

Abschlussbericht

Doktorandenprojekt der P-Campus Graduiertenschule

Phosphorus speciation in soil and sediment indicating
transformation processes from terrestrial to aquatic
ecosystems

Julia Prüter

BetreuerIn: Prof. Peter Leinweber, Dr. Dirk Michalik

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1 Summary and conclusion

Phosphorus (P) is an indispensable nutrient element for all organisms but at the same time contributes significantly to excessive eutrophication in aquatic ecosystems. Knowledge about the chemical P speciation is essential to assess possible risks of P transport towards vulnerable aquatic ecosystems. To date, this knowledge, especially for transects including soil and sediment samples from terrestrial to aquatic ecosystems, is still insufficient. Thus, the general objective of the present work was to characterize the P speciation and to detect transformation processes along transport pathways at the fluent boundaries between land and sea including terrestrial soils, semiterrestrial wetlands and aquatic sediments. Besides analyses of elemental concentrations and water contents, terrestrial/semiterrestrial soils and aquatic sediments from micro-, meso- and macroscale ecosystems were analyzed by a multi-method approach including sequential P fractionation, P *K*-edge X-ray absorption near edge structure (XANES) spectroscopy and ³¹P nuclear magnetic resonance (NMR) spectroscopy. The detection of specific organic P species with terrestrial origin in arable soils as well as in adjacent sediments supported the assumption of direct P transport processes from terrestrial towards aquatic ecosystems. The complementary results confirmed a transition from labile and moderately labile Fe- and Al-associated P and great variety of organic P species in terrestrial soils towards more stable Ca- and Mg-P and less different organic P species in aquatic sediments. Kettle hole sediments, coastal wetland soils and Baltic Sea lagoon sediments acted as sinks for accumulations of especially stable P and complex organically bound P species. Some of the stable P species in aquatic sediments at the bottom of lake or sea waters can originate from Ca-P dominated fish fecal matter. To protect vulnerable aquatic ecosystems from further P inputs, kettle holes, coastal wetland buffer strips and deeper areas in shallow Baltic Sea lagoons have to be preserved and maintained or created in areas where they have not been available so far. Forthcoming studies have to focus on possible P recycling from terrestrial as well as aquatic sinks to contribute to closing P cycles in agricultural fertilization and adjacent ecosystems.

2 Introduction and objectives

Phosphorus (P) is essential to all living organisms and in the form of fertilizer an integral part of the current intensive agriculture. Once applied to arable fields, translocation and leaching of P from the soils is possible. However, inputs of P to aquatic environments can cause severe problems all over the world. Next to nitrogen (N), P has long been identified as main contributor to eutrophication of aquatic

ecosystems and even small quantities can cause serious consequences such as excessive algae blooming, release of toxins, reduced water transparency, oxygen depletion, and a general deterioration of the water quality (Bonsdorff et al., 1997).

Phosphorus can be present in many inorganic (P_i) and organic (P_o) chemical forms in different ecosystems (Cade-Menun et al., 2019). Soil P_i can appear as orthophosphate anions in solution, orthophosphate minerals or orthophosphate sorbed to mineral surfaces and organic matter (OM), while P_o occurs in forms where P atoms are bonded to carbon (C), such as phosphomonoesters or phosphodiesteres. Furthermore, there are condensed forms of P, such as pyrophosphate or polyphosphates with origins in living organisms (McLaren et al., 2020). The chemical form of P is one of the major factors influencing the risk of P mobilization and transportation from arable soil to aquatic environments (Kerr et al., 2011). The P speciation in terrestrial soils and aquatic sediments depends on various biological and chemical transformation processes such as sorption, reduction/oxidation, precipitation and mineralization. Thus, many current studies addressed the research topic of chemical P speciation in agricultural soils (e.g. Koch et al., 2018; Turner et al., 2008; Schmieder et al., 2020), sediments (e.g. Frankowski et al., 2002; Łukawska-Matuszewska and Bolalek, 2008; Kraal and Slomp, 2014) and biogenic materials such as organic fertilizers or biological soil crusts (Ajiboye et al., 2007; Baumann et al., 2017). However, it is not only important to investigate the P speciation in individual samples such as soil, water or sediment, but also to reveal functional relations between P from different environmental compartments to derive knowledge about transformation processes along transport pathways of P in terrestrial and aquatic ecosystems. Investigations about the P speciation and their transformation processes along transects for example from arable soil to aquatic sediments are scarce.

The general objective of the present work was to uncover the P speciation and to detect functional interactions between single chemical P compounds and transformation processes of P along transport pathways from terrestrial to aquatic environments including terrestrial, arable soils, semiterrestrial wetlands and aquatic sediments along sequences in different sized ecosystems. In this context, the specific objectives were (i) to characterize the P speciation in terrestrial and semiterrestrial soils and aquatic sediments by a multi-method approach, (ii) to detect transformations within the P composition along transport pathways from terrestrial soils towards aquatic sediments in three ecosystems of different size, and (iii) to uncover possible sinks in the environment where P can be trapped and conserved.

3 Material and methods

The present work covers three investigation areas with various sizes including soil and sediment samples along sequences from terrestrial to aquatic conditions (Figure 1). The first investigation area, rather small in size, is represented by samples along a transect from arable soil to sediments from a kettle hole with a maximum distance of about 70 m between all samples. The second sampling set of soils and sediments along a sequence starting at arable soils leading to sediments from a small lagoon of the Baltic Sea, called 'Bodden', within about 700 m illustrates a mesoscale ecosystem. Finally, the largest investigated area representing a macroscale transect, includes soils and sediments from coastal areas up to the central Baltic Sea with distances up to 600,000 m between the samples.

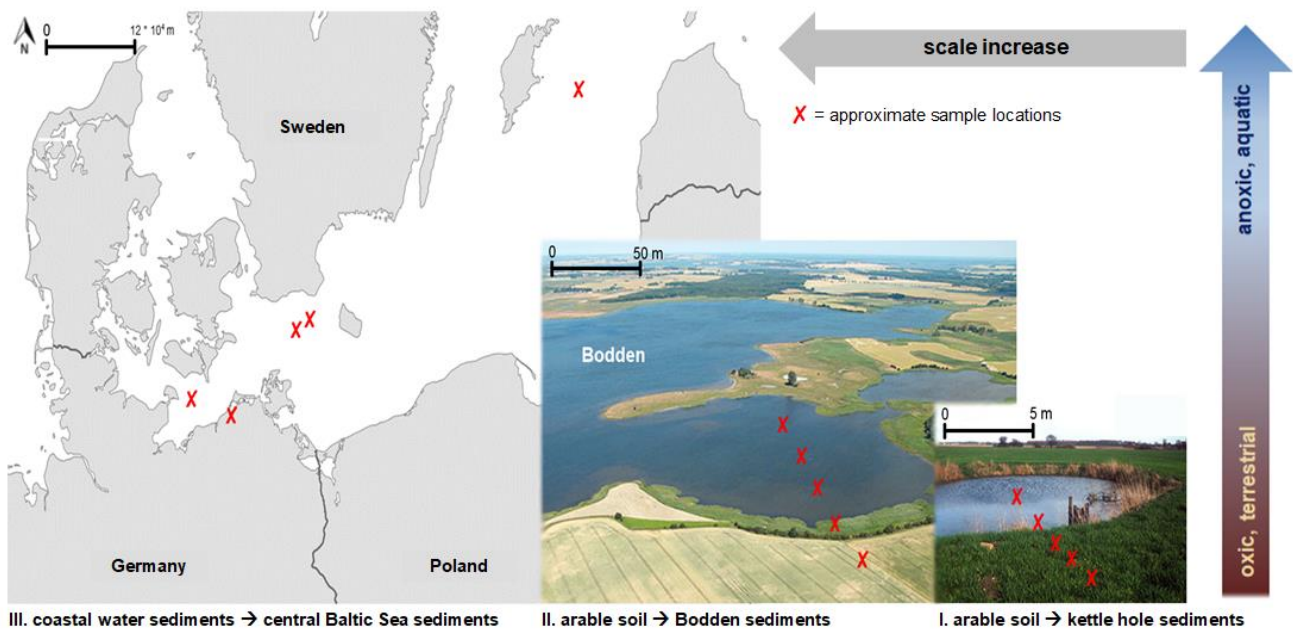


Figure 1 Sampling strategy in three differently sized environments (I – microscale, II – mesoscale, III - macroscale).

To gain a comprehensive picture of the P speciation in soil and sediment samples from different environments, a combination of traditional methods in P research, such as chemical fractionations with advanced state-of-the-art methods, such as X-ray absorption near edge structure (XANES) spectroscopy and ^{31}P NMR spectroscopy is reasonable.

The sequential P fractionation established by Hedley et al. (1982), modified by Tiessen and Moir (1993), is one of the most widely used wet chemical methods to characterize pools of P in different environmental samples (Condon and Newman, 2011; Kruse et al., 2010; Negassa and Leinweber, 2009). In this fractionation scheme, a series of reagents with increasing extraction strengths is used to

successively extract P pools on the basis of interactions between P moieties and other components of the soil matrix (Condon and Newman, 2011).

The P *K*-edge XANES spectroscopy is an element-specific method to describe the P speciation in terrestrial soils as well as aquatic sediments (e.g. Acksel et al., 2016; Koch et al., 2018; Li et al., 2015; Prietzel et al., 2013). In contrast to sequential fractionation procedures, XANES spectroscopy has the advantage of direct sample usage without further extraction or destruction (Kruse et al., 2010; Prüter et al., 2020). However, XANES spectroscopy preferentially provides information about P_i species in soils and sediments. Thus, results of P_o concentrations from sequential fractionation or ³¹P NMR spectroscopy can be used to complement the XANES spectroscopy (Gu and Margenot, 2021).

Solution ³¹P NMR spectroscopy on soil extracts has been used for over 40 years to detect simultaneously all forms of P_o that could be brought into solution (McLaren et al., 2021). Prior to analysis with solution ³¹P NMR spectroscopy, P needs to be extracted from soil or other environmental samples such as sediments or organic amendments, with NaOH-EDTA by shaking the sample together with the extractant for several hours (Kruse et al., 2015). Most signals in NMR spectra of soil extracts were found in the orthophosphate and phosphomonoester region but there were also signals detected in the regions of phosphodiester, pyrophosphate, polyphosphate and phosphonate (Turner et al., 2002; Cade-Menun, 2005; McLaren et al., 2020).

4 Results and discussion

The general findings of the present work confirmed previous research results that agricultural soils were dominated by labile and moderately labile P fractions such as resin-P, NaHCO₃-P and NaOH-P, whereas most aquatic sediments contained more stable fractions such as H₂SO₄-P and residual-P determined by sequential P fractionation. Terrestrial, arable soils from the direct surrounding area of a kettle hole contained about 127 mg kg⁻¹ NaOH-P_o and 114 mg kg⁻¹ NaHCO₃-P_i. Sediments from the kettle hole were clearly dominated by residual-P (maximum of 1729 mg kg⁻¹). Soils from the coast of northern Germany from an experimental site at the University of Rostock contained up to 137 mg kg⁻¹ NaOH-P_i, while sediments from the Baltic Sea had concentrations of about 500 mg kg⁻¹ H₂SO₄-P.

The direct comparison of P concentrations between terrestrial/semiterrestrial soils and aquatic sediments was enabled by a similar sample pretreatment of the soils and sediments including drying, sieving and grinding. Although water contents of aquatic sediments are naturally different from

terrestrial soils and thereby the soils and sediments also differ in density, P concentrations were referred to dried samples and thus comparable at a standardized scale.

Results from XANES spectroscopy were similar for most soils but different from the sediments. We determined about 61 to 100% Fe-P and Al-P in terrestrial soils around a kettle hole. The aquatic sediments from the kettle hole still contained maxima of 74% Fe-P but also up to 43% Ca-P. Terrestrial soils from an experimental site in Rostock were dominated by maxima of 92% Fe-P and Al-P, whereas up to 100% of Ca-P was present in aquatic sediments from the Baltic Sea. In arable soils from an agricultural site near a shallow lagoon of the Baltic Sea we determined a maximum of 92% Ca-P and in the corresponding aquatic sediments 54 to 100% Ca-P. The high proportions of Ca-P in the soil can be explained by elevated concentrations of CaCO_3 in the terrestrial soil from the underlying parent material, promoting the formation of Ca-associated P compounds, whereas the dominance of Ca-P species in the aquatic lagoon sediments agreed with the results from other aquatic environments of the present investigations.

The results of sequential P fractionation and XANES spectroscopy were generally consistent among the three different scales of investigations of a kettle hole environment, soils and sediments around a shallow lagoon of the Baltic Sea and the total Baltic Sea. Although these investigated microscale, mesoscale and macroscale ecosystems differed not only in size but also in their biogeochemical basic conditions such as influence of agricultural land use, climate and weather or local flora and fauna, these factors did not fundamentally affect their P speciation. The general trend of transitions from labile and moderately labile P fractions and Fe and Al associated P species in terrestrial soils towards more stable P fractions and higher proportions of Ca-P in aquatic sediments was approved along different sequences for the first time. This transition appeared for the microscale kettle hole environment, as well as for the mesoscale Bodden test area with some exceptions within the soils due to erosion and the macroscale Baltic Sea investigation.

In addition to the results from sequential P fractionation and XANES spectroscopy, kettle hole soils and sediments and the Baltic Sea soils and sediments were analyzed with the method of ^{31}P NMR spectroscopy and the Baltic Sea sediments with SEM-EDX. The analysis of Baltic Sea sediments with SEM-EDX resulted in high numbers of Fe-phosphate particles near the coast and thereby supported the findings of sequential fractionation and XANES spectroscopy on the single-particle level. ^{31}P NMR spectroscopy determined a decrease of the variety of mono- and diester P compounds and an increase of ortho-P with growing distance from the coast in the Baltic Sea sample set. Also in the

kettle hole sediments, a lower variety of P_o species was found compared to the surrounding soils, particularly in the phosphomonoester region.

In addition to P transformations on its transport pathways from land to sea, the present work detected accumulations of stable Ca-P species and complex forms of P_o in kettle hole sediments compared to surrounding soils and especially moderately stable P, Al-P and P_o compounds in wetland soils between agricultural soils and natural aquatic sediments. Thus, kettle hole sediments as well as wetland soils act as sinks for P-containing particles and thereby can help to prevent direct transfer of various P species, e.g., by leaching or runoff during erosion events from agricultural soils to adjacent freshwater and/or marine resources protecting vulnerable aquatic ecosystems from further eutrophication.

Origins of P compounds in aquatic sediments can be diverse, depending on several factors such as evolution, geographical location and geological history of the respective water system. To discover, if P in sediments can also originate from aquatic organisms such as fish naturally occurring in fresh- and saltwater ecosystems, the P speciation of fish fecal matter was investigated. The solid waste from African Catfish in a recirculation aquaculture system (RAS) exclusively contained Ca-bound P compounds as determined by XANES spectroscopy. Thus, the contribution of fish fecal matter can be an additional influencing factor on the Ca dominated and stable P species in aquatic sediments of the present investigations, even if further research about naturally occurring fish is needed next to studies about fish cultured in cages and from RAS.

Movement of P can occur in dissolved or particulate forms and it already has been identified a long time ago that 75 to 90% of P in agricultural land is transported by soil erosion (Sharpley et al., 1993). The present investigation confirmed transport pathways of P from arable soils to kettle hole sediments, Baltic Sea lagoon sediments and Baltic Sea sediments. Although the general P speciation changed from soil to sediment in all investigated test areas, specific P compounds within the sediments suggested their terrestrial origin. The P_o compound of IP_6 has been identified abundantly in terrestrial soils because it is known to act as P storage compound in plants, especially in developing seeds and has strong sorption properties to clays and soil OM (e.g. Turner et al., 2002; Negassa et al., 2010; Noack et al., 2014; Alewell et al., 2020). In aquatic sediments, IPs have been detected in substantial quantities, although only few aquatic organisms are able to synthesize them (Turner et al., 2002). Thus, most of the IPs present in aquatic sediments are suggested to be derived from terrestrial environments. We were able to determine *scyllo*- IP_6 , *myo*- IP_6 and *neo*- IP_6 in arable soils as well as in

adjacent kettle hole sediments confirming transport processes of IP_6 from terrestrial soil to aquatic sediments. Furthermore, the entry of soil aggregates to fluvial systems was also confirmed earlier by differentiating parameters such as density, porosity and settling velocity between soil aggregates and fluvial sediments (Droppo et al., 2004).

Phosphorus compounds entering aquatic environments are either released to the water column and consumed by aquatic organisms such as algae and macrophytes or accumulated within the sediment. Soil particles, containing high concentrations of Fe-associated P, can be transported towards aquatic ecosystems with sediments under anaerobic reducing conditions. The lack of oxygen in sediments results in a reduction of ferric (Fe^{3+}) to ferrous (Fe^{2+}) phosphates of which the latter are more soluble in water (Thomas, 1970). Investigations of kettle hole sediments and adjacent soils and Baltic Sea lagoon sediments confirmed that especially stable P fractions such as H_2SO_4 -P and P associated with Ca and/or Mg can be accumulated and conserved in deeper sediments lowering the probability of P mobilization into the above water column. The input of P from agriculture to adjacent waters can also be significantly reduced by wetland soils along shore areas. We identified accumulations of especially moderately stable P, Al-P and P_o compounds in *Phragmites* wetland soils saving adjacent aquatic ecosystems from direct inputs of these P species. Thus, it is one of the most important tasks of the future to create new, or to preserve and maintain existing buffer strips and coastal wetlands by removing P with harvests of the plant cover such as reeds or to find ways to regain P from the terrestrial as well as aquatic sinks to enable nutrient recycling. This is the only way we can slow down the progressive eutrophication of waterbodies around the world including associated problems such as biodiversity losses, ecosystem degradation, harmful algae blooms and oxygen deficiency (Oliveira and Machado, 2013). At the same time we can contribute to P recycling in agricultural areas in order to be able to use the limited reserves of rock phosphate more economically and environmentally friendly to answer global food necessities.

5 References

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Appendix

List of posters and presentations at conferences and public relations activities

25.11.2022	Presentation „ <i>Phosphorus speciation in soil and sediment indicating transformation processes from terrestrial to aquatic ecosystems</i> “ at the International P-Campus Symposium
06.01.2022	Poster „ <i>Influence of sample pretreatment on P speciation in sediments evaluated with sequential fractionation and P K-edge XANES spectroscopy</i> “ at the International P-Campus Symposium
20.05.2021	Presentation „ <i>Phosphor entlang seiner Transportwege – von terrestrischen in aquatische Ökosysteme</i> “ at P-Campus Lecture Series
17.11.2020	Poster „ <i>Organic matter composition and P speciation of solid waste from an African Catfish Recirculating Aquaculture System</i> “ at the International P-Campus Symposium
13.09.2019	Presentation „ <i>Transformation processes of phosphorus along transects in the coastal region of Northeastern Germany</i> “ at the WETSCAPES conference
26.08.2019	Poster „ <i>Phosphorverbindungen entlang eines Transektes in der Nordostdeutschen Küstenregion</i> “ at the Annual Meeting of the German Soil Science Society
25.04.2019	Support of the exhibition of P-Campus at the Long Night of Science at Rostock
19.11.2018	Presentation „ <i>Phosphorus speciation in soil and sediment: clue to understanding the P-dynamics at the land-sea boundary</i> “ at the P-Campus Symposium
14.09.2018	Presentation „ <i>Phosphorus speciation in soil and sediment: clue to understanding the P-dynamics at the land-sea boundary</i> “ at the Leibniz Conference on Sustainable Development Goals

List of research stays outside of the supervising institutions

05. - 12.11.2017	SLRI Thailand: XANES analyses of soil and sediment samples
04. - 13.11.2018	SLRI Thailand: XANES analyses of soil and sediment samples
05. - 19.11.2019	ETH Zürich: NMR analyses of soil and sediment samples

List of publications

- February 2023 Prüter, J., Schumann, R., Klysubun, W., Leinweber, P.:
„*Characterization of phosphate compounds along a catena from arable and wetland soil to sediments in a Baltic Sea lagoon*“ published in Soil Systems
- November 2022 Prüter, J., McLaren, T.I., Pätzig, M., Hu, Y., Leinweber, P.:
„*Phosphorus speciation along a soil to kettle hole transect: sequential P fractionation, P XANES, and ³¹P NMR spectroscopy*“ published in Geoderma
- April 2022 Prüter, J., Hu, Y., Leinweber, P.:
„*Influence of sample pretreatment on P speciation in sediments evaluated with sequential fractionation and P K-edge XANES spectroscopy*“ published in Communications in Soil Science and Plant Analysis
- October 2020 Prüter, J., Strauch, S. M., Wenzel, L., Klysubun, W., Palm, H.W., Leinweber, P.:
„*Organic matter composition and phosphorus speciation of soil waste from an African Catfish Recirculating Aquaculture System*“ published in Agriculture
- December 2019 Prüter, J., Leipe, T., Michalik, D., Klysubun, W., Leinweber, P.:
„*Phosphorus speciation in sediments from the Baltic Sea, evaluated by a multi-method approach*“ published in Journal of Soils and Sediments