Nguyen Trai, Thanh Xuan, Hanoi, Viet Nam.

² Institute for Nuclear Science and Technology, 179 Hoang Quoc Viet, Hanoi, Viet Nam.

³ Faculty of Environment, Vietnam National University of Agriculture, Trau Quy, Gia Lam, Hanoi, Viet Nam.

⁴ Chemical Engineering Discipline, School of Engineering, Monash University, Jalan Lagoon Selatan, 47500 Bandar Sunway, Selangor Darul Ehsan, Malaysia.

⁵ Institute for Global Food Security, Queen's University Belfast, David Keir Building, Malone Road, BT9 5BN, Northern Ireland, UK.

* Correspondence: Dr. Minh N. Nguyen, E-mail: minhnn@vnu.edu.vn

1. Introduction

Rice straw derived phytolith, a siliceous structure formed through Si precipitation in rice plant tissues (Parr and Sullivan, 2005), has been widely known as a potential source to sustain soil Si nutrient pools when it is cycled to soils (Dobermann and Fairhurst, 2000; Nguyen et al., 2014; Nguyen et al., 2015; Seyfferth et al., 2013). However, there has been a lack of systematic information on the fate of rice straw derived phytolith in soils, and to what extent this nutrient pool can benefit crops. Phosphorus (P) is among the most important nutrient elements in rice. P is taken up from the soil solution by root absorption, transport across cell membranes and compartmentation, and redistribute within different parts of rice plant (Agarie et al., 1996; Schachtman et al., 1998; Yamaji et al., 2008). During rice growth,

P can be re-translocated through rice's xylem/phloem system (Schachtman et al., 1998), and some parts might be trapped within phytolith structure (so-called phytOP) when Si precipitates. This means that even if rice straw is cycled to soil, this P pool might still exist inside phytolith and unavailable for plant uptakes. There has been a knowledge gap and lack of experimental evidence for the fate of phytOP and its capacity to serve as an additional P source for crops.

Generally, dissolution of phytolith is regulated by the desilification in aqueous solution. This process occurs via hydrolysis of $\equiv\text{Si-O-Si}\equiv$ bonds of the silica surface in which water molecules orient their electronegative pole towards the Si atoms in silica structure, resulting in a transfer of electron density to the $\equiv\text{Si-O-Si}\equiv$ bonds, thereby raising their length and consequently breaking them (Dove and Crerar, 1990). pH is well understood as a crucial factor driving phytolith dissolution kinetics via protonation or deprotonation reactions (Frayssé et al., 2009; Nguyen et al., 2014) which can either increase resistance of the surface or make the surface more vulnerable from nucleophilic attacks (Dove and Crerar, 1990). On the other hand, occluded organic matter can be another factor in preserving phytolith since it may act as a protective shield against hydrolysis of the silica (Nguyen et al., 2014; Parr and Sullivan, 2005). In this study, the release of P was inspected in considering the dissolution rate of phytolith's silica body and influence of occluded organic matter content.

Open-field burning of rice straw after harvesting is a conventional method of disposal of straw to return nutrients to the soils in many rice-based countries. Although this method causes significant emission of greenhouse gases (e.g. CO_2 , CO, NO_x), losses of nutrients (e.g. N, K, P,) and contributes to air pollution (Andreae and Merlet, 2001; Bijay et al., 2008; Dobermann and Fairhurst, 2000), it is the easiest way of returning most nutrients to the soils, and at present rice growers have little incentive to change this method. As burning of rice straw at different temperatures might result in various degrees of dehydroxylation of biogenic

silica and organic matter contents, rice straw phytolith ash samples which were obtained from 300 to 1000°C ashing temperatures were used to examine the release of P in batch experiments. X-ray tomographic microscopy (Marone, 2010), provided a visualization for siliceous structure of phytolith. Scanning electron microscopy (SEM), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), specific surface area (SSA) and chemical composition analysis were carried out to evaluate transformation of phytolith and its effect on P release. Statistical analysis using Pearson's test can help to provide an additional information of correlation between the released Si and P. Based on the identification of relation between the release of P and dissolution of rice-straw phytolith, this study clarifies the dynamics of the phytOP and suggest possible management options on one hand to maximize the use of this phytOP pool, and on the other hand to adjust fertilization strategy.

2. Materials and Methods

2.1. Study site and sample production

Rice-straw was collected from a paddy field (105°44' E long, 20°59' N lat) in the Red River Delta (RRD), a second largest delta in Vietnam. In the RRD, approx. 12 tons ha⁻¹ of the rice straw can be annually burnt and returned to the fields which serves as large amount of phytolith for paddy soils. The rice-straw was air-dried, milled and passed through a 1.0-mm sieve. Ashing was conducted by heating the rice straw sample in an furnace at a target temperature range between 300 and 1000°C for 2 h. The derived phytolith containing ash was washed with DI water, followed by centrifugation and decantation, and finally were dried at 60°C in an oven for 24 h. The samples from pretreatments at 300, 400, 500, 600, 700, 800, 900 and 1000°C were used for chemical analysis, mineralogical characterizations, SSA analysis and batch dissolution experiments. For kinetic experiments, the samples treated at 400, 700 and 1000°C were selected.

2.2. Sample characterization

Tomographic and morphological analysis: a 3D image of the siliceous structures of a rice plant was built by using YaDiV software (Friese et al., 2013), and input data from X-ray tomographic microscopy by TOMCAT (TOMographic Microscopy and Coherent rAdiology experimenTs) beamline at the Swiss Light Source, Paul Scherrer Institute, Villigen, Switzerland. Dry rice leaf was scanned under monochromatic beam from a 2.9 T superbending magnet at an energy of 11.1 keV. Micromorphology of the sample was inspected by using SEM (FEI Quanta 600 FEG, USA).

Chemical composition: To examine chemical composition, the samples derived from pretreatments at 300 to 1000°C were examined using the Particle Induced X-Ray Emission (5SDH-2 Pelletron accelerator system, USA). Total organic carbon (TOC) in the samples was measured by wet digestion with concentrated H₂SO₄ and 0.5 M K₂Cr₂O₇ at 170 – 180 °C.

X-ray diffraction, FTIR and surface area analysis: Silica phases of the ash samples were examined by powder X-ray diffraction (Bruker AXS D5005, Germany). The surface groups of the ash samples were identified by using FTIR measurements (Nicolet™ iS™5 FT-IR, Thermo Scientific, USA). The SSA of the samples was measured with a Gemini VII 2390p surface area analyzer.

2.3. Kinetic batch experiments

To examine P and Si solubility, 300 mg of sample was mixed with 300 mL of DI water in 500-mL plastic tubes. Suspensions were gently shaken and allowed to stand for 24 h at room temperature. For the samples pre-treated at 400, 700 and 1000°C, the batch experiments were in triplicates and extended up to 6 d with sampling at 24 h intervals. The suspensions were passed through a 0.45 µm filter paper. Soluble P was determined using ion chromatography (DX-600, Dionex-Thermo, USA) while soluble Si was examined by molybdate blue method with a Spectrophotometer UV-Vis (L-VIS-400, Labnics, USA). Since the ash samples still contain certain amounts of OC, dissolution of the OC might affect

releases of Si and P by changing phytolith exposure to solution. In all kinetic experiments, DOC was also analysed using a TOC Analyzer (VCPH, Shimadzu), to identify whether the released P was related to dissolution of occluded organic matter. Correlations between the released Si and P derived from all dissolution kinetic experiments were also evaluated by Pearson's test using SPSS 20.0 software program. The strength of correlation can be evaluated by using two levels of significance 0.01 and 0.05, representing relations between the released Si and P occurring by chance are 1 and 5%, respectively.

3. Results

3.1. Sample characterization

The arrangement of phytolith's siliceous structure in the rice leaf was visualized in Fig. 1. A high-porosity system with various size holes which might represent longitudinal vascular bundles, veins or fibers of the rice plant (Botha, 2013). Si covers, throughout, internal- and external surfaces of the vascular system to form a coating layer, coloured yellow, as shown in Fig. 1a. This layer is known as silica-rich epidermis which is developed to become the main strengthening tissue in the stem and leaf (Islam et al., 2009; Yamanaka et al., 2009). The holes can be fulfilled by organic matter which appears in violet (Fig. 1b). Excessive precipitation of Si can likely to form "closed holes" within the vascular bundles, capturing embedded organic matter, as well other occluded substances including P. (Figure 1)

Heat treatments of the rice straw resulted in significant changes in chemical composition and SSA as shown in Tab. 1. The organic carbon was almost completely removed by heating at >800 °C, whereas, only less than 30% of TOC was removed at 300 °C. In rice straw the total P and Si contents were 1.02 and 73.6 g kg⁻¹, respectively. However, the contents of P changed from 1.21 to 2.55 g kg⁻¹ and those of Si varied from 87.6 to 184.2 g kg⁻¹, when the heating temperatures of rice straw increased from 300 to 1000°C. A severe

decrease of the SSA was obtained with increasing heating temperature. The SSA decreased from 36.2 to 0.22 m² g⁻¹ with an increase of heating temperature from 300 to 1000°C, indicating an enhanced condensation of silica structures at higher temperatures as shown in Fig. 2.

(Table 1)

(Figure 2)

The XRD for phytolith derived from different treatment temperatures is shown in Fig. 3a. At temperatures < 700°C, no specific reflection was observed, suggesting that silica occurs in amorphous form. Temperatures > 800°C can favor crystalization and cause the formation of more stable silica crystalline structures, i.e., cristobalite and tridymite (Kordatos et al., 2008). Functional groups of the samples were revealed through FTIR spectra as described in Fig. 3b. The bands at ~1100 cm⁻¹ and 800 cm⁻¹, representing for the stretching vibration of the SiO₄ tetrahedron and the bending vibration of inter-tetrahedral ≡Si-O-Si≡ bonds, were apparent for almost all pretreatments.

(Figure 3)

3.2. Solubility of Si and P

Dissolution kinetics

Batch experiments for the samples derived from different heat-treatments showed that the concentration of soluble P and Si significantly increased with time at different rates for 400 and 700°C -treated samples, whereas trivial increases of soluble P and Si were observed for 1000°C -treated sample (Fig. 4a, b). After 6 d, P and Si releases were 1.2 and 41.0 mg L⁻¹ for the 400°C treated sample, 2.0 and 55.0 mg L⁻¹ for the 700°C -treated sample, and 0.04 and 3.3 mg L⁻¹ for the 1000°C -treated sample, respectively. For 400 and 700°C -treated samples, P concentration showed a remarkable increase within the first day, and minor increases after

day 1 while Si concentration in the supernatant continuously increased over 6 d. The releases of DOM in batch experiments were also examined, but no clear trend has been observed. (Figure 4)

Correlation coefficients obtained from Pearson's test for samples treated at 400 and 700°C were 0.968 and 0.878 (respectively), indicating a relation between released Si and P. In contrary, a correlation coefficient of -0.504 implies that no clear relation exists between released Si and P for the sample treated at 1000°C. For the samples treated at 400 and 700°C, a significant correlation at the 0.01 level is an obvious evidence for a strong relation between Si and P, suggesting that release of P relates to the dissolution of phytolith. For the sample treated at 1000°C, the relation between released Si and P cannot be affirmed due to a very low solubility of phytolith. A normalized correlation coefficient of 0.922 was obtained when Pearson's test was executed for all the samples together, and this correlation showed a significance at 0.01 level.

Effect of treatment temperature

The solubility of P and Si of the phytolith samples showed a strong dependence on heating temperature (Fig. 5). When the heating temperature was changed from 300 to 600°C, increases of the soluble P and Si from 0.4 to 1.3 mg L⁻¹ and 7.2 to 13.5 mg L⁻¹, respectively, were observed. At >700°C, the solubility of P and Si was readily reduced. The lowest values of soluble P and Si were 0.04 and 0.27 mg L⁻¹ (respectively) for the sample treated at 1000°C. Over the entire range of heating temperature from 300 to 1000°C, P showed a relatively lower solubility as compared to Si. It can be recognized that soluble P and Si showed similar "peak shape" trends as depicted in Fig. 5, and the highest values were at ~600°C. This phenomenon suggested that a similar mechanism drove the dissolution of P and Si from the samples. (Figure 5)

4. Discussion

Si and P assimilated from soil are transferred to rice leaf and stem. While Si tends to be immobilized by precipitation that forms siliceous phytolith structure (Nguyen et al., 2014; Parr and Sullivan, 2005), it is likely that P is more mobile in plant. P stored in older leaves can be readily re-translocated to both young leaves and growing roots via the xylem and phloem systems (Jeschke et al., 1997). X-ray tomographic microscopy revealed various holes within the phytolith and these holes were filled with occluded organic substances (Fig. 1). However, these holes as well as morphology of the phytolith can be strongly affected by heat-treatment as shown in Fig. 2, in which high treatment temperatures resulted in a rearrangement and followed by crystallization of silica in phytolith structure as deduced from XRD spectra in Fig. 3a. As P is dominant in the transport sap (Schachtman et al., 1998), P and organic compounds might also be trapped inside siliceous structure of phytolith when precipitation of Si forms phytolith. Chemical analysis revealed that phytOP contents of the samples obtained from various treatment temperatures were up to 2.55 g kg^{-1} .

Six-day extractions of the samples treated at 400 and 700°C resulted in Si concentrations of 41 and 55 mg L^{-1} , respectively, which were both equivalent to ~31 % of the total Si introduced in the experiment. This result is in between the findings of Wickramasinghe and Rowell (2006), Nguyen et al. (2014) and Wilding et al. (1979), who measured a Si extractability of 20-38%, 46% and 50-75%, respectively. It can be inferred that burning process resulted in robust destruction of the rice straw and produced a structure with low resistance to dissolution. In contrast, soluble amount of the sample treated at 1000°C was less than 2 % indicating its high resistance against dissolution. This implies that burning rice straw at high temperatures ($> 800^\circ\text{C}$) can lead to a long-term stabilization of rice straw phytoliths under actual soil conditions.

In the kinetic experiments, the solubility of Si was increased with time as a consequence of phytolith desilification, and a corresponding increase in the P concentration

suggested a relationship between the released Si and P. Scatter plots as shown in Fig. 6 demonstrate a strong correlation between the released Si and P amounts. A positive correlation, $R^2 = 0.847$, indicates ~85% of the variance in the soluble P is related to soluble Si. Also, strong correlation coefficients at the 0.01 level obtained from Pearson's test allow generalizing a relation between P and Si, in which the P release is regulated by the dissolution of the phytolith and treatment temperature appears as a primary factor driving this process. (Figure 6)

Changes in the removal rate of occluded organic matter and transformation of the silica phases during the heating were recognized as two of the major factors directing Si and P release from rice straw phytoliths. High organic matter content in the samples heated at low temperature is likely a major factor to inhibit Si and P liberates. It might be because the heating treatments resulted in an inter-embedded organic-silica structure and this matter acts as a "two-surface shield", by which each of organic matter and phytolith silica can be preserved from the other. As a consequence, the presence of occluded organic matter can decrease of dissolution rate of the phytolith and phytOP as well. The present result is in agreement with other findings (Nguyen et al., 2014; Parr and Sullivan, 2005) in which it was stated that occluded organic matter "strengthens" the phytolith surface and its resistance to desilification. When heating temperature increases, dehydroxylation of silanol groups will favor a formation of siloxane bonds (Fig. 2a), and the surface is hydrophobic (Zhuravlev, 2000). This reaction diminishes adsorption of water molecules on the surface and decreases the breakage of the surface $\equiv\text{Si-O-Si}\equiv$ bonds (Parr and Sullivan, 2005). It can, therefore, explain why lesser Si and P were liberated from the phytoliths in this case. Additionally, the formation of stable silica phases at high temperature favored a product with low SSA and less activity (Kordatos et al., 2008), likely also resulting in a decline of P and Si release. An illustration for the effects of occluded organic matter, SSA and crystallization degree together

on co-release of Si and P was depicted in Fig. 7. Burning rice straw at a temperature range from 500 to 700°C was suggested as an optimal practice toward serving more available Si and P for soils and crops.

(Figure 7)

5. Conclusions

Rice straw phytoliths contain up to 2.5 g P kg⁻¹, indicating that this P-pool can be an additional source for crops when the straw is returned to the soils. By integrating the results from batch experiments, chemical analysis, and X-ray tomographic microscopy, this study revealed that P in the phytolith structure might be unavailable for plants prior to desilification of the phytolith structure. Co-release of P and Si was observed and the dissolution of the phytolith could be concluded as the main factor controlling P release. The heating temperature of the rice straw could affect P and Si releases by intensifying the removal of occluded organic matter or crystallizing the silica surface of the phytolith. The maximum values of soluble P and Si observed from 500 till 700°C recommended that the pretreatment of the rice straw at this temperature range is optimal in serving as available P and Si for crops and soils. As it is still unclear to what extent organic-P can be transformed to inorganic P accompanying with heat treatment of the rice straw, this process and its relation to P-release will be considered in future works.

Acknowledgements

This research was funded by the Vietnam National Foundation for Science & Technology Development (Project 105.09-2015.01).

References

Agarie, S., Agata, W., Uchida, H., Kubota, F., Kaufman, P.B., 1996. Function of silica bodies in the epidermal system of rice (*Oryza sativa* L.): testing the window hypothesis. *Journal of Experimental Botany* 47, 655 - 660.

- 250 Andreae, M.O., Merlet, P., 2001. Emission of trace gases and aerosols from biomass burning.
251 Global Biogeochemical Cycles 15, 955-966.
- 252 Bijay, S., Shan, Y.H., Johnson-Beebout, S.E., Yadvinder, S., Buresh, R.J., 2008. Chapter 3
253 Crop Residue Management for Lowland Rice-Based Cropping Systems in Asia,
254 Advances in Agronomy. Academic Press, pp. 117-199.
- 255 Botha, T., 2013. A tale of two neglected systems—structure and function of the thin- and
256 thick-walled sieve tubes in monocotyledonous leaves. Frontiers in Plant Science 4.
- 257 Dobermann, A., Fairhurst, T., 2000. Rice. Nutrient disorders and management, Handbook
258 series. Potash and Phosphate, Singapore.
- 259 Dove, P.M., Crerar, D.A., 1990. Kinetics of quartz dissolution in electrolyte solutions using a
260 hydrothermal mixed flow reactor. Geochim. Cosmochim. Acta 54, 955-969.
- 261 Fraysse, F., Pokrovsky, O.S., Schott, J., Meunier, J.-D., 2009. Surface chemistry and
262 reactivity of plant phytoliths in aqueous solutions. Chemical Geology 258, 197-206.
- 263 Friese, K.I., Cichy, S.B., Wolters, F.E., Botcharnikov, R.E., 2013. Analysis of tomographic
264 mineralogical data using YaDiV—Overview and practical case study. Comp. Geosci. 56,
265 92-103.
- 266 Islam, M.T., Sarwar, A.K.M.G., Begum, H.H., Ito, T., 2009. Epidermal features of rice leaf
267 CV. BRRI Dhan29. Bangladesh Journal of Plant Taxonomy 16, 4.
- 268 Jeschke, W.D., Kirkby, E.A., Peuke, A.D., Pate, J.S., Hartung, W., 1997. Effects of P
269 deficiency on assimilation and transport of nitrate and phosphate in intact plants of castor
270 bean (*Ricinus communis* L.). Journal of Experimental Botany 48, 75-91.
- 271 Kordatos, K., Gavela, S., Ntziouni, A., Pistiolas, K.N., Kyritsi, A., Kasselouri-Rigopoulou,
272 V., 2008. Synthesis of highly siliceous ZSM-5 zeolite using silica from rice husk ash.
273 Microporous and Mesoporous Materials 115, 189-196.

- 274 Marone, F.M., R; Modregger, P; Fife, J; Pinzer, B; Thüring, T; Mader, K; Mikuljan, G;
275 Isenegger, A; Stampanoni, M 2010. Present and Future X-ray Tomographic Microscopy
276 at TOMCAT., 10th International Conference on X-Ray Microscopy, Chicago, Illinois,
277 (USA), 15 August 2010 - 20 August 2010, 116-119.
- 278 Nguyen, N.M., Dultz, S., Guggenberger, G., 2014. Effects of pretreatment and solution
279 chemistry on solubility of rice-straw phytoliths. *Journal of Plant Nutrition and Soil*
280 *Science* 177, 349-359.
- 281 Nguyen, N.M., Dultz, S., Picardal, F., Bui, T.K.A., Pham, Q.V., Schieber, J., 2015. Release of
282 potassium accompanying the dissolution of rice straw phytolith. *Chemosphere* 119, 371-
283 376.
- 284 Parr, J.F., Sullivan, L.A., 2005. Soil carbon sequestration in phytoliths. *Soil Biol. Biochem.*
285 37, 117-124.
- 286 Schachtman, D.P., Reid, R.J., Ayling, S.M., 1998. Phosphorus uptake by plants: from soil to
287 cell. *Plant Physiology* 2, 447-453.
- 288 Seyfferth, A.L., Kocar, B.D., Lee, J.A., Fendorf, S., 2013. Seasonal dynamics of dissolved
289 silicon in a rice cropping system after straw incorporation. *Geochimica et Cosmochimica*
290 *Acta* 123, 120-133.
- 291 Wickramasinghe, D.B., Rowell, D.L., 2006. The release of silicon from amorphous silica and
292 rice straw in Sri Lankan soils. *Biol Fertil Soils* 42, 231-240.
- 293 Wilding, L.P., Hallmark, C.T., Smeck, N.E., 1979. Dissolution and Stability of Biogenic
294 Opal1. *Soil Sci. Soc. Am. J.* 43, 800-802.
- 295 Yamaji, N., Mitatni, N., Ma, J.F., 2008. A Transporter Regulating Silicon Distribution in Rice
296 Shoots. *The Plant Cell* 20, 1381-1389.

297 Yamanaka, S., Takeda, H., Komatsubara, S., Ito, F., Usami, H., Togawa, E., Yoshino, K.,
298 2009. Structures and physiological functions of silica bodies in the epidermis of rice
299 plants. *Applied Physics Letters* 95, 1-3.

300 Zhuravlev, L.T., 2000. The surface chemistry of amorphous silica. Zhuravlev model. *Colloids*
301 *and Surfaces A: Physicochemical and Engineering Aspects* 173, 1-38.

302

Table caption

Table 1. Specific surface area (SSA), chemical composition of the rice straw and its phytolith ash samples obtained from 300 to 1000°C ashing temperatures.

Figure captions

Figure 1. 3D images of the phytolith's siliceous structure (a) and it is embedded by organic matter (b)

Figure 2. SEM images of a leaf fragment in treated rice-straw samples: 400°C (a), 700°C (b)

Figure 3. XRD patterns (a) and IR spectra (b) of rice-straw samples treated at different temperatures

Figure 4. Release of Si (a) and P (b) from rice straw samples treated at 400°C, 700°C and 1000°C in a time sequence up to 6 d. Data points represent mean values with standard deviations for 3 replicates. Error bars might be smaller than the symbol size.

Figure 5. Solubility dependence of Si and P on treatment temperature. Data points represent mean values with standard deviations for 3 replicates. Error bars are smaller than the symbol size.

Figure 6. Correlation between soluble Si and P in the supernatant of 400°C-, 700°C-, and 1000°C-treated samples in the kinetic experiments. Where not visible, error bars are smaller than the symbol size.

Figure 7. Illustration of temperature effect on P and Si co-release from rice straw derived phytolith via changing occluded organic matter content, surface area and crystallization degree.