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Phosphorus content in water extractable soil colloids over a 2000 years chronosequence of paddy-rice management in the Yangtze River Delta, China

Xiaoqian Jiang [a,b,*], Amelung Wulf [b,c], Roland Bol [b,d], Erwin Klumpp [b]

[a] School of Agriculture, Sun Yat-sen University, Guangzhou, Guangdong 510275, PR China
[b] Institute of Bio- and Geosciences, Agrosphere Institute (IBG-3), Forschungszentrum Jülich GmbH, Jülich 52428, Germany
[c] Institute of Crop Science and Resource Conservation, Soil Science and Soil Ecology, Nussallee 13, University of Bonn, Bonn 53115, Germany
[d] School of Natural Sciences, Bangor University, Bangor LL57 2UW, United Kingdom

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ABSTRACT

Soil phosphorus (P) is usually hardly present in soluble forms as most of it is strongly bound to minerals. Therefore, transformations of soil colloidal P can play a key role in enhancing soil fertility. Here, we examined water extracts from a chronosequence of soils with 2000 years of paddy rice and 700 years of non-paddy cropping, and fractionated them into <1200 nm, <450 nm, and <3 kDa size fractions using sequential ultrafiltration. Asymmetric-flow field-flow fractionation (AF4) coupled to an organic carbon detector (OCD) and inductively coupled plasma mass spectrometry (ICP-MS) was used for organic carbon and elemental screening in the 3 kDa-450 nm colloidal size range. We found that P increased in all colloidal fractions both with long-term paddy rice and non-paddy managements. Furthermore, P in soil colloids of 3 kDa-450 nm size, as fractionated by AF4, peaked in three size ranges: nanoparticles (NP, ~3 kDa-20 nm), fine colloids (FC, ~20–225 nm), and medium-sized colloids (MC, ~225–450 nm). The NP fraction originally contained mainly P and Ca for tidal wetland (TW), but turned into a complex of OC-Ca-P colloidal forms immediately after onset of arable management, with little temporal change thereafter. Fine colloidal P (FC-P) was detected only after > 50 years for the non-paddy and > 100 years under paddy management, associated with oxides, organic matter and fine clay fragments in the paddy soils and additionally Ca-bound P in the non-paddy ones, respectively. Increasing portions of medium-sized colloidal P appeared after 50 years paddy and non-paddy management, though not exceeding the concentrations of FC-P and with little changes during prolonged arable land use. Overall, after initial formation of FC-P during the first 50–100 years of management, longer term dynamics of colloidal P in these arable soils was fairly independent of paddy or non-paddy managements, but remained closely coupled to the increased presence or generation of fine colloidal clays, oxides and the associated P, thus potentially linking P fertility largely to the dynamics of fine colloids.

1. Introduction

>50 % of the world́s population feeds on rice and submerged rice cultivation results in the formation of paddy soils. Paddy soils occupy 11 % (1,956,000 km^2) of cultivated land area (Kögel-Knabner et al., 2010; Huang et al., 2013; Huang et al., 2021). Regular chemical and organic fertilization containing P are needed to improve and maintain soil fertility and rice yield (Huang et al., 2015). Large proportions of P in water and soil, however, are usually associated with colloids (i.e. particles with size of 1–1000 nm) (Jiang et al., 2015; Gottselig et al., 2017), which are both mobile carriers and potential sinks for plant-available P (Gottselig et al., 2014; Montalvo et al., 2015; Konrad et al., 2021). Transformations of colloidal P including alterations in size distribution and particle compositions are thus of crucial environmental and ecological significance (Li et al., 2021).

Long-term paddy rice cultivation results in a significant alteration of hydromorphic soil properties due to paddy soil formation initiated by redox cycles (Kölbl et al., 2014). Many studies have reported that drying/rewetting cycles could simulate the release of soil colloids and nanoparticles (i.e., particle size <100 nm) (Klitzke and Lang, 2007;

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* Corresponding author at: School of Agriculture, Sun Yat-sen University, Guangzhou, Guangdong 510275, PR China.
E-mail address: jiangxq7@mail.sysu.edu.cn (X. Jiang).

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Majdalani et al., 2008; Mohanty et al., 2015; Gu et al., 2018). Correspondingly, the formation and distribution of P-containing soil colloids and nanoparticles could also change during the long-term paddy rice management. Transport, solubility, reactivity, and bioavailability of colloidal P complexes are all directly linked to the form of P associated with different soil minerals and its particle size (Lindsay et al., 1989; Beauchemin et al., 2003; Liu et al., 2014; Huang et al., 2021). For example, P associated with Fe-(hydr)oxides is sensitive to reducing conditions and could be potentially released and available after the reduction of Fe-(hydr)oxides. However, calcium phosphate precipitates are perceived to be more vulnerable to pH changes and the bioavailability of P increases under acidic condition (Lindsay et al., 1989; Beauchemin et al., 2003; Liu et al., 2014). Additionally, colloidal particles with smaller size such as nanoparticles can be highly mobile, thus playing a disproportionate role in P transport and bioavailability (Huang et al., 2021). Currently, some studies have investigated colloidal P according to different particles size in other ecosystems (Gottselig et al., 2014, 2017; Jiang et al., 2015, 2017b; Missong et al., 2018a; Gu et al., 2020; Li et al., 2021). However, information about the different-sized colloidal P transformation during long-term paddy managements remains scarce, despite its potential pivotal role for long-term crop P supply. The emergence of asymmetrical flow field flow fractionation (AF4) coupled online with organic carbon detector (OCD) and an inductively coupled plasma mass spectrometer (ICP–MS) has been widely used in the last decade both in aquatic and soil ecosystems (Stolpe et al., 2013). This technique nowadays provides continuous real-time analysis of organic matter and elemental compositions of the different-sized particles.

Water-extractable colloids (comparable with water dispersible colloids, WDC) are easily extractable on contact of soil with water and are considered as indicators for mobile soil colloids (de Jonge et al., 2004; Jiang et al., 2014; Missong et al., 2018b). Although, of course they do not reflect the full status of colloids in rice-paddy or non-paddy soils. Water-extractable colloids have however previously been used to compare the redox-driven colloidal changes in paddy soils or flooding grassland soils (Jiang et al., 2017a,b;Said-Pullicino et al., 2021). In this study, we used AF4 coupled with OCD and ICP-MS to characterize P distribution and binding of different-sized colloids in soil water extracts. We compared chronosequences of soils that had been under 0–2000 years of paddy-rice and 0–700 years of adjacent non-paddy cropping in the Yangtze River Delta, China. The study aimed to assess how prolonged paddy management influenced the transformation of colloidal P with regards to particle size distribution and composition.

2. Materials and methods

2.1. Site description and water extraction

The studied soils were located in the Bay of Hangzhou near the city of Cixi (30°10′N, 121°14′E), Zhejiang Province, China. The last 2000-year dike building for land reclamation resulted in a chronosequence of soils containing paddy and non-paddy soils. The marine Yangtze River sediment in a tidal wetland (TW, 0–20 cm) was taken as substrate reference. After 30-year land embankment behind the youngest dike built in 1977, an initial salt marsh (SM, 0–13 cm) with the growth of salt-tolerant shrubs was considered as the starting point for our land use. The soil chronosequence contained six sites under paddy cultivation for 50, 100, 300, 700, 1000, and 2000 years (PR50-2000), as well as four adjacent sites under non-paddy cultivation for 50, 100, 300, and 700 years (NPR50-700), respectively. The investigated soils were used for paddy rice cultivation in the wet season, followed by wheat or another upland crop during the dry season (Fan et al., 2005; Roth et al., 2011; Zou et al., 2011). The non-paddy soils were mainly cropped with vegetables, rape (Brassica sp.), and wheat (Lehndorff et al., 2014). Organic fertilization (including animal manure, rice straw and other crop residues, often fermented with sediments taken from the river or channel, but also charred residues), liming, and mineral fertilizer were applied in these fields (Gong, 1983; Cao et al., 2006; Kögel-Knabner et al., 2010). We cannot guarantee that a constant management was conducted at all the sites during the 2000 years of paddy and 700 years of non-paddy soil developments. Nevertheless, the changes of general managements and environmental conditions likely comparably affected both paddy and non-paddy soils at sampling sites along the chronosequence (Kalbitz et al., 2013).

In late spring 2008, shortly after the harvest of the upland crops, at the time of rice transplanting, profiles at every site on different fields were sampled for analyses, with a distance of at least 50 m from each other. Three field replicates were sampled, except for PR50 and PR700 here only two and for TW, SM and PR2000 where only one independent field replicate was collected. All topsoil samples (~0–15 cm) were air-dried on site during about 24 h, and sieved to a size <2 mm. The pH values of surface soil were 8.2 and 7.8 in the youngest soil (TW and SM) and declined to 5.1–5.9 after 100–2000 years of paddy-rice management and 700 years of non-paddy-rice management (Kölbl et al., 2014) (Table S1, Supplementary Materials). All paddy soils were classified as Cambisols or Stagnosols (IUSS Working Group WRB, 2006). More details were provided in other publications (Fan et al., 2005; Roth et al., 2011; Kölbl et al., 2014). The 5 g of paddy and non-paddy soils were horizontally shaken at 150 rpm with 40 mL of ultrapure water for 24 h. The soil suspensions were subsequently centrifuged at 3000 × *g* for 10 min to remove coarse particles and then were filtered through 1200 nm, 450 nm, and 3 kDa cellulose membranes to obtain soil water extracts of <1200 nm, < 450 nm, and <3 kDa. Then P from those three soil water extracts was measured by ICP-MS.

It is not avoidable that the long-term storage of air-dried soils before experiments could potentially affect soil oxidation and reaggregation. Yet, Bollen (1977) has found that soil samples in dry storage for 54 yr retained their ability to respire and oxidize, suggesting that potential bias is small. Nevertheless, increasing time of storage after drying increases the risk of elevated surface acidity, solubility and oxidizability of soil organic matter (Bartlett and James, 1980). Siebers et al. (2018) showed that air-drying led to reaggregation of soil particles with slightly changed morphology, however, the authors did not investigate subsequent effects on the potential release of water-dispersible colloids. There is no indication that any bias by prolonged storage is dissimilar for the different soils and their management, particularly since both paddy and non-paddy soils were sampled under no-flooded conditions, at field-dry state. Therefore, sample storage is unlikely to affect temporal peculiarities in the potential release of water-dispersible colloids.

2.2. Measurements of soil colloidal size distributions and compositions

The colloids from soil water extracts of <450 nm were determined by AF4, which is a chromatography-like elution technique (Baalousha et al., 2011) that separates colloids on a continuous scale based on their ability to diffuse against a cross flow of liquid in an open channel. The AF4 was coupled online to an OCD for assessing organic carbon concentrations, and ICP-MS for monitoring the P, Fe, Al, Si, and Ca contents of the colloids, respectively. The AF4 system was equipped with 500 μm spacer and a 1 kDa polyethersulfone (PES) membrane. The details about the carrier and separation method were described in our previous study (Jiang et al., 2015). In brief, A 25-mM NaCl solution at pH 5.5 served as the carrier, which provided a good separation condition. During the focusing time of 15 min, soil solution containing small particles with size <1 kDa passed through the PES membrane and only > 1 kDa particle elements remained in the separation channel. Thereafter, the cross flow was maintained at 2.5 mL min^{-1} for the first 8 min of elution time, then set to decrease linearly to 0.1 mL min^{-1} within 30 min, and maintained it for 60 min. Finally, it then declined to 0 mL min^{-1} within 2 min and kept 20 min for eluting the residual particles. In addition, to allocate the mineral composition within and on the soil colloids, the soil water extract of PR300 and NPR300 was centrifuged to obtain a suitable concentration of colloid for transmission electron microscopy (TEM) and energy dispersive X-ray spectroscopy (EDX) measurements.

2.3. Statistical analyses

Elemental concentrations in different-sized soil fractions from paddy and non-paddy managements were tested for significant differences (set to $P < 0.05$) using SPSS 18.0 (SPSS Inc., Chicago, IL, USA). An independent-samples *t* test was conducted to determine the significance of differences between paddy and non-paddy soils at a given age sites. One-way ANOVA was used to test significant differences among the full paddy soil chronosequence and non-paddy soil chronosequence, respectively. Data were assessed with Shapiro–Wilks and Brown–Forsythe tests to meet the criteria of normal distribution and homogeneity of variances respectively.

3. Results and discussion

The physical and chemical properties for soil samples beyond the size range of water extractable colloids as characterized by AF4 and TEM were not analyzed, because this information was available from other studies (Roth et al., 2011; Kölbl et al., 2014). The existing information about mineralogy, and potentially relevant for understanding the variability in the colloid mass of recovered soil, i.e., grain size distribution, and chemical composition is summarized in Table S1 (Supplementary Materials). All the samples contained similar type of minerals, including quartz, feldspars (i.e., plagioclase, little orthoclase), mica, calcite, dolomite, kaolinite, chlorite, and vermiculite. It is noteworthy that the portion of clays increased upon paddy and non-paddy cultivation, thus potentially promoting release or formation of soil colloids. The soil chemical composition was also similar among sites except for Ca. The Ca concentration decreased upon cultivation due to the decalcification (Kölbl et al., 2014), which suggests that under the long-term cultivation also the chemical composition of soil colloids could change.

It is nearly unavoidable that the processes of soil air-drying and subsequent rewetting during the water extraction bring about a potential release of colloidal P (mainly by a physical process, i.e., soil aggregates disruption) (Gu et al., 2018; Nguyen et al., 2020). Therefore, although water-extractable colloids were generally chosen to investigate the redox-driven changes of soil colloids (Said-Pullicino et al., 2021), the natural amount of soil colloids was likely overestimated inevitably. In any case, the tidal wetland (TW) sediment, i.e., the parent formation material of paddy and non-paddy soils, had extremely low P concentration in surface soil <1200 nm colloidal fractions (Fig. 1). After land embankment, soil formation started with the salt marsh (SM), showing an increase of colloidal P fractions for the 3 kDa-450 nm fraction. Thereafter, with the prolonged paddy management up to 2000 years (yr), the P concentrations in larger colloidal fraction (i.e., 450–1200 nm) did not significantly increase. The <3 kDa fraction did only show elevated P concentrations in the oldest (2000 yr) paddy soil. The colloidal P concentrations of <1200 nm, due to the increase of colloids between 3 kDa and 450 nm, did increase in the paddy managements > 100 yr. The colloidal P concentrations <1200 nm within the non-paddy soils did also show similar temporal trends. The clay and Fe/Al oxides content in the paddy and non-paddy soils did accumulate with the duration of the crop managements and thus soil formation (Kölbl et al., 2014). It is thus likely that observed changes in colloidal size distribution and respective P contents are mainly controlled by pedogenesis, weathering and C accrual, rather than solely by the presence of redox cycles (Pan et al., 2004; Huang et al., 2013; Kölbl et al., 2014; Jiang et al., 2017a,b). Overall, however, there were no significant differences of three soil fractions between paddy and non-paddy soils.

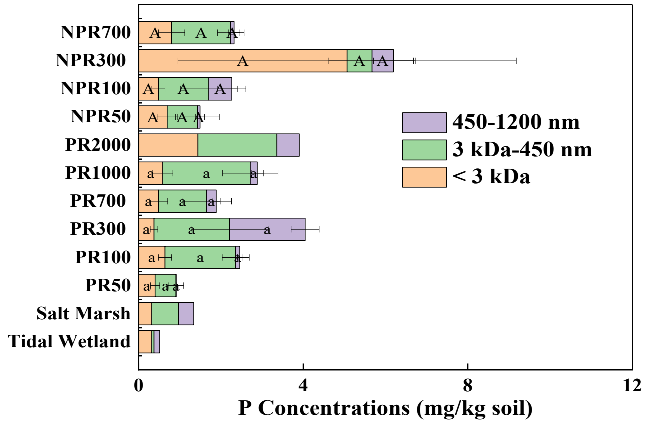

Fig. 1. Phosphorus concentrations of <3 kDa, 3 kDa-450 nm, and 450–1200 nm soil fractions (mg/kg soil) during 2000 years paddy and 700 years non-paddy managements. PR50–2000: 50–2000 years paddy soils; NPR50–700: 50–700 years non-paddy soils. Different lowercase and uppercase letters indicate significant differences of soil fractions among soil chronosequences for paddy and non-paddy managements, respectively (one-way ANOVA with Tukey's HSD test; $P < 0.05$).

The AF4-OCD-ICP-MS results (Figs. 2 and 3) showed that the soil colloids between 3 kDa and 450 nm consisted of three size fractions: namely, nanoparticles (NP, ~3 kDa-20 nm), fine colloids (FC, ~20–225 nm), and medium-sized colloids (MC, ~225–450 nm). Size assessment was based on dynamic light scattering results and supported by the spiking of sulfate latex standard particles (Jiang et al., 2015). The NP fraction contained P, Ca, and OC, i.e., it was likely dominated by OC-Ca-P associations, while Fe, Al, and Si were not detected. This result was similar with the study of paddy soils by Li et al. (2021), but was different from our own previous research on Luvisol and Stagnosols (Jiang et al., 2015, 2017b), where nano-sized colloidal P consisted of OC-Fe/Al-P associations, and other studies of paddy/non-paddy soils, where no or only few elements were detected in nano-sized fraction (Said-Pullicino et al., 2021). The soils of this study had developed from marine sediment with alkaline pH and contained large amounts of $CaCO_3$ (Kölbl et al., 2014). It seems that this played an important impact on the combination formation of soil nanoparticle P. The TW had a high pH of 8.2 and contained high Ca content (32.1 mg/g) (Kölbl et al., 2014). The NP of TW samples did contain mainly Ca and P. After the initiation of arable management, nanoparticulate P was likely associated with OC and Ca due to the increased accumulation of OC. In calcareous soils, Ca^{2+} ions do presumably bridge P and negatively charged organic substances with carboxyl or phenolic groups (Lin et al., 2017; Yang et al., 2019; Wang et al., 2020). As the P concentrations of NP fractions remained relatively stable over the chronosequence, we assumed that the nanoparticulate P was not influenced by the duration or type of arable management.

The concentration of OC in the NP fraction was low in the TW sample, but rose rapidly after 50 yr of arable management, and remained stable thereafter, which was in line with the common total C accrual over time in these soils (Kalbitz et al., 2013). The concentrations of Ca in NP fraction did not have a significant increase with a longer duration of the arable managements. As a result, the P concentration of NP did not significantly change with the chronosequence of soils, suggesting that the actual binding capacity of P to NP was limited. Li et al. (2021) found identical results for the arable soils from the same region in China.

A small amount of medium-sized colloidal (i.e., MC) P was detected after 50 yr in both the paddy and non-paddy soil, which remained constant with longer arable management (Fig. 3). Furthermore, fine colloidal (i.e., FC) P was generated only after > 50 yr non-paddy and > 100 yr paddy management, but then in higher concentrations than for medium-sized colloidal P (Fig. 3). Clearly, long-term paddy and non-paddy managements both enhanced the generation of colloidal P, dominated by 20–225 nm fractions (i.e., FC). However, there was no significant difference for the P concentrations in fine colloids between prolonged paddy and non-paddy managements. Paddy-rice

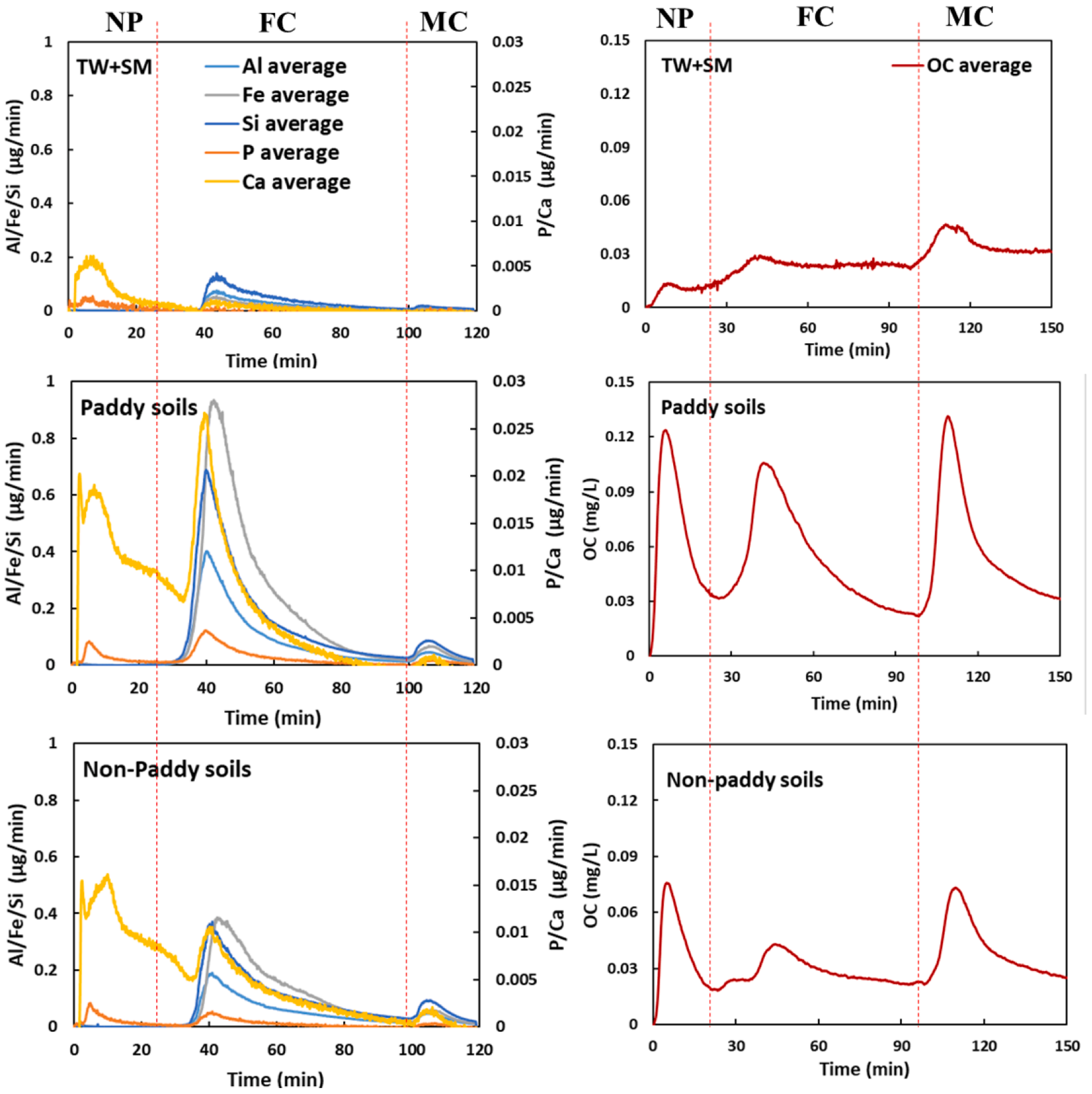

Fig. 2. Asymmetric flow field-flow fractionation (AF4) average fractograms of colloids <450 nm from Tidal Wetland + Salt Marsh (n = 2), paddy soils (n = 14), and non-paddy soils (n = 12). The fractograms show the Al, Fe, Si, P, and Ca mass flow monitored by inductively coupled plasma mass spectrometer (left) and the organic carbon concentration (OC) monitored by organic carbon detector (right).

management could also generate high amounts of fine soil colloids (e.g., in PR100 and PR1000). This in view of the annual flooding and drainage, could be associated with high potential transport and loss risk. Thus, inconsistent patterns of colloidal P accumulation under prolonged paddy managements in this study probably reflected localized differences in the intensity of overall soil and crop management, as well as flooding or floodwater removal. The FC and MC fractions all contained P, Fe, Al, Si, Ca, and OC. While Fe and Ca can be directly involved in the binding of P, the majority of Si and Al was likely part of clay minerals (Jiang et al., 2015). Fig. 4 illustrates TEM images and EDX spectra of colloidal particles with size of <450 nm for PR300 and NPR300. Both samples coincided with a dominance of either C and Si or of various elements including C, Fe/Ti, Al, Si, Ca, and P. It highlights that the colloids from paddy and non-paddy soil comprised a complex of organic carbon and minerals probably including quartz, clay minerals, Fe/Ti, and Ca containing minerals.

With the significant changes in the contents of fine colloids occurred after 100 yr of paddy management, fine-colloidal P, Fe, Al and Si were formed concomitantly, suggesting that oxides and fragments of clay minerals were largely liberated after this time of management. This release coincided with a drop in pH values by about 2 units from the 50 to the 100 yr old paddy soil (Kölbl et al., 2014) (see also Table S1, Supplementary Materials), suggesting that acidification and related decalcification likely facilitated oxide dissolution and deterioration of Ca-stabilized aggregates, and therewith promoting also the release of associated fine-colloidal P. Additionally, the organic carbon also accumulated after 100 yr of paddy-rice cultivation and thus elevated Ca^{2+} concentration due to decalcification leads to more bridging between OC and clay minerals (Jiang et al., 2019), which results in the formation of more organo-minerals on clay-OM basis but also Fe and Al oxide basis. On the other side, our previous study related to these soils found that organic P accumulated in bulk soil after both paddy and non-paddy cultivation (Jiang et al., 2017a,b). Here AF4-ICP-MS does not differentiate between inorganic and organic P forms, which likely bound different to oxides and fine clay fragments, respectively.

In the non-paddy soils, fine colloidal P (i.e. FC) generates after 50 yr cultivation (Fig. 3A). The decalcification and acidification were delayed (Kalbitz et al., 2013), and pH remained elevated until 700 years of non-paddy management (Kölbl et al., 2014) (Table S1). This delay likely reflected the increased mechanical disturbance in these non-paddy fields by upland cropping, known to promote oxidative losses of soil organic matter and therewith of additional aggregate bonding agents. The

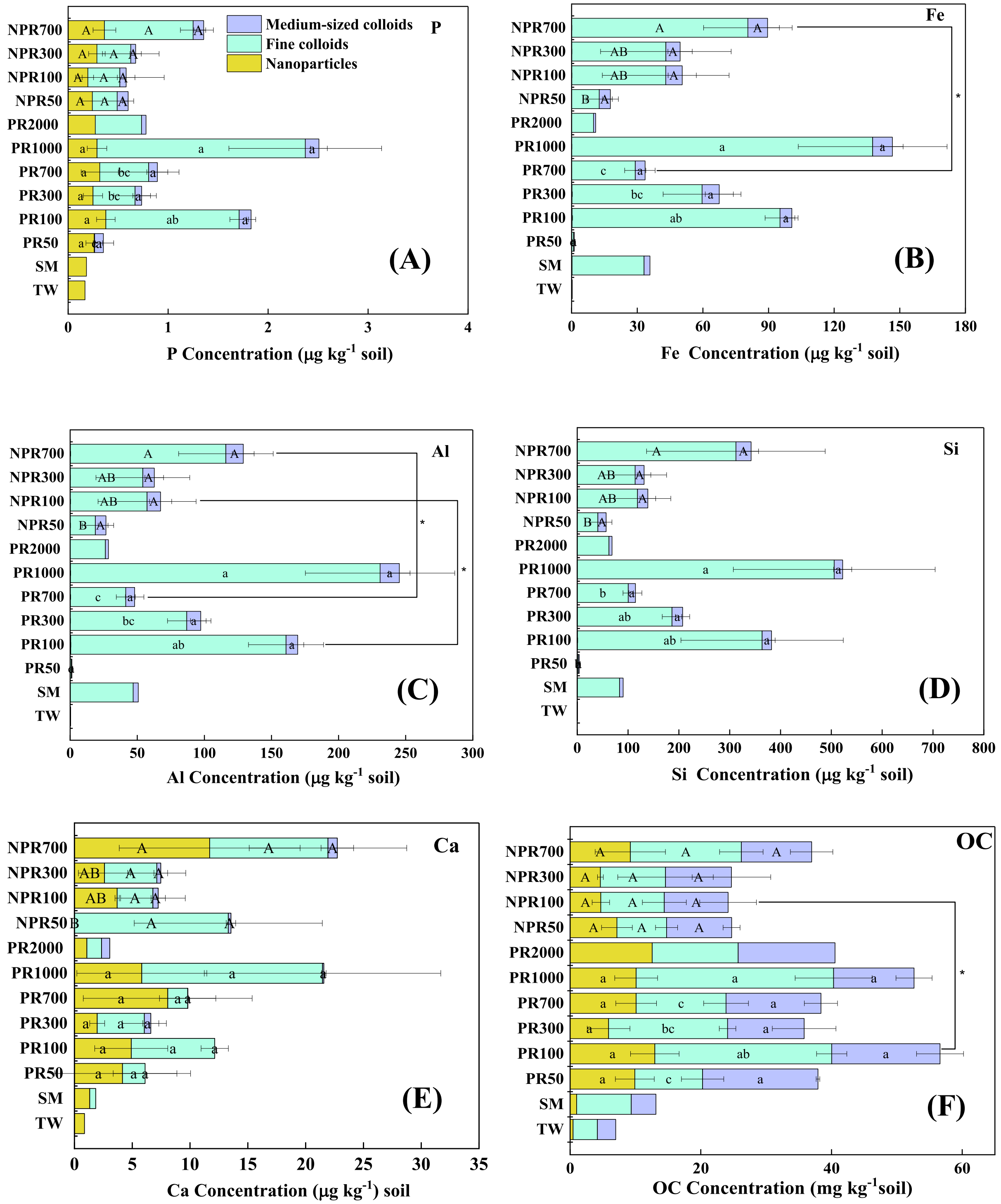

Fig. 3. Elements and OC concentrations of nanoparticles (NP, 0–20 nm), fine colloids (FC, 20–225 nm), and medium-sized colloids (MC, 225–450 nm) from soil water extracts <450 nm measured by AF4-OCD-ICP-MS. Different lowercase and uppercase letters indicate significant differences ($P < 0.05$) of soil fractions among the chronosequence for paddy and non-paddy managements, respectively. Significant difference of paddy and non-paddy soils are shown with star.

elevated Ca contents in the fine colloids of the 50 year-old non-paddy fields (Fig. 3) additionally confirmed that apatite-bound P likely contributed in larger portions to overall colloidal P from these fields than from the paddy sites.

The drop of pH values by 2 units from the 50 to the 100 yr old paddy soils facilitated deterioration of Ca-stabilized aggregates, which likely promoted the release of associated fine-colloidal P and shifted colloidal composition from Ca dominated colloids to Fe/Al oxides enriched ones. Furthermore, Said-Pullicino et al. (2021) has found that anoxic conditions enhanced colloid dispersion with a preferential release of the small colloid fractions (<30 nm and 30–240 nm fractions). The reductive dissolution of Fe oxides under anoxic conditions can also release associated organic C into solution, with large contributions of colloidal C. Our soils became temporary anoxic at least during the flooding season. In line with Said-Pullicino et al. (2021), our results also pointed to a significant increase of fine colloidal C (i.e. 20–225 nm) from the 50 to the 100 yr old paddy soils (Fig. 3F). In line with that study we thus attribute reduction of Fe to be responsible for the dispersion of larger

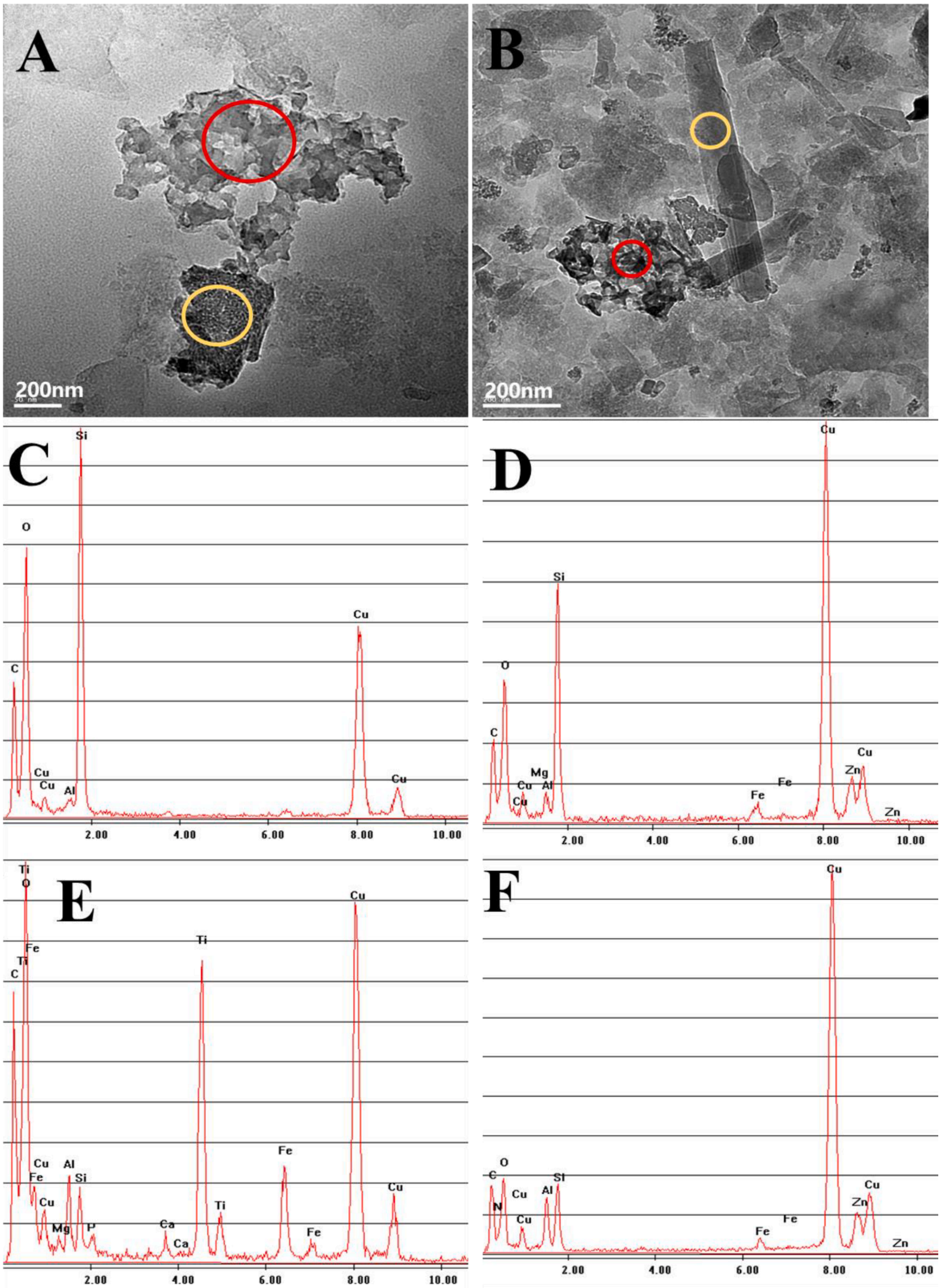

Fig. 4. Transmission electron microscopy (TEM) and energy dispersive X-ray spectroscopy (EDX) spectra of fine colloids from PR300 (A, C, E) and NPR 300 (B, D, F). The red circle in (A) and (B) belongs to the EDX spectrum (C) and (D) respectively, the yellow circle in (A) and (B) belongs to the EDX spectrum (E) and (F). The large Cu peak present in the EDX spectra is caused by the sample holder containing Cu. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

colloids with increasing Fe, Al, and Si contents. Yet, the drop of pH in our soils discounts the possibility that smallest colloids dispersed under pH rise as observed by, e.g., Buettner et al. (2014) and Said-Pullicino et al. (2021). After initial formation of FC-P during the first 50–100 yr of management, colloidal P compositions are likely less prone to future pH changes, and also redox cycles do only affect a minor part of these colloids but not the majority which are more related to clay-bound ones. In this temporal context, the long-term dynamics of colloidal P are likely becoming ever more closely coupled to the fate of fine colloidal clays.

4. Conclusion

The paddy-rice and non-paddy soil colloids (3 kDa-450 nm) consisted of three main size fractions: 3 kDa-20 nm, 20–225 nm, and 225–450 nm. The nanoparticulate fraction (i.e., 3 kDa-to-20 nm) in tidal wetland and salt marsh was dominated by Ca and P indicative of calcium phosphate precipitates. However, nanoparticulate P under arable management was associated with OC and Ca. The fine colloidal fraction (i.e., 20–225 nm) was dominated by P in association with clay minerals, metal oxides, organic matter, and/or apatite. This study suggested that long-term cropping in the studied soils induces a potential for more plant available P by the generation of more fine colloidal clays and associated P, but without being significantly affected by cropping duration or frequency of redox cycles.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.geoderma.2022.116296.

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