



New Perspective into the Impact of Drying Pretreatment on Phosphorus Performance in Sediments

Zhuoman Wu^{1,2} · Yuepeng Yin^{3,4} · Wenxi Pan^{1,2} · Jinyong Tang^{1,2} · Xi Cao^{1,2} · Wen Zhang^{1,2}

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Abstract

Purpose of Review Different drying methods can cause errors in determining the content and evaluating the biological effectiveness of various forms of phosphorus (P) in sediment. The drying pretreatment effectively promotes the conversion of amorphous iron and aluminium oxides to their crystalline form, which ultimately impacts the adsorption capabilities of sediment. However, limited research has been conducted in this field previously. Sediments from the Weiyuan River, Baoenqiao Reservoir, and Honghu Lake in China were pretreated using freeze-drying, air-drying, and oven-drying methods. The effects of pretreatment methods on P were evaluated through P fractionation and isothermal adsorption experiments. The study also investigated the proportion and amount of amorphous iron and aluminium (Fe_{ox} and Al_{ox}) transformed into crystalline forms.

Recent Findings The results revealed that drying pretreatment markedly increased the potentially bioavailable P(BAP) (6.73%). This increase can be attributed to the rise in loosely-bound P (48.30%) and P bound to metal oxides (9.51%), which are predominant contributors to BAP. Furthermore, sediment adsorption performance significantly decreases after drying pretreatment. This is due to the reduced content of Fe_{ox} (64.02%) and Al_{ox} (36.61%), which exhibit higher P adsorption capacity. Additionally, drying led to a significant reduction in SP_{max} (25.09%) and PSI (28.20%), along with an increase in EPC₀ (24.96%) and DPS (6.83%).

Summary Different drying treatments affected the P forms and sorption properties of the sediment to varying degrees, with the overall effect being oven-drying > air-drying > freeze-drying > fresh samples. Consequently, when fresh sediment is not available for laboratory analysis, freeze-drying may be a more realistic method for characterizing P properties.

Keywords Sediment · Drying pretreatment · Phosphorus fractions · Adsorption properties

Introduction

The eutrophication of river and lake reservoir waters has emerged as a significant global ecological and environmental challenge [1]. Phosphorus (P) is one of the main factors that cause eutrophication in water bodies and is a key nutrient for most aquatic and terrestrial ecosystems, including algal blooms [2, 3]. In the water environment, the process of P adsorption by sediments is an essential part of the natural P cycle and constitutes a major source of the internal P release [4, 5]. With reduced external loads of P, the eutrophication of water bodies does not slow down and the recovery period may continue for decades [6, 7]. Consequently, it is to investigate the morphological changes and sorption behaviour of P within sediments and emphasise the influence of sediment endogenous loading on water quality [8].

It is recognised from existing studies that sediment total P (TP) concentrations have always been an indicator

Zhuoman Wu and Yuepeng Yin contributed equally to this article.

✉ Wen Zhang
zhangwen2014@cdut.edu.cn

- ¹ State Key Laboratory of Geohazard Prevention and Geoenvironment Protection, Chengdu University of Technology, Chengdu 610059, China
- ² State Environmental Protection Key Laboratory of Synergetic Control and Joint Remediation for Soil & Water Pollution, College of Ecology and Environment, Chengdu University of Technology, Chengdu 610059, China
- ³ CAS Key Laboratory of Soil Environment and Pollution Remediation, Institute of Soil Science, Chinese Academy of Sciences, Nanjing, China
- ⁴ University of Chinese Academy of Sciences, Beijing 100049, China

of managed P [9], but changes in bioavailable P (BAP) in TP can affect the assessment of water quality [10]. The various fractions of P in sediment include loosely-bound P (Loosely-P), ferrous iron P (Fe(II)-P), P bound to metal oxides (CDB-P), organic P (OP), and calcium-carbonate-precipitated P (Ca-P). Among them, there are bioavailable P (BAP), including Loosely-P, Fe(II)-P, CDB-P, and OP [11••], and it is closely related to the form of P that is adsorbed to the sediment [12]. Therefore, accurately assessing the potential bioavailability of P is a fundamental basis for evaluating water quality in aquatic systems [10, 13, 14]. Furthermore, investigating adsorption properties represents a pivotal facet of sedimentary P within aquatic environments [15]. Among the main indicators of focus are the P sorption maximum (SP_{max}), equilibrium P concentration (EPC_0), P sorption index (PSI), and degree of P saturation (DPS). They can determine and predict the ability of sediment to adsorb or release P, as well as the ability of sediment to retain P [16–20]. Therefore, it is necessary to explore the distribution characteristics of P species and adsorption characteristics in sediments to accurately assess the potential availability of P and the risk of P release to water bodies [21–23].

Regrettably, there is currently no unified standard for the selection of sediment drying pretreatment. In sediment research, fresh sediment is the primary choice for studying sediment P. However, for the sake of convenience, it is common practice to dry the sediment before analysis or experimental treatment. Common drying pretreatment includes air-drying, freeze-drying, and oven-drying, which are used to preserve samples for subsequent experimental analysis. For instance, in the study of P bioavailability in sediment from the Yangtze River Basin, researchers have employed various drying pretreatment, including air-drying [24, 25] and freeze-drying [26, 27], due to the challenges of preserving a large number of samples. Similar situations also occurred in other regions [28, 29]. However, prolonged exposure to natural air drying can lead to changes in the physicochemical properties of sediment, such as an increase in oxidation-reduction potential (Eh) and a consequent reduction in ferrous phosphate content [30, 31]. Moreover, some studies have observed shifts in the particle size of air-dried sediments towards larger particles [32, 33]. This phenomenon could result in a decrease in the sediment's affinity for phosphate, as the majority of P tends to accumulate in the finer fractions [34]. Conversely, freeze-drying better preserves the original sediment environment and is the drying method closest to natural conditions [35]. It is worth noting that the changes in the amorphous structure within the sediment after drying have not received much attention [36–38]. Amorphous iron and aluminium play a crucial role in the adsorption and immobilization of P in sediments, aiding in reducing the concentration and solubility of P in water bodies and thus contributing to the mitigation of eutrophication issues [39]. Amorphous iron and aluminium (Fe_{ox} and Al_{ox}) are typically influenced by factors such as pH,

temperature, and water chemical conditions [40•, 41]. Elevated temperatures and dehydration promote the transformation of Fe_{ox} and Al_{ox} into crystalline iron and aluminium oxides, thus diminishing the adsorption capabilities of the sediment [42]. Therefore, the use of different drying pretreatments may introduce biases in research results, leading to experimental failures or alterations in P form and adsorption properties. Although there are also a few people who prefer to use freeze-drying to process experimental samples, it is worth noting that they rely solely on experience to determine the process without considering the changes in the crystal structure within the sediment [43–45]. Our research has indeed demonstrated that the increase in crystallinity of iron and aluminium during the drying process leads to a decrease in sediment adsorption capacity, a point that is rarely mentioned in other studies.

To fill this knowledge gap, sediment samples were collected from the Weiyuan River, Baoenqiao Reservoir, and Honghu Lake to explore the transformation between Fe_{ox}/Al_{ox} and crystalline structures through drying pretreatment experiments. Based on this, sediment P fractions and isothermal adsorption experiments to explain the changes in sediment adsorption performance further. The degree of influence of different drying pretreatment on P fractions and adsorption properties was also clarified to screen out relatively reasonable pretreatment. Ultimately, this study will standardise the pretreatment method for sediment P and give researchers a reference solution for proper drying pretreatment.

Materials and Methods

Study Area

The sediment and overlying water samples for this experiment were collected from the Weiyuan River of Tuojiang Catchment, the Baoenqiao Reservoir of the Dadu Catchment, and the Honghu Lake of the Yangtze Catchment (Fig. 1). The Weiyuan River basin covers an area of 3490 km², with a mainstream length of 197 km and a significant tributary, Xushui River, which extends 129.9 km. The average water retention time is 8.87 days, resulting in a dispersion of exogenous input of P is dispersed to some extent due to the flow of the water [46]. The dam site is located in Tianci Village, Fuxi Township, and it is a reservoir with a large water storage capacity that contributes to year-round farming activities. This reservoir is comprised of a dam, spillway, and water release infrastructure, primarily serving the purpose of agricultural irrigation. Honghu Lake is the largest freshwater lake in Hubei Province, with a long hydraulic residence time and poor mobility. Located within the middle and lower reaches of the Yangtze River, this region is among the most severely affected worldwide by lake eutrophication. Over time, unsustainable development and use of the

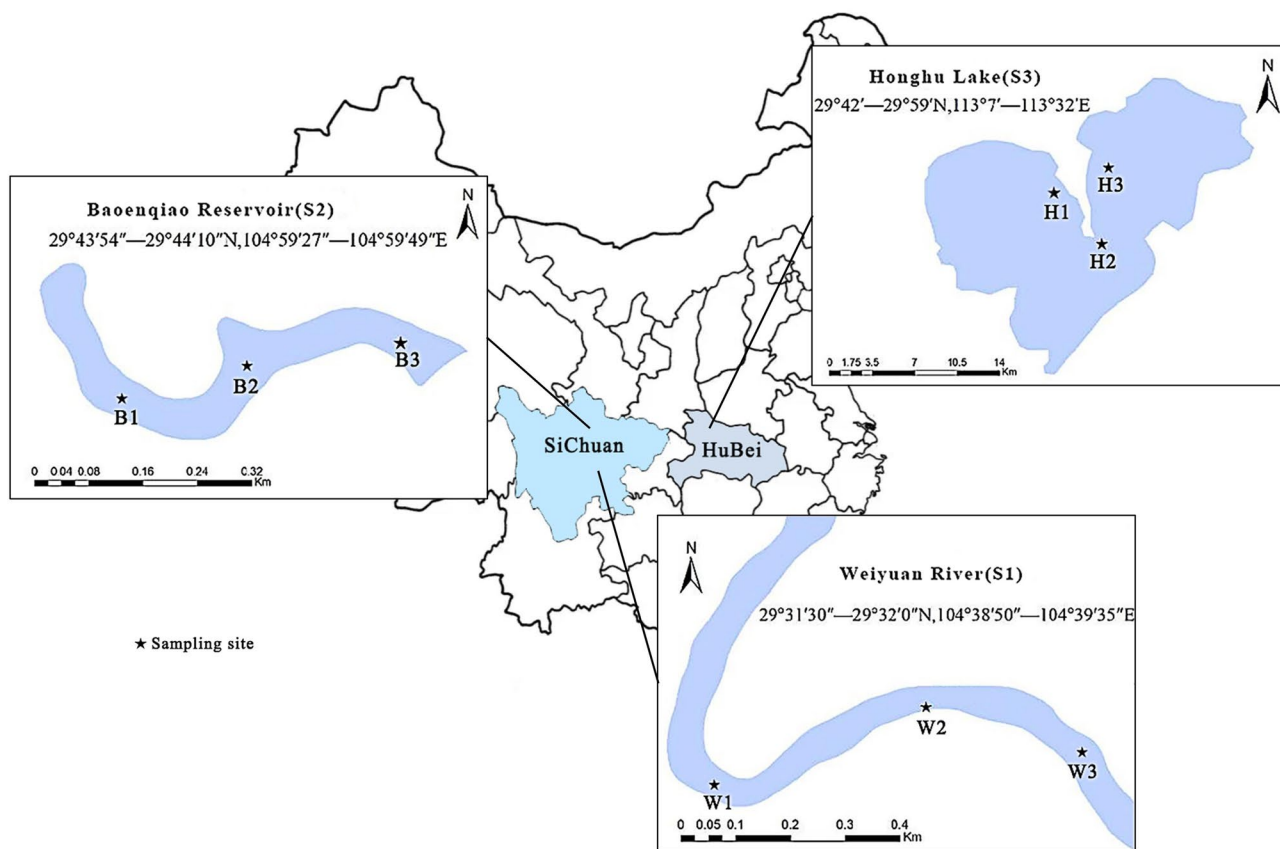


Fig. 1 Study area and sampling sites. Each water body has three sampling points, namely Weiyuan River (W1, W2, W3), Baoenqiao Reservoir (B1, B2, B3), and Honghu Lake (H1, H2, H3)

lake, including field enclosures, intensive fishing, excessive farming, and the inflow of upstream water pollution, have culminated in significant eutrophication within Honghu Lake [47]. All three water bodies are typical of watersheds that are subject to high levels of P pollution of sediments due to P inputs.

Sample Collection

Surface sediments were collected using a Peterson sampler. To ensure the representativeness of the sediments, three sampling points were selected for each water body, and the three samples were mixed. The mixed sediments from the Weiyuan River, Baoenqiao Reservoir, and Honghu Lake were named S1, S2, and S3, respectively, and they were mixed and sealed in air-excluded Polyethylene bags, transported back to the laboratory to be stored in the refrigerator at 4 °C, and protected from the light. The sediment was prepared in duplicate: one for the determination of the physicochemical properties, such as the particle size and organic matter (OM) of each form of sediment, and one for freeze-drying (−40 °C, 18 h), natural air-drying (25 °C, 48 h), and oven-drying (40 °C, 24 h). Then, the samples were ground in

an agate mortar, sieved (0.149 mm), and stored in a sealed container for subsequent experimental analysis.

Analytical Methods

Physicochemical Properties of Sediments

The pH and Eh of sediments at each sampling site were monitored by a portable parameter water quality instrument. After drying and filtering the sediment samples, 1 g of each fresh sediment sample was calcined in a muffle furnace. Amorphous Al and Fe (Fe_{ox} , Al_{ox}) were extracted using oxalic acid (0.1 mol/L) and ammonium oxalate (0.175 mol/L) under complete darkness for 2 h [48]. Metal-Ca, Fe, Mn, Mg, and Al were determined using inductively coupled plasma atomic absorption spectrometry (ICP-AES) after digesting 0.5 g of sediment sample with nitric acid-hydrofluoric acid (HJ/T 166-2004) (SEPA, 2004). OM was measured as a loss on ignition (550 °C for 4 h). Through the analysis of LS 230 laser diffraction particle analyzer (Microtrac S3500), the sediment particle size is divided into three categories: (1) clay, < 4 μm; (2) silt, 4–63 μm; (3) sand, > 63 μm [49].

P Fractionation Schemes

The collected sediment samples were pretreated by freeze-drying (−20 °C), natural air-drying (25 °C), and oven-drying (40 °C), and then they were ground in an agate mortar, sieved (0.149 mm), and used for the continuous extraction of each sediment P form and isothermal sorption experiments. The sediment P fractions were extracted using a modified SEDEX method for graded extraction, which classified the sediment into five fractions of P: Loosely-P, Fe(II)-P, CDB-P, Ca-P, and OP [11••], as shown in Fig. S1. Notably, this method employed 0.2% 2,2'-bipyridine to separately extract the Fe(II)-P fraction from anaerobic sediments and Fe(II)-P as a truly bioavailable form of P allows for a more accurate assessment of BAP. The sediment total P (TP) was determined using the alkali fusion-mo-sb anti spectro-photometric method (HJ632-2011).

Adsorption Experiment of Phosphorus in Sediments

The dry sediment samples of 1 g into a 50-ml screw cap centrifuge tubes, and add 20 ml phosphate standard solutions (anhydrous KH₂PO₄) with various concentrations ranging from 0 to 75 mg/l (0, 0.05, 1.00, 5.00, 10.0, 15.00, 25.00, 50.00, and 75.00 mg/l P) [50]. To inhibit bacterial activity, two drops of chloroform were introduced. Subsequently, the centrifuge tubes were sealed and incubated at 25 ± 1 °C on an orbital shaker at 200 rpm. After 24 h of equilibration, the solutions were subjected to centrifugation at 4200 rpm for 15 min and then filtered through sediment samples using a 0.45-µm GF/C filter membrane. The filtrate was collected for phosphate analyses.

When the net P absorption is zero, the equilibrium P concentration (EPC) is determined by the intersection of the P adsorption isotherm and the x-axis. The sorbed P (SP) is calculated from the difference between the measured filtrate EPC and the initial P concentration. The sorption maximum (SP_{max}) is calculated by plotting the reciprocal of EPC against the ratio EPC/SP and determining the slope. Isothermal sorption curves were fitted using the Langmuir equation [51]:

$$SP = \frac{SP_{max} \times k \times EPC}{1 + k \times EPC} \tag{1}$$

where SP (mg/g) and SP_{max} (mg/g) are the equilibrium amount of P adsorbed to the sediment and the maximum P sorption by the sediment, EPC (mg/L) is the equilibrium P concentration, and k is the Langmuir equilibrium constant.

The sediment PSI (L/g) was calculated using the following equation [20, 52•, 53]:

$$PSI = \frac{X}{lgC} \tag{2}$$

the equation calculates the P adsorption index, represented as PSI (L/g), using the P adsorption capacity of 1 g of sediment, denoted as X (mg/g), and the EPC of filtrate after 24 h, represented as C (g/L).

Sediment DPS (%) was determined using the following equation [54]:

$$DPS = \frac{TP_s}{TP_s + SP_{max}} \times 100\% \tag{3}$$

where DPS (%) indicates the degree of P saturation, and TP_s (mg/g) is the TP (mg/g) content of the sediment.

Calculations and Statistics

All the statistical analyses were performed using IBM SPSS Statistics 24.0 software. Significant differences are reported at the *p* < 0.05 level, and these were generated using Origin 8.0. Measurements were conducted in triplicate for each sample, and the arithmetic mean was reported in the results. The Global Positioning System (GPS) was utilized to record sample locations, and ArcGIS (version 10.6) software was used for geostatistical analysis and mapping.

Results

Sediment Physicochemical Characteristics

There was little difference in the sediment pH between the three sampling sites (pH = 7.2, 7.3, and 7.6 in S1, S2, and S3, respectively) and the Eh was negative (Eh = −191, −122, and −78 in S1, S2, and S3, respectively) (Table 1), indicating that the sediments were under anaerobic environment. The OM content at sampling sites S2 (14.2%) and S3

Table 1 Physicochemical characteristics of sediments

Sampling point	pH	Eh (mV)	OM (%)	Particle size (µm) %			Al (mg/g)	Ca	Fe	Mn	Mg
				<4	4–63	>63					
S1	7.2	−191	6.6	8.6	34.3	57.1	59.6	18.5	24.4	0.4	8.7
S2	7.3	−122	14.2	17.3	43.5	39.2	83.3	11.4	46.2	0.8	17.8
S3	7.6	−78	11.6	21.4	51.3	27.4	75.9	19.7	44.2	0.9	13.7

(11.6%) was significantly higher than that at S1 (6.6%), due to a large amount of OM that was deposited in the sediment as a result of farming activities at S2 (reservoir) and the deposition of OM at S3 due to the input of exogenous OM and over-farming. The sampling site S1 (river) was influenced by the changing hydrodynamic conditions and was therefore not conducive to OM enrichment. The proportion of fine particles in S1 (8.6%) was smaller than those in S2 (17.3%) and S3 (21.4%), probably because the river flow was not conducive to the settling of fine particles. Moreover, Fe and Al were the main metal minerals in the sediments at all three sampling sites.

Effect of the Drying Pretreatment on the P Fractions

Overall, the drying pretreatment had little effect on the TP (Fig. 2A). There was no significant difference in the TP after the three drying pretreatments when compared with that of the fresh sediment. Air-drying may result in higher sediment TP concentrations than freeze-drying due to P release from microbial degradation transformation [36], but this was not found in this study.

The site S2, a reservoir, had higher Loosely-P (147.97 mg/kg) and Fe(II)-P (120.35 mg/kg) than those of S1 (river) (39.11 mg/kg, 9.06 mg/kg) and S3 (lake) (6.80 mg/kg, 16.62 mg/kg), indicating that reservoir is at a greater risk of P release than rivers and lakes (Fig. 2B, C). In addition, site S3

(320.94 mg/kg) had slightly more CDB-P than S1 (339.58 mg/kg) and S2 (560.14 mg/kg), while S2 (347.56 mg/kg) had the highest Ca-P content. The sediment Loosely-P content increased considerably after drying when compared with that of the fresh samples (Fig. 2B). Air-drying and oven-drying dramatically increased the Loosely-P by 13.82 to 61.70% and 17.29 to 90.41%, respectively, whereas freeze-drying only increased it by 1.54 to 25.43%. The Fe(II)-P of the sediment was dramatically lowered by the three drying pretreatments (Fig. 2C), with the greatest changes in Fe(II)-P after oven-drying, which decreased by 89.62%, 59.55%, and 85.17% S1, S2, and S3, respectively, followed by air-drying, which decreased by 72.63%, 56.43%, and 81.92%, and freeze-drying, which decreased by only 30.14%, 17.66%, and 58.60%. In contrast, the CDB-P content increased after drying (Fig. 2D). The CDB-P increased by 7.02%, 4.01%, and 2.06% after freeze-drying for S1, S2, and S3, respectively; by 13.45%, 9.93%, and 10.85% after air-drying; and by 15.20%, 11.36%, and 11.74% after oven-drying. Additionally, drying caused a dramatic decrease in the sediment Ca-P when contrasted with that of the fresh sediment (Fig. 2E). There were no significant changes between air-drying and oven-drying, but the effects were much greater than those of freeze-drying. The drying pretreatment also significantly enhanced the OP of both the S1 and S3 sediments, whereas the freeze-drying treatment had no significant influence on the OP of the S2 sediments (Fig. 2F). In general, the sediment P was more notably altered after the air-drying

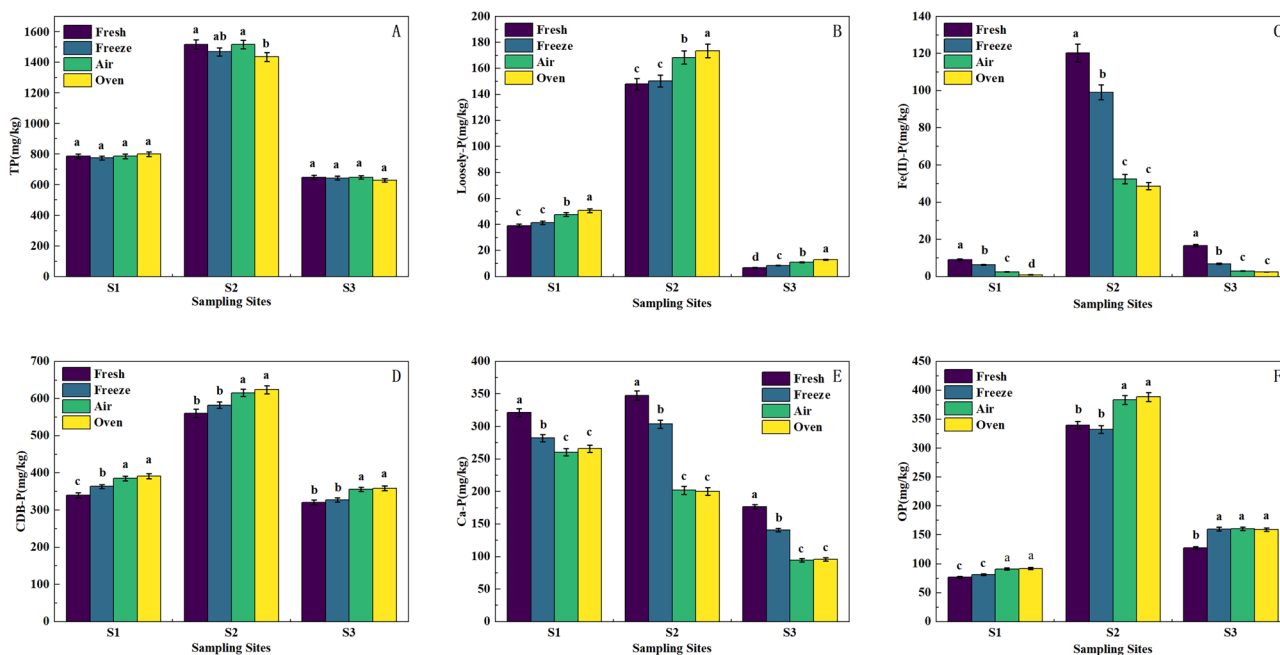


Fig. 2 Contents of the total phosphorus (TP) (A), loosely-bound phosphorus (Loosely-P) (B), ferrous iron phosphorus (Fe(II)-P) (C), phosphorus bound to metal oxides CDB-P (D), calcium-carbonate-precipitated phosphorus (Ca-P) (E), and organic phosphorus (OP) (F)

with different drying pretreatment. Significant differences in the different fractions of P ($p < 0.05$) are represented by different lowercase letters

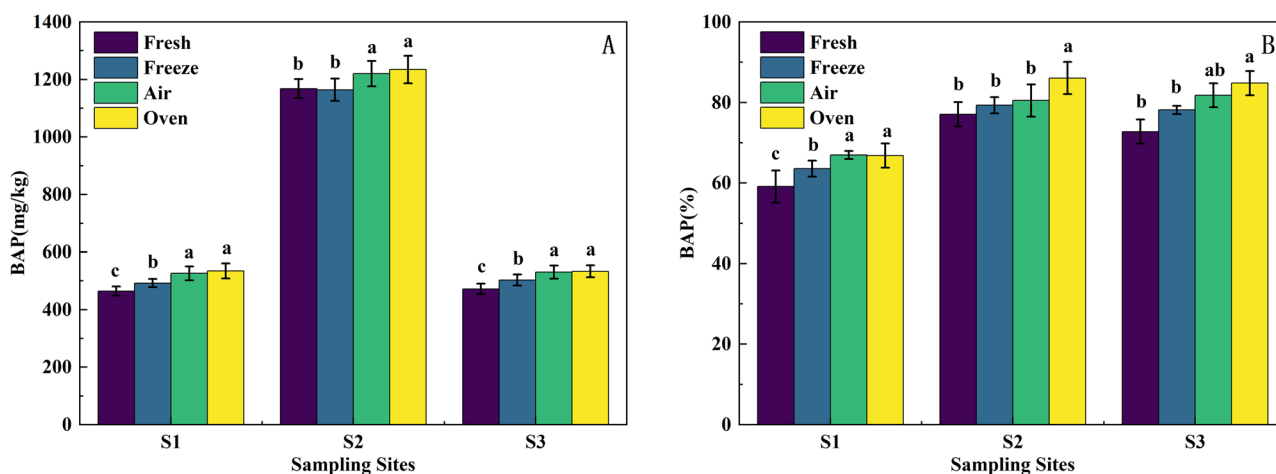


Fig. 3 Bioavailable phosphorus (BAP) concentration (A) and percentage of bioavailable phosphorus (BAP) to total phosphorus (TP) (B) with different drying pretreatment. Significant differences in BAP ($p < 0.05$) are represented by different lowercase letters

and oven-drying pretreatments than after freeze-drying. The degree of impact is Fe(II)-P > Ca-P > OP > CDB-P.

Significantly, when compared with the fresh samples, freeze-drying (2.60%), air-drying (8.18%), and oven-drying (9.41%) all increased the BAP at each sampling site (Fig. 3A). After the pretreatment, the proportion of BAP to TP was higher in all the samples than that in the fresh samples, but freeze-drying had the least effect on the proportion of BAP to TP (5.78%) (Fig. 3B). Additionally, the proportion of BAP in S1, S2, and S3 was 59.11 to 66.97%, 77.07 to 86.05%, and 72.76 to 84.80%, respectively, accounting for most of the sediment.

Effect of the Drying Pretreatment on the P Adsorption

Equilibrium P Concentration and P Sorption Maximum

All the sampling sites' sediments showed an increasing trend in the EPC_0 after the drying pretreatment (Table 2). In comparison with the fresh sediment, the EPC_0 increased by 3.26, 1.08, and 4.4 times at the sampling sites S1, S2, and S3, respectively, after oven-drying and by 3.31, 0.68, and 3.84 times after air-drying, while it only increased by 1.34, 0.28, and 1.53 times after freeze-drying. The SP_{max} of all the sediments declined dramatically after the different drying pretreatments (Table 2). Among these, oven-drying resulted in a decrease in the SP_{max} by 22.24%, 41.74%, and 47.30% in S1, S2, and S3, respectively; the air-drying resulted in a decrease of 29.81%, 27.73%, and 28.46%; and the freeze-drying resulted in a decrease of only 11.93%, 10.88%, and 4.56%. This shows that the amount of change in the EPC_0 and SP_{max} in the sediment after freeze-drying was much less than that of the sediment after oven-drying and air-drying.

In addition, we measured the amounts of Fe_{ox} and Al_{ox} in the sediments after the different drying pretreatments (Fig. 4). Drying reduced the Fe_{ox} and Al_{ox} contents, especially air-drying and oven-drying, with a maximum reduction in Fe_{ox} of 76.10%, 49.67%, and 66.29% for S1, S2, and S3, respectively, and a maximum reduction in Al_{ox} of 33.10%, 21.86%, and 54.88%. Moreover, freeze-drying had less of an influence on Fe_{ox} and Al_{ox} , while significant changes in Fe_{ox} and Al_{ox} occurred after oven-drying and air-drying. An average of 0.43% of the amorphous iron was converted to crystalline iron in S1, S2, and S3 after freeze-drying when contrasted with 2.09% and 2.05% after air-drying and oven-drying, respectively (Table S1). This study provides new insights into changes in sediment adsorption properties following drying pretreatment by examining changes in the crystal structure within the sediment.

Table 2 Equilibrium phosphorus concentration (EPC_0) and phosphorus sorption maximum (SP_{max}) of sediments with different drying pretreatment

	Pretreatment	EPC_0 (mg/L)	SP_{max} (mg/kg)
S1	Fresh	0.008	791.056
	Freeze	0.018	696.709
	Air	0.034	555.217
	Oven	0.034	615.155
S2	Fresh	0.132	741.072
	Freeze	0.170	660.478
	Air	0.222	535.551
	Oven	0.275	431.735
S3	Fresh	0.006	1022.378
	Freeze	0.015	975.768
	Air	0.029	731.365
	Oven	0.032	538.782

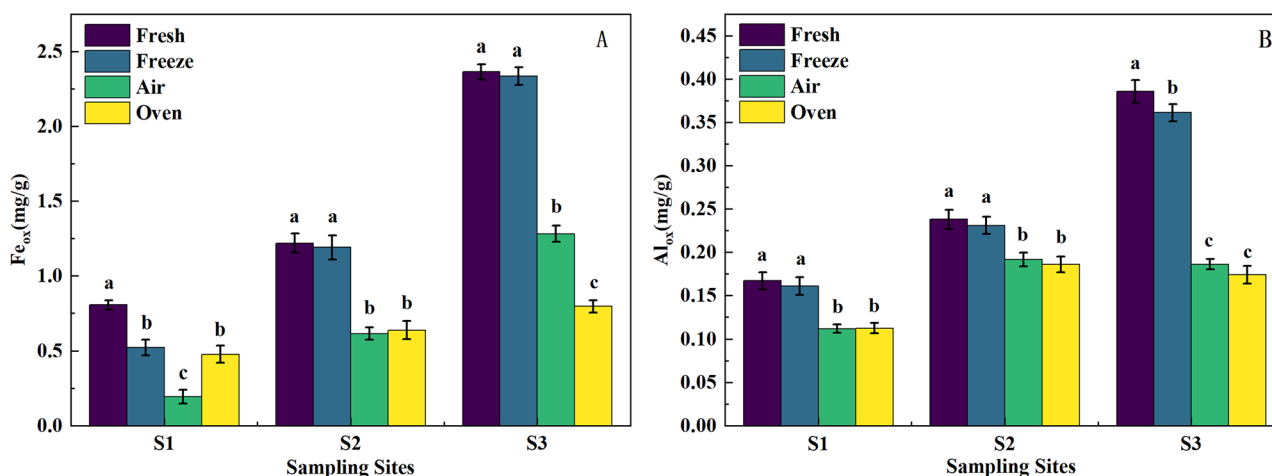


Fig. 4 Iron (hydro)oxides (Fe_{ox}) (A) and aluminium (hydro)oxides (Al_{ox}) (B) of sediments with different drying pretreatment. Significant differences ($p < 0.05$) are represented by different lowercase letters

Degree of P Saturation and P Sorption Index

The effects of different drying pretreatments on the sediment DPS and PSI are shown in Table S2. All the sediment DPS values increased to varying degrees after the different drying pretreatments, and there were significant differences in the effect of the pretreatment on the sediment DPS. In comparison with the fresh sediments, the DPS increased by 6.72 to 15.04% in S1, S2, and S3 after oven-drying, respectively, by 6.73 to 8.77% after air-drying, and by 0.93 to 2.81% after freeze-drying. In contrast, all the sediment PSI values decreased after drying. In contrast with the fresh sediments, the sediment PSI decreased by 36.76 to 47.36% after oven-drying, 29.39 to 32.47% after air-drying, and 8.91 to 14.30% after freeze-drying. The results of the three kinds of drying pretreatments on the DPS and PSI of the sediment were in the order of oven-drying > air-drying > freeze-drying > fresh samples. The increase in the DPS and decrease in the PSI after the drying pretreatment of the sediment both indicate that drying reduced the P adsorption capacity of the sediment.

Discussion

Different pretreatment methods will significantly affect changes in sediment P components and even adsorption properties [37, 38, 55]. However, some researchers may not be aware of this extensive impact [25, 56]. This results in a lack of understanding of why drying pretreatment affects sediment P properties. Unreasonable pretreatment methods may produce widely different experimental results, thereby misjudging the risk of sediment P release [57]. It is worth noting that this study demonstrated the irrationality of these methods through experimental results, theoretically

elaborated on the impact of drying on the P properties of sediments, focusing on the impact of drying on the P properties of sediments, to provide long-term storage experiments. Samples provide theoretical guidance.

Relationships of Drying and P Fractionation

Drying is already a very common pretreatment method, yet the diverse effects of various drying methods on experimental results have been insufficiently explored [58]. Our study reveals that drying can have a significant effect on P forms and sediment physicochemical properties, thereby affecting the P adsorption properties of the sediment. From the P forms analysed above, it is evident that BAP is active in sediment P fractionation and is an indicator of the risk of P release. The BAP is part of the biogeochemical cycle of P and maintains the self-regulation of P [59, 60], and it is generally a relatively abundant form of P in sediments, accounting for 80–98% of the TP [12]. Notably, three drying pretreatments all increased the BAP at each sampling site indicating, a positive effect of drying on the potential BAP concentrations in the sediment. Interestingly, the most pronounced changes were still seen with oven-drying, which may have resulted in a high BAP due to the conversion of some OP into bioavailable IP [61]. Drying increased the proportion of BAP (7.86 to 12.04%), and there are several possible explanations for this result. On the one hand, drying kills the microbial cells through the Birch pulse effect, causing them to lyse and release P [62]; on the other hand, it causes a reduction in the P affinity of the sediment [63]. This may be due to changes in the sediment binding sites as a result of oxidation or changes in the drying of the sediment [62]. These findings underscore the significant impact of drying pretreatment on BAP levels and, thus, the risk of P

release from the sediment can be overestimated. This may lead to sediments that are at the borderline of risk management being at risk and thus misleading river managers.

Additionally, our study also found that dry pretreatment altered sediment P forms, leading researchers to inaccurate assessments of the biogeochemical P cycle and its potential contribution to eutrophication. Loosely-P is highly susceptible to environmental factors (e.g. pH, Eh, temperature, and human disturbance) [64]. The oven-drying of the sediment at higher temperatures accelerated the tight adsorption of Loosely-P onto the sediment clay minerals, resulting in higher Loosely-P in the oven-dried samples (79.06 mg/kg) than that in the other two pretreatment methods (Fig. 5) [36].

The potential biological effectiveness of Fe(II)-P and CDB-P depends mainly on the redox environment of the sediment [11••, 65], e.g. in fluvial environments where the redox conditions of the sediment change frequently, when the redox potential of the sediment decreases, Fe(II)-P and CDB-P intermittently participate in redox reactions and are released from the sediment into the upper water column to be bioavailable [60]. The sediment Fe(II)-P content decreased significantly after drying, while the CDB-P content increased after drying in both cases (Fig. 5). This is due to the rapid oxidation of Fe(II) to Fe(III) in the sediment during drying, resulting in a decrease in the amount of Fe²⁺ bound phosphate and consequently altering the fractionation of the deposited P, which also increased the CDB-P. The other reason for elevated CDB-P levels in the sediments is due to the vulnerability of the anoxic sediments to the strong

influence of pyrite oxidation, which generates sulphate and iron hydroxide. The resulting acid dissolution affects apatite, releasing phosphate that subsequently binds to newly formed hydroxylated iron oxide [66]. Thus, pyrite oxidation facilitates the transformation of authigenic Ca-P into Fe-P [11••, 38, 66], which leads to a reduction in sediment Ca-P (Fig. 5).

Relationships of Drying and Adsorption Properties

In this study, the P sorption capacity of the sediment after the drying pretreatment in this study was significantly lower than that of the fresh sediment, with the degree of the effect being in the order of oven-drying > air-drying > freeze-drying > fresh samples (Fig. S2). Similarly, a study of the effect of drying on P uptake in subtropical river sediments revealed that elevated EPC₀ due to drying was found to indicate a substantial increase in the likelihood of P release from the sediment during the next wetting event [67]. Drying caused an increase in EPC₀ (0.02~0.03 mg/L) and several factors could explain this observation. Firstly, the mineralisation of OM during drying, lysis of cells during extreme drying, and microbial damage may also lead to the release of P on rewetting, thereby increasing the EPC₀ [68, 69]. Moreover, rewetting of the sediment after drying also affected the P release process from the sediment by altering the redox potential, pH, and microbial biomass, resulting in reduced sediment P sorption. This included (1) a reduction in the redox potential of the sediment, prompting a reduction reaction with Fe(OH)₃ and an increase in Al(OH)₃ solubility, resulting in

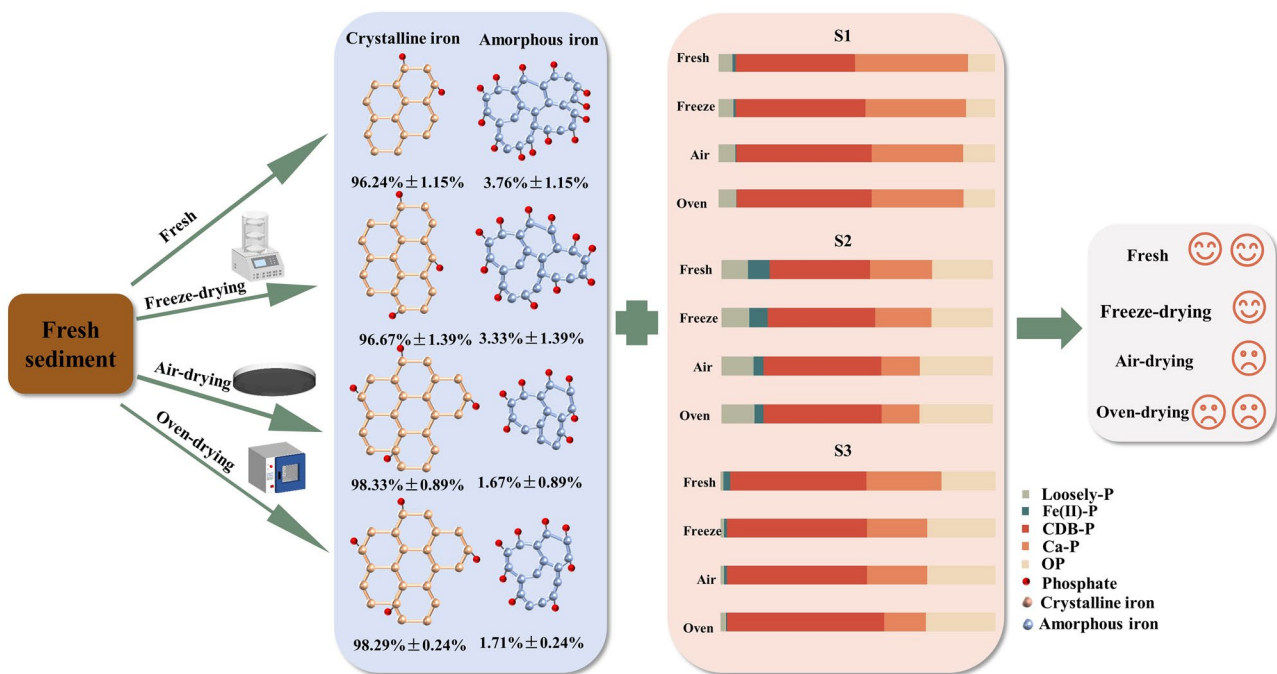


Fig. 5 Schematic representation of the changes in the sediment phosphorus fractions and sorption properties with different drying pretreatment

Fe/Al-P release and (2) air-drying killed approximately 76% of the microbial biomass [70], which reduced the microbial population and re-desorption of microbially absorbed P into the overlying water. Therefore, future research will further observe and explore the differences between different drying pretreatments based on microbial experiments.

As shown in Fig. 5, the percentage of amorphous iron in the sediment decreased after drying and the adsorption capacity for P was reduced. Additionally, the effect of freeze-drying was significantly less than that of air-drying and oven-drying (Fig. 5). A study involving sediments from seven shallow lakes in Western Australia, subjected to air-drying conditions, revealed that 72% of the observed variability in P sorption capacity could be accounted for by changes in iron crystallinity after drying [32]. Thus, the reduced affinity of the dried sediment for P may be a result of the significant increase in the concentration of unstable iron from dry to wet sediment. Unstable iron primarily exists in an amorphous form, possesses high reactivity, and has a high specific surface area. The ageing process of these amorphous iron phases is accompanied by a reduction in P affinity and, consequently, a decrease in the P adsorption capacity of the sediments [62]. Drying accelerates the transformation of Fe_{ox}/Al_{ox} to a more crystalline structure, referred to as mineral ageing. This process entails the loss of potential P sorption sites, which results in a reduction in the sorption capacity [32, 71]. Analogous to the present study, both air-dried and freeze-dried sediments in other studies were able to oxidise poorly crystalline iron (as measured by oxalic acid extracts). This oxidation process could shift the iron species from more amorphous, highly P-reactive iron (oxyhydrogen) oxides to more crystalline, less P-reactive iron oxides [72]. The increase in the DPS in the dry pretreated sediment in this study is also related to the decrease in the Fe_{ox} content, which contains numerous sorption sites, whereas a higher PSI indicates a higher sorption capacity of the sediments for P, and the decrease in the PSI after drying in the pretreated sediment in this study indicates that drying reduced the sorption capacity of the sediments for P [19, 73]. This principle aligns with the observed effects of drying on SPmax and DPS in the sediments. From an economic evaluation perspective, fresh samples are the most cost-effective and time-efficient option, followed by air-drying. On the other hand, freeze-drying and oven-drying methods require a substantial amount of energy, making them more costly. It is also worth noting that while freeze-drying may have a higher upfront cost, it may offer better long-term preservation and maintain sample integrity for certain analytical applications. Nonetheless, it is essential to acknowledge the current research limitations, including the absence of a standardized method for characterizing changes in the internal structure of sediment before and after drying.

Conclusions

Our study explores the effect of different drying pretreatments on P fractions and the risk of P release under laboratory conditions, particularly studies comparing freeze-drying, air-drying, and oven-drying with fresh samples. The main findings were as follows:

1. Drying pretreatment resulted in a decrease in Fe(II)-P and Ca-P but an increase in Loosely-P, CDB-P, and OP in sediments, with the effects of oven-drying and air-drying being more significant than freeze-drying. Drying pretreatment increased the BAP to varying degrees, leading to a biased assessment of BAP and, thus, an overestimation of the risk of P release from the sediments. Therefore, to accurately measure the risk of P release from sediments, a freeze intervention treatment is recommended.
2. Drying caused a transition from amorphous (reactive) metal (hydro)oxides to crystalline metal (hydro) oxides, thus, reducing the affinity and adsorption capacity of the sediment for P. Besides, it led to the release of phosphate due to microbial cell cleavage and increased the P concentration in the overlying water. Reducing the adsorption sites of sediment phosphorus inevitably increases the risk of sediment phosphorus release. In a sense, it is similar to exaggerating the content of BAP.
3. Overall, the degree of influence of drying on the sediment P is in the order of oven-drying > air-drying > freeze-drying. Therefore, when fresh sediment cannot be guaranteed as a sample for laboratory analysis, freeze-drying may more realistically characterise the composition of the different fractionations of P in the sediment than oven-drying and air-drying, and it is a more desirable method for sediment drying pretreatment.

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Data Availability No datasets were generated or analysed during the current study.

Compliance with Ethical Standards

Conflict of Interest The authors declare no competing interests.

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