



# Spatial variation of soil phosphorus in the water level fluctuation zone of the Three Gorges Reservoir: Coupling effects of elevation and artificial restoration

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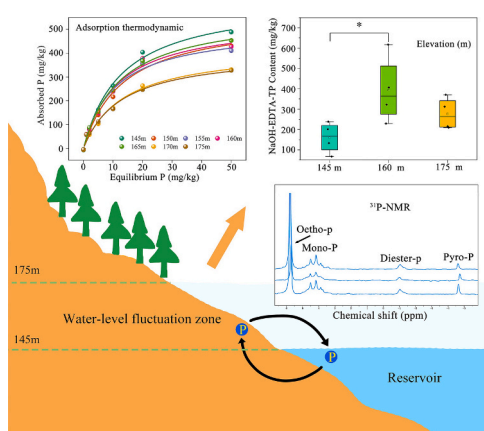
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## HIGHLIGHTS

- 86 soils from 13 locations and 7 elevations were collected from the water level fluctuation zone.
- HCl-P<sub>i</sub> was the largest P pool among all P forms, with mean concentration of 338.5 mg/kg.
- Phosphate monoester and phosphate diester accounted for 1.4 % to 46.2 % of NaOH-EDTA TP.
- The sorption equilibrium concentration of WLFZ soil decreased with increasing elevation.
- Ca and Fe content, particle size, and elevation were key factors affecting soil P fraction.

## GRAPHICAL ABSTRACT



## ARTICLE INFO

Editor: José Virgílio Cruz

## ABSTRACT

The water level fluctuation zone (WLFZ) is a distinctive and important component of the reservoir ecosystem. Due to periodic inundation, the fraction, spatial distribution, and chemical reactivity of soil phosphorus (P) within the WLFZ can potentially impact the loading of P into reservoir waters. However, a detailed study of this

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<https://doi.org/10.1016/j.scitotenv.2023.167000>

Received 11 July 2023; Received in revised form 21 August 2023; Accepted 9 September 2023

Available online 16 September 2023

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**Keywords:**

Three Gorges Reservoir  
 Water level fluctuation zone  
 Phosphorus fraction  
<sup>31</sup>P NMR  
 Adsorption and isotherms

subject is lacking. In this study, the soil P in the WLFZ of the Three Gorges Reservoir, China, was examined using a combination of chemical sequential extraction, <sup>31</sup>P NMR, and adsorption experiments. The results of chemical sequential extraction showed that HCl-P<sub>i</sub> constituted the largest P pool among all P forms, with a mean concentration of 338 mg/kg. The content of HCl-P<sub>i</sub> decreased significantly toward the dam, while the content of Res-P decreased in the opposite direction. The highest contents of most P forms and total P were observed at an elevation of 160 m. <sup>31</sup>P NMR measurements showed that NaOH-EDTA P<sub>i</sub> detectable in WLFZ soils at 145 m, 160 m, and 175 m elevation consisted mainly of orthophosphate and pyrophosphate, while NaOH-EDTA P<sub>o</sub> contained phosphate monoesters and phosphate diesters, accounting for 1.4 % to 46.2 % of NaOH-EDTA TP. Adsorption experiments showed that soil P in the WLFZ was a potential P source for reservoir waters, with chemisorption being the dominant mechanism of P sequestration. The adsorption equilibrium concentration of WLFZ soil was lower at higher elevations (>170 m) compared to lower elevations (<150 m), exhibiting a decrease in the average maximum adsorption from 271 mg/kg to 192 mg/kg. Statistical analysis suggested that Ca and Fe content, particle size, elevation, and artificial restoration were key factors affecting the fraction and content of soil P in the WLFZ. Our findings contribute to an improved understanding of the behavior of soil P in the WLFZ of large reservoirs and its potential contribution to the reservoir waters.

## 1. Introduction

Dams are built for specific purposes globally, such as water supply, flood control, irrigation, navigation, and hydropower, which are the cornerstones of water resource development and management. The reservoir formed by the dam is an important buffer zone for temporary flooding during the flood season (Chen et al., 2022). However, the construction of the dam also significantly alters the hydrological and sedimentary conditions within the reservoir, which otherwise affect the nutrient processes in the river (Tang et al., 2023). As of 2020, the International Commission on Large Dams (ICOLD) reported a global number of approximately 59,000 reservoirs with a total storage capacity of about 7800 km<sup>3</sup>. The total number of reservoirs in China exceeded 98,000 as of 2015, among which 9215 are categorized as large and medium-sized reservoirs (e.g., ~25 % of the world total) (Mulligan et al., 2020).

The water-level fluctuation zone (WLFZ) is a distinct seasonal wetland ecosystem created by the artificial water level management of dammed reservoirs, connecting aquatic and terrestrial ecosystems. It serves as a vulnerable natural alternation zone between wet and dry seasons (Li et al., 2022b; Wang et al., 2022). Importantly, WLFZ functions as a dynamic transition zone between land and aquatic environments, playing a pivotal role in the transport and transformation of matter and energy in ecosystems (Li et al., 2022a; Shen et al., 2022; Su et al., 2017). However, the continuous exposure to counter-seasonal cyclical dry-wet cycles leads to the original vegetation in the WLFZ, causing ecosystem degradation (Huang et al., 2022). Additionally, the periodic water level fluctuations cause landslide deformation, leading to the destruction of natural vegetation on the reservoir banks (Yao et al., 2019). The importance of the WLFZ to the reservoir ecosystem has been noted, and the conservation of the WLFZ has garnered increasing attention. To prevent soil erosion and maintain the stability of the ecosystem, a large amount of vegetation restoration has been carried out in the subsidence zone of the Three Gorges Reservoir (Ye et al., 2015). Natural fallow zone vegetation is dominated by herbaceous plants, and artificial restoration of fallow zone vegetation is based on herbs, trees, and shrubs (Ye et al., 2014).

Phosphorus (P) can be considered as a key limiting element for eutrophication in water. (Chen et al., 2021; Wu et al., 2016). The construction of dams substantially influences the fate and transport of P in the ecosystem, which in turn has an important implication for the P cycle in the river-reservoir ecosystem. It is estimated that in 2000, 12 % of global river phosphorus loads were retained in dam reservoirs, and this will increase to 17 % by 2030 with the construction of >3700 new dams (Maavara et al., 2015). The soils in the WLFZ are prone to release P during the dry-wet cycles, due to the coupled physical, chemical, and biological processes that include destruction of physical structure, desorption of soil matrix, chemical dissolution of metal oxides, and degradation of soil microorganisms (Brödlin et al., 2019; Dinh et al.,

2017; Maranguit et al., 2017). Flooding can cause the pH of WLFZ soil to approach neutral (Sun et al., 2007). The dissolution of Fe and Al oxides due to changes in soil pH can also lead to P release, a process that may be controlled by the P species and organic matter (OM) in the soil. (Gu et al., 2019; Maranguit et al., 2017). Previous studies reported that the spatial distribution of total and active P in the soil of WLFZ increased with decreasing elevation (Ye et al., 2019). However, the spatial distribution and chemical activity of P in WLFZ soils and the associated controlling factors remain elusive. Given that the WLFZ serves as a critical transition zone linking land and water bodies, it is important to understand P characteristics in the WLFZ soils for the safety of aquatic ecosystems.

Three Gorges Reservoir (TGR) is located in the upper reaches of the Yangtze River and is one of the world's largest hydraulic projects (Ren et al., 2020; Yang et al., 2014). TGR has various important benefits in flood control, water storage, power generation, navigation, and agricultural irrigation, but it also creates a series of environmental problems, including eutrophication, ecosystem degradation, biodiversity reduction, and habitat destruction (Tang et al., 2019; Wu et al., 2021). To meet the needs of flood control, navigation, power generation, water storage, and agricultural irrigation, the TGR maintains low water-level operation mode during the rainy season by releasing flood water, while maintaining high water-level operation mode during the dry season by storing water. Due to the unique scheduling mechanism, the TGR forms a WLFZ with a maximum water height of 175 m and a vertical height of 30 m, covering an area of about 350 km<sup>2</sup> at the lowest water level. (Bao et al., 2015; Tang et al., 2018; Wang et al., 2021; Xiao et al., 2022). WLFZ soil samples from different spatial locations and elevations of TGR were collected and analyzed by a suite of complementary approaches, including the chemical sequential extraction method, <sup>31</sup>P NMR, and adsorption isotherms and kinetics. The specific aims of this research were to: (i) investigate the effects of periodic wet and dry alternation on the spatial distribution of different forms of inorganic and organic phosphorus in soil; (ii) investigate the differences in phosphorus species and chemical activity in natural WLFZ soils and artificially restored WLFZ soils; (iii) investigate the phosphorus adsorption characteristics of WLFZ soils, elucidate the potential contribution of WLFZ soil phosphorus to reservoir waters. Our findings could provide a scientific basis for the restoration and management of WLFZ in large reservoirs.

## 2. Materials and methods

### 2.1. Study area

The TGR is 667 km long with a total surface area of 1084 km<sup>2</sup>. The reservoir basin is located in a subtropical monsoon climate, with an annual rainfall of 1100–1800 mm and an average annual temperature of 16.8 °C (Wang et al., 2021). The total phosphorus (TP) concentration in

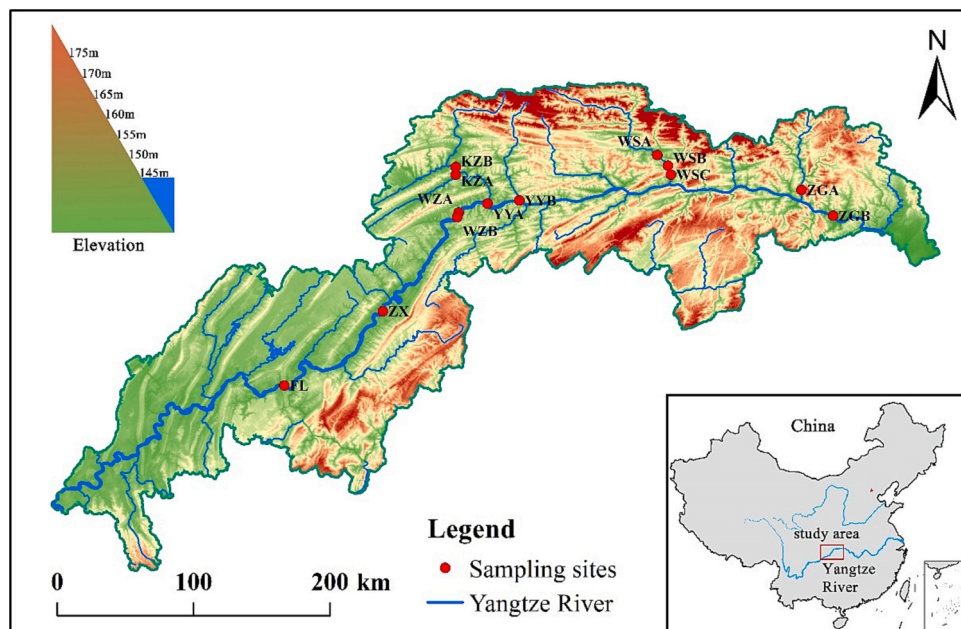


Fig. 1. Location of study area and 13 sampling sites in the Three Gorges Reservoir basin of the Yangtze River in China.

the water body of the Three Gorges Reservoir is 0.077 mg/L–0.34 mg/kg, and the soluble reactive phosphorus (SRP) concentration is below 0.030 mg/L (Tang et al., 2023). The WLFZ is the area formed by the rise and fall of the water level. Specifically, when the water level after TGR impoundment operates at an elevation of 175–145–175 m, the vertical fluctuation of the water level forms a WLFZ with an area of about 350 km<sup>2</sup> (Bao et al., 2015; Xiao et al., 2022). The water level of the Three Gorges Reservoir changes periodically, with a minimum level of 145 m from June to September and a maximum level of 175 m from November to January of the following year (Xiao et al., 2022). In this study, sampling sites were selected in the TGR from upstream to downstream, including: (i) main stream sites such as Fuling (FL), Zhongxian (ZX), Wanzhou (WZA and WZB), Yunyang (YYA and YYB), and Zigui (ZGB); and (ii) tributary sites such as Kaizhou (KZA and KZB), Wushan (WSA, WSB, and WSC), and Zigui (ZGA) (Fig. 1). Among these, Wanzhou, Yunyang, Kaizhou, and Zigui all collected artificially restored WLFZ samples (WZA, YYA, KZA, and ZGA) and natural WLFZ samples (WZB, YYB, KZB, and ZGB), respectively, while Fuling, Zhongxian, and Wushan collected only natural WLFZ samples (Fig. 1). Samples were collected at elevations of 145 m (always flooded), 150 m (flooded for 320 days), 155 m (flooded for 251 days), 160 m (flooded for 220 days), 165 m (flooded for 150 days), 170 m (flooded for 80 days), and 175 m (not flooded) (Nsabimana et al., 2023; Zhang et al., 2023). The main models of artificial restoration in WLFZ are the near-natural model of planting herbaceous plants in low-elevation areas and trees in high elevation areas, the artificial wetland model of constructing wetlands in flat, low-lying areas, and the bioengineering model of combining biological measures with engineering measures (Li et al., 2021).

## 2.2. Sample collection and preservation

In August 2021, at the selected 13 sampling sites (Fig. 1) starting from 145 m elevation, a sample square was randomly chosen at the 5 m elevation interval (i.e., 145 m, 150 m, 155 m, ..., 175 m) and a sample square of 1 m in length and 1 m in width was randomly selected; the debris, such as plants and stones, was removed from the soil surface. Five points were selected within the sample square using the plum-shaped placement sampling method, and soil samples were collected from 0 to 5 cm of the surface layer using a ring knife. The collected soil samples were mixed, placed in sealed polyethylene bags, and

transported back to the laboratory to dry at room temperature in a cool place. One part of the sample was used to determine the soil particle size, and the other part was ground and passed through a 100-mesh screen for the remaining indicators, and then all was placed at  $-20^{\circ}\text{C}$  for storage.

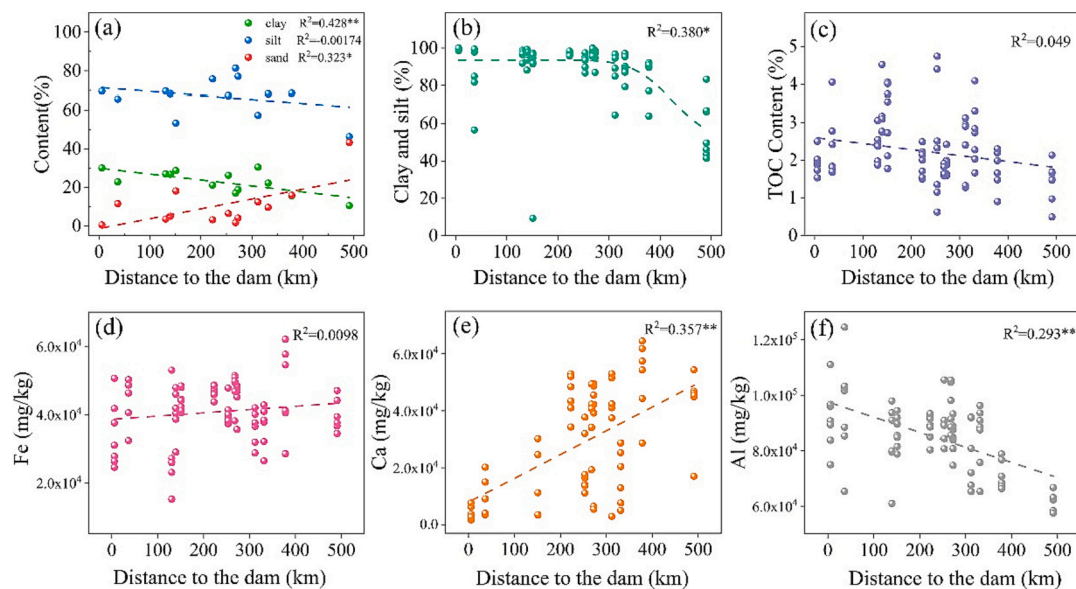
## 2.3. Sample analysis

### 2.3.1. Analysis of total organic carbon (TOC), particle size, and elemental composition of WLFZ soils

The carbonate in the soil samples was removed with an excess of 1 mol/L HCl solution for 24 h. The samples were then rinsed several times with ultrapure water until the pH value was neutral. The samples were freeze-dried and ground, and the total organic carbon (TOC) of the soils was determined using an organic elemental analyzer (Elementar-vario MACRO, DEU, accuracy  $<0.2\%$ ). Hydrochloric acid (HCl) and hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) were added to the soil samples to remove carbonate and organic matter, and the particle size of the samples was determined using a laser particle size analyzer (Mastersizer 2000, UK). Particles with sizes  $<4\ \mu\text{m}$ , between 4 and 64  $\mu\text{m}$ , and  $>64\ \mu\text{m}$  were defined as clay, silt, and sand, respectively. Particles  $<64\ \mu\text{m}$  in size are defined as fine particles (Bing et al., 2022; Tang et al., 2018). Finally, soil samples were digested with a concentrated acid mixture of HCl and nitric acid ( $\text{HNO}_3$ ), and the contents of Fe, Mn, Ca, and Al in the digest were determined by inductively coupled plasma-atomic emission spectrometry (ICP-OES) (Bing et al., 2022).

### 2.3.2. Soil P speciation analysis

The P form of soil samples was determined by a modified sequential extraction method (Hedley et al., 1982). The used extractants included ultrapure water ( $\text{H}_2\text{O}$ ), 0.5 mol/L  $\text{NaHCO}_3$  (pH = 8.5), 0.1 mol/L NaOH, and 1 mol/L HCl. After adding each extractant, the tubes were shaken for 16 h and centrifuged at 3600 rpm for 10 min. Afterwards, the supernatant was passed through a 0.45  $\mu\text{m}$  membrane to obtain  $\text{H}_2\text{O-P}_i$ ,  $\text{NaHCO}_3\text{-P}_i$ ,  $\text{NaOH-P}_i$ , and  $\text{HCl-P}_i$ . The remaining soil residue was freeze-dried and ashed for 2 h on a muffle furnace, which was then shaken with 3.5 mol/L HCl solution for 16 h and centrifuged at 3600 rpm for 10 min. The supernatant was passed through a 0.45  $\mu\text{m}$  filter membrane to obtain residual P (Res-P). The P concentration in each extract was determined by the molybdenum blue method (Murphy and Riley, 1958).



**Fig. 2.** Relationship of particle size (<4  $\mu\text{m}$  clay; 4–64  $\mu\text{m}$  silt; and > 64  $\mu\text{m}$  sand), total organic carbon (TOC), and metal elements in WLFZ soil with the distance to the dam. (a) Clay, silt, and sand content with the distance to the dam. (b) Fine particle (clay + silt) content with the distance to the dam. (c) TOC content with the distance to the dam. (d) Fe content with the distance to the dam. (e) Ca content with the distance to the dam. (f) Al content with the distance to the dam.

### 2.3.3. $^{31}\text{P}$ NMR analysis of soil organic P

The collected soil sample (1.00 g) was weighed into a 50 mL centrifuge tube, and extracted with 10 mL of a mixture of 0.25 mol/L NaOH and 0.05 mol/L ethylenediaminetetraacetic acid (EDTA) at room temperature for 16 h (Cade-Menun et al., 2005). The extracts were freeze-centrifuged at 13,000 rpm for 15 min, and a portion of the supernatant was diluted to determine the NaOH-EDTA total P (TP) and NaOH-EDTA inorganic P ( $\text{P}_i$ ) concentrations in the extracts, and the NaOH-EDTA-TP/ $\text{P}_i$  concentration was determined by the same method described above (Murphy and Riley, 1958). The concentration of NaOH-EDTA organic P ( $\text{P}_o$ ) in the extracted solution was calculated from the difference between total P and inorganic P. The remaining extract was freeze-dried into powder, which was then dissolved by a mixture of 10 mol/L NaOH and  $\text{D}_2\text{O}$ , sonicated at room temperature, and centrifuged at high speed before analysis using  $^{31}\text{P}$  NMR, following the procedures described in the literature (Doolette et al., 2011; Jin et al., 2022; Qu et al., 2019).

### 2.3.4. Kinetics and isotherms of phosphate adsorption onto WLFZ soils

All adsorption experiments were carried out at  $25 \pm 1$  °C under phosphate concentrations of 0, 1, 2, 5, 10, 20, and 50 mg/L, respectively. The adsorption and isotherms were studied in batch experiments with the following equations.

The pseudo-first-order kinetic model and pseudo-second-order kinetic model were used to describe the adsorption kinetic process by Eqs. (1,2), respectively:

$$Q_t = Q_e [1 - \exp(-k_1 t)] \quad (1)$$

$$Q_t = \frac{k_2 Q_e t}{1 + k_2 Q_e t} \quad (2)$$

where  $Q_t$  and  $Q_e$  are the amount of phosphate adsorbed on soil (mg/kg) at time  $t$  and at equilibrium  $t$ , respectively, and  $k_1$  and  $k_2$  are the pseudo first-order rate constant and pseudo second-order rate constant, respectively.

The Langmuir model was used to describe the adsorption isotherm, and its formula is (3).

$$\frac{C_e}{Q_e} = \frac{C_e}{Q_m} + \frac{1}{K_l Q_m} \quad (3)$$

where  $Q_m$  is the maximum monolayer unit adsorption of phosphate in solution by the soil (mg/kg),  $C_e$  is the phosphate concentration in solution at the moment of equilibrium (mg/L),  $Q_e$  is the unit adsorption of phosphate in solution species by the soil (mg/kg), and  $K_l$  is the equilibrium constant of Langmuir adsorption (L/mg).

Kinetic data were measured at an initial P concentration of 50 mg/L with time intervals set at 0, 0.5, 1, 4, 8, 16, 24, and 48 h, respectively. Isotherms were measured at seven initial P concentrations (0, 1, 2, 5, 10, 20, and 50 mg/L), and the oscillation time was set at 24 h.

### 2.4. Data analysis

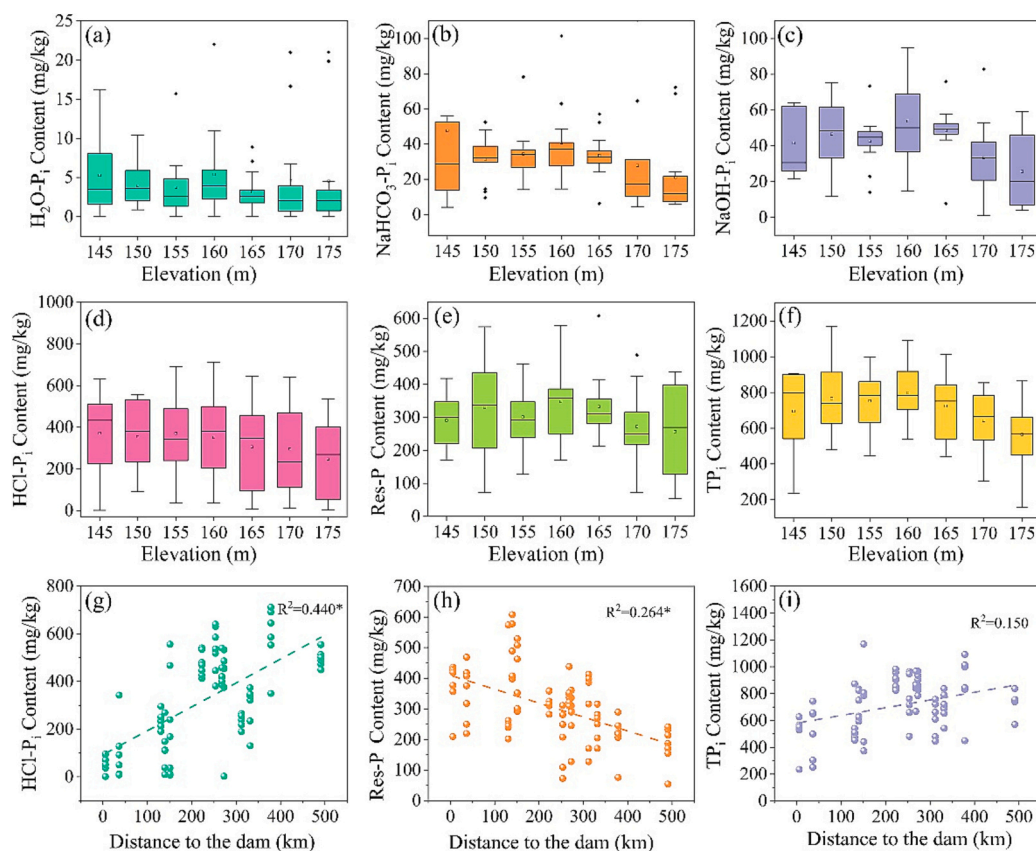
One-way analysis of variance (ANOVA) was performed for the statistical analyses of the collected data using IBM SPSS software (v. 27.0.1.0). Origin (v. 10.0.5.153) was used to plot the graphs and fit the adsorption and isotherms of P on soils.

## 3. Results and discussion

### 3.1. Distribution characteristics of soil grain size, TOC, and metal elements in WLFZ

WLFZ soils are mainly composed of fine particles (clay + silt) that account for >80 % of all particle sizes (Fig. 2b). The clay particle content increases significantly toward the dam. The silt content increases toward the dam, but this is not significant. Sand content decreases significantly toward the dam (Fig. 2a). The content of fine particles (<64  $\mu\text{m}$ ) showed an increasing and then flattening trend toward the dam, from about 60 % to about 90 % (Fig. 2b). Previous studies showed that the content of sand and clay in WLFZ soil particles increased with elevation, while the opposite trend occurred for the content of silt (Tang et al., 2018; Wang et al., 2020a). Others found that in the lateral direction, the fine particle content showed a significant increasing trend toward the dam (Bing et al., 2022). Tributary sediment input from upstream and frequent events such as landslides, avalanches, and mudslides downstream of the Jinsha River deliver large amounts of sediment to the Three Gorges. During the dry season, coarser sediment particles are deposited preferentially upstream of the reservoir, while fine particles are transported downstream of the TGR (Li et al., 2021).





**Fig. 3.** Relationship of sequentially extracted P and residual P concentrations in WLFZ soil with elevation and the distance to the dam ( $n = 3$ ). (a)  $\text{H}_2\text{O-P}_i$  with elevation, (b)  $\text{NaHCO}_3\text{-P}_i$  with elevation, (c)  $\text{NaOH-P}_i$  with elevation, (d)  $\text{HCl-P}_i$  with elevation, (e) Residual-P with elevation, (f)  $\text{TP}_i$  with elevation, (g)  $\text{HCl-P}_i$  with the distance to the dam, (h) Res-P with the distance to the dam, and (i)  $\text{TP}_i$  with the distance to the dam.

Total organic carbon (TOC) in soil affects the soil carbon pool, influences the activities of soil microorganisms, and has an important impact on carbon cycling. The soil TOC content of WLFZ increases toward the dam (Fig. 2c). The change in Fe content ( $4.1 \times 10^4$  mg/kg) toward the dam is not significant. However, the content of Al increases significantly toward the dam, with the average content increasing from about  $8.0 \times 10^4$  mg/kg to about  $1.0 \times 10^5$  mg/kg. The content of Ca decreases significantly toward the dam, with the average content decreasing from  $4.7 \times 10^4$  mg/kg to  $4.0 \times 10^3$  mg/kg (Fig. 2d-f).

### 3.2. Spatial variation of soil P speciation in WLFZ

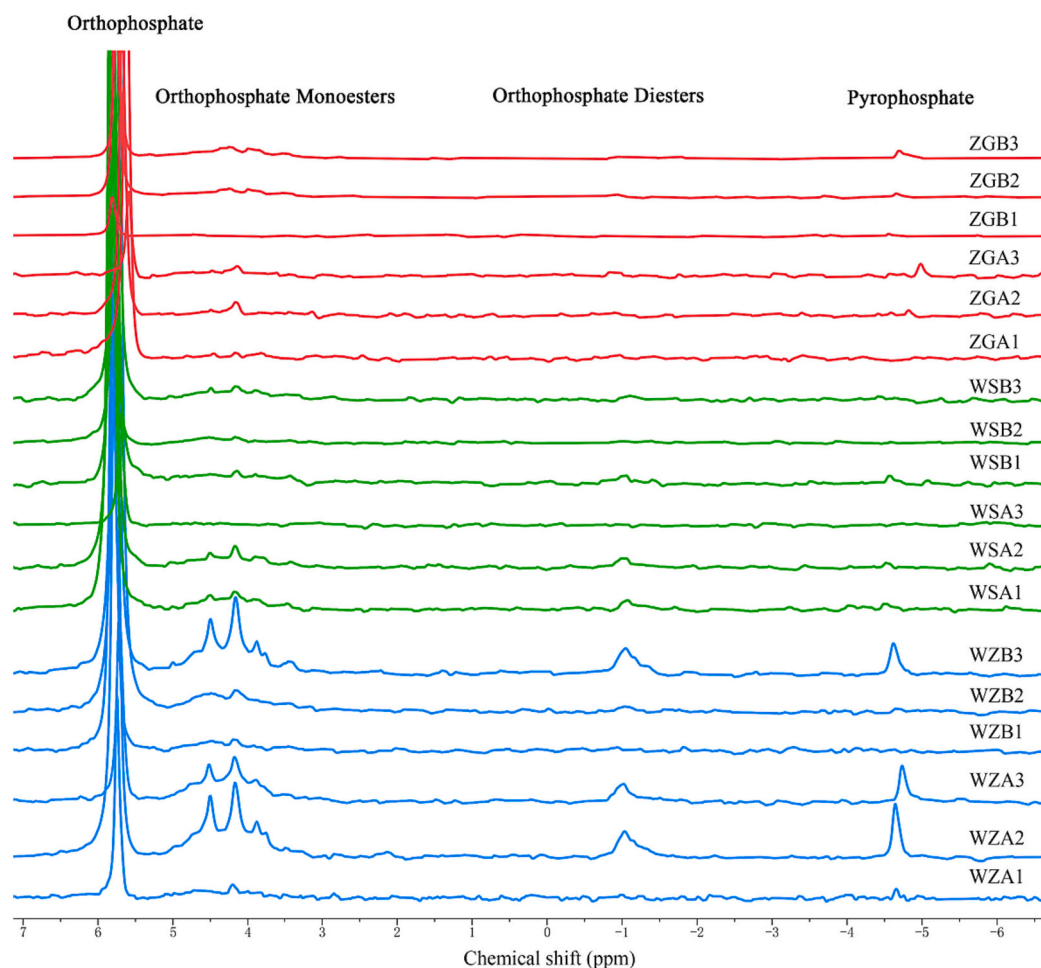
The content of various P forms in the WLFZ soils followed the following order:  $\text{HCl-P}_i$  (mean: 338 mg/kg) >  $\text{NaOH-P}_i$  (mean: 47.7 mg/kg) >  $\text{NaHCO}_3\text{-P}_i$  (mean: 33.0 mg/kg) >  $\text{H}_2\text{O-P}_i$  (mean: 4.30 mg/kg) (Fig. 3a-e). The two bioavailable P pools of  $\text{H}_2\text{O-P}_i$  and  $\text{NaHCO}_3\text{-P}_i$  ranged from 2.50 mg/kg to 10.8 mg/kg and 20.5 mg/kg to 50.6 mg/kg, respectively, which occupies a small proportion (< 8.4 %) of the total P.  $\text{NaOH-P}_i$  is seen as inorganic P adsorbed to Fe/Al minerals, which can also be regarded as conditionally bioavailable under altered redox conditions or enzymatic action (Hou et al., 2018a). The  $\text{NaOH-P}_i$  content of WLFZ soils ranged from 25.2 mg/kg to 55.7 mg/kg, which was comparable to the  $\text{NaHCO}_3\text{-P}_i$  content.  $\text{HCl-P}_i$  is mainly Ca-bound P but also includes some poorly crystalline P occluded by Fe and Al oxides (Gu et al., 2020; Zhang et al., 2022).  $\text{HCl-P}_i$  in WLFZ soils was one of the most abundant P forms, ranging from 250 mg/kg to 380 mg/kg (mean: 335 mg/kg), accounting for about 46.2 % of the total P content. Res-P is normally considered not bioavailable (Nakayama et al., 2021), and its content ranged from 300 mg/kg to 351 mg/kg (mean: 305 mg/kg). The content of different forms of P in WLFZ soils showed differences with

elevation. Except for  $\text{H}_2\text{O-P}_i$  and  $\text{NaHCO}_3\text{-P}_i$ , the content of different forms of P and total inorganic P was highest at 160 m elevation (Fig. 3a-f). Soils at mid-elevation (around 160 m), with frequent dry-wet alternations due to periodic fluctuations in water levels, are able to enrich P that is lost at higher elevations, and thus have a higher P content. The variation of P content in different forms with elevation reflects the modification of WLFZ soil element content and speciation by fluctuating dry-wet cycles.

The content of soil  $\text{HCl-P}_i$  decreased significantly toward the dam ( $R^2 = 0.440$ ,  $p < 0.05$ ), while the content of soil Res-P increased significantly toward the dam ( $R^2 = 0.264$ ,  $p < 0.05$ ) (Fig. 3g-h). In the direction away from the dam, the  $\text{HCl-P}_i$  concentration increased from 93.0 mg/kg to 584 mg/kg. In contrast, the Res-P concentration decreased from 410 mg/kg to 186 mg/kg. Previous studies reported no difference in the total P concentration of WLFZ soils over distance, with a mean content of 595 mg/kg (Zhang et al., 2012b). It has also been found that the WLFZ soil TP content was higher in the vicinity of the dam (Wang et al., 2020a). The difference between our results and previously documented findings may be due to the different sampling locations, which also implies that the changes in soil total P and P speciation along the reservoir WLFZ are complex and may be controlled by several factors such as soil loss, vegetation cover, and anthropogenic activities.

### 3.3. Spatial distribution of soil organic P in WLFZ

Reported inorganic P that can be detected by solution  $^{31}\text{P}$  NMR spectroscopy includes orthophosphate, pyrophosphate, and polyphosphate (Cade-Menun and Liu, 2014), while detectable organic P includes phosphate monoesters, phosphate diesters, and phosphonates.



**Fig. 4.** Characterization of the  $^{31}\text{P}$  NMR changes of NaOH-EDTA- $\text{P}_o$  in the selected samples of the WLFZ soils in the Three Gorges Reservoir basin. (1 represents 145 m elevation, 2 represents 160 m elevation, 3 represents 175 m elevation.)

**Table 1**

The concentration percentage of different P and the ratio of diester-P: mono-P in all samples. Ortho-P, orthophosphate; mono-P, phosphate monoesters; diester-P, phosphate diesters; pyro-P, pyrophosphate.

Sample	Elevation (m)	Ortho-P (%)	Mono-P (%)	Diester-P (%)	Pyro-P (%)	Diester-P/Mono-P
WZA	145	0.72	0.28	0.00	0.00	0.00
	160	0.57	0.29	0.09	0.05	0.29
	175	0.37	0.48	0.09	0.06	0.18
WZB	145	0.83	0.13	0.03	0.02	0.20
	160	0.85	0.14	0.02	0.00	0.13
	175	0.58	0.30	0.09	0.03	0.29
WSA	145	0.77	0.14	0.06	0.03	0.44
	160	0.78	0.15	0.05	0.02	0.37
	175	0.71	0.18	0.06	0.05	0.32
WSB	145	0.76	0.18	0.05	0.01	0.30
	160	0.81	0.12	0.06	0.02	0.47
	175	0.72	0.17	0.07	0.03	0.42
ZGA	145	0.93	0.07	0.00	0.00	0.00
	160	0.75	0.16	0.04	0.04	0.27
	175	0.53	0.27	0.12	0.07	0.45
ZGB	145	0.59	0.13	0.22	0.06	1.68
	160	0.70	0.26	0.04	0.00	0.14
	175	0.52	0.36	0.07	0.04	0.20

Orthophosphate is considered the most bioavailable form of P in soluble IP (Steinman et al., 2012). Phosphate monoester is the main component of soil organic P, including inositol phosphates, mononucleotides, sugar phosphates, and by-products of phospholipid catabolism.

The results of solution  $^{31}\text{P}$  NMR for selected samples are shown in Fig. 4. The main form of NaOH-EDTA  $\text{P}_i$  was orthophosphate, ranging from 27.6 mg/kg to 498 mg/kg, which accounts for 37.4 %–92.6 % of NaOH-EDTA TP. Pyrophosphates are synthesized by algae, fungi, and bacteria and are the most unstable P compounds (Qu et al., 2019; Zhu et al., 2015). The content of pyrophosphate was 1.80 mg/kg to 13.1 mg/kg, accounting for 0.7 % to 7.6 % of NaOH-EDTA TP. The main form of NaOH-EDTA  $\text{P}_o$  is phosphate monoester, with 8.70 mg/kg to 114 mg/kg, accounting for 7.4 % to 47.6 % of NaOH-EDTA TP. Phosphate monoester can be rapidly mineralized to phosphate in an anaerobic environment and utilized by aquatic organisms (Cade-Menun et al., 2005). The content of phosphate diester was 3.90 mg/kg to 34.8 mg/kg, accounting for 1.7 % to 21.9 % of NaOH-EDTA TP. Phosphate diesters mainly include nucleic acid and phospholipid, and the degradation rate of phosphate diesters is faster than that of phosphate monoesters (Makarov et al., 2002).

In natural environment, organic P accounts for approximately 20 %–80 % of total soil P (Tiecher et al., 2012). Phosphate diester is more easily mineralized than phosphate monoester. The ratio of phosphate diester to phosphate monoester can provide more information on the mineralization of organic P. The average ratio of phosphate diester/phosphate monoester was lower in the artificially restored WLFZ than in the natural WLFZ (Table 1), suggesting that possibly the rate of organophosphorus mineralization was faster in the natural WLFZ. Inorganic phosphorus after mineralization of organic P is a source of P for plants, especially in highly weathered soils (Vincent et al., 2010).

The NaOH-EDTA TP,  $\text{P}_i$ , and  $\text{P}_o$  contents of natural WLFZ were

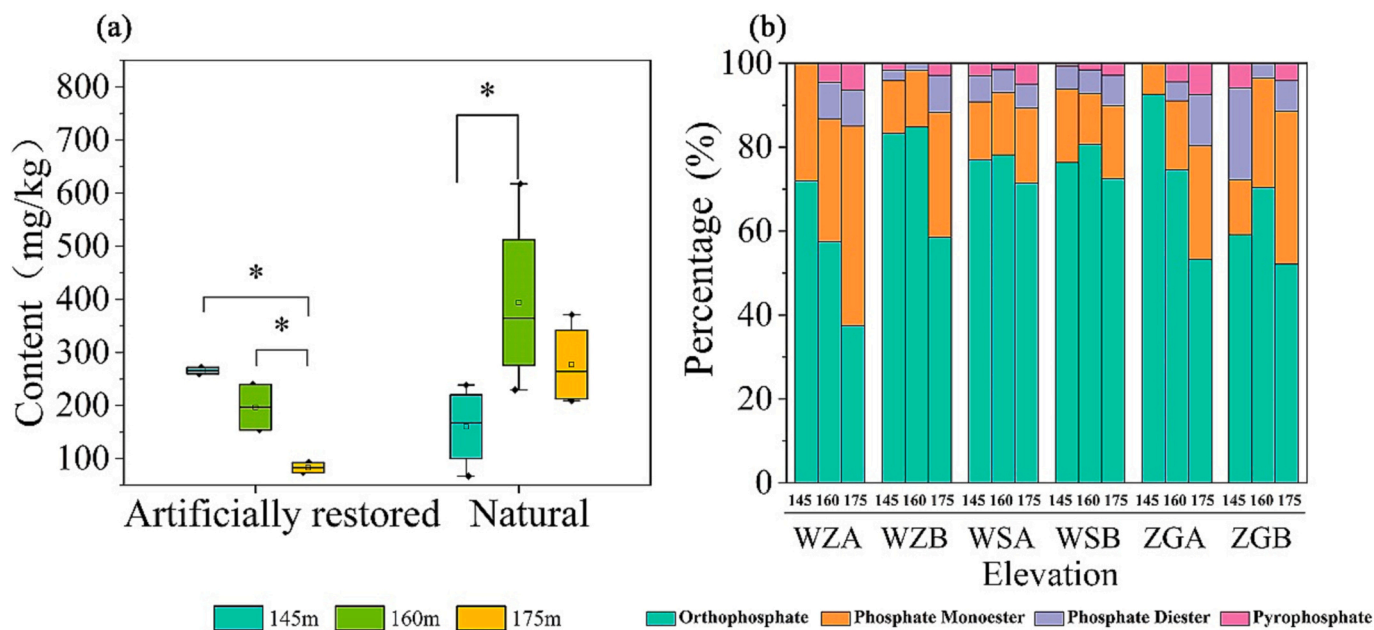


Fig. 5. Variation in the content of NaOH-EDTA-extracted WLFZ soil P. (a) Relative content of P at different elevations obtained from solution <sup>31</sup>P NMR analysis (b) Content of each component of P at different elevations.

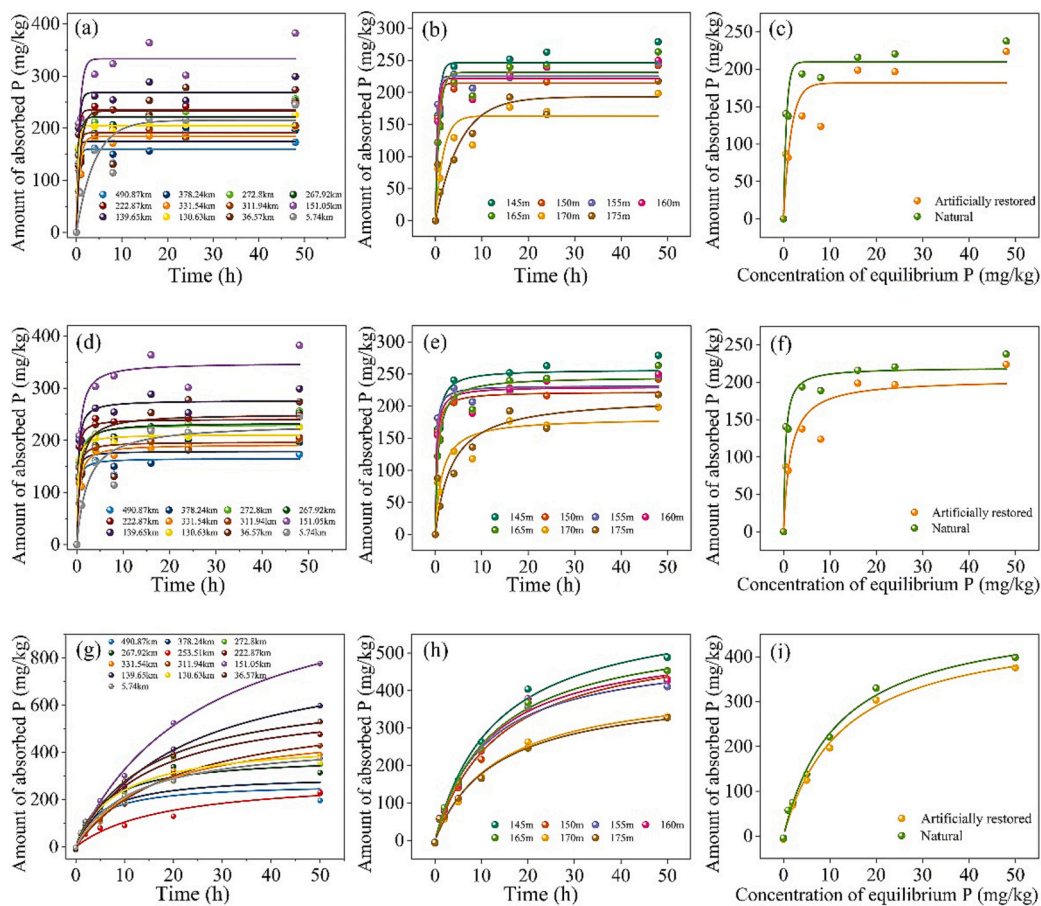


Fig. 6. Kinetic and isotherm curves of phosphate adsorption onto WLFZ soil (n = 3). (a) pseudo first order model curves at different distances to the dam, (b) pseudo second-order model curves at different distances to the dam, (c) Langmuir model curves at different distances to the dam, (d) pseudo first order model curves at different elevations, (e) pseudo second-order model curves at different elevations, (f) Langmuir model curves at different elevations, (g) Artificial restoration and natural WLFZ pseudo first order model curves, (h) Artificial restoration and natural WLFZ pseudo second-order model curves, (i) Artificial restoration and natural WLFZ Langmuir model curves.

greater than those of artificially restored WLFZ. In the artificially restored WLFZ, the content of NaOH-EDTA TP showed a trend toward decreasing with increasing elevation. The highest NaOH-EDTA TP content (328 mg/kg) was found in the natural WLFZ at 160 m elevation,

while the lowest NaOH-EDTA TP content occurred at 145 m elevation. The content of orthophosphate accounts for most of the total P content, so the trend of orthophosphate is also similar to the trend of total P (Fig. 5a). The relative content of phosphate monoester tended to



**Table 3**

Parameters of the equations of the quasi primary and quasi secondary kinetic models for adsorption.

Sample	Pseudo first order kinetic model			Pseudo second-order kinetic model		
	Q <sub>e</sub>	k <sub>1</sub>	R <sup>2</sup>	Q <sub>e</sub>	k <sub>2</sub>	R <sup>2</sup>
Artificially restored	224	0.158	0.737	224	0.00196	0.870
Natural	238	0.279	0.495	238	0.00403	0.875
145 m	279	0.345	0.538	280	0.00535	0.912
150 m	243	0.338	0.423	242	0.00795	0.916
155 m	247	0.478	0.450	247	0.00584	0.829
160 m	250	0.319	0.357	250	0.00572	0.883
165 m	263	0.256	0.623	263	0.00321	0.923
170 m	198	0.214	0.752	198	0.00186	0.873
175 m	218	0.134	0.803	218	0.00123	0.863
FLB (490.87 km)	160	2.40	0.833	165	0.0270	0.877
ZXB (378.24 km)	174	3.10	0.870	179	0.0375	0.895
WZA (272.80 km)	222	1.75	0.899	231	0.0127	0.942
WZB (267.92 km)	222	1.58	0.896	234	0.0100	0.949
YYB (222.87 km)	235	2.61	0.959	240	0.0237	0.978
KZA (331.54 km)	184	1.39	0.928	192	0.0116	0.960
KZB (311.94 km)	191	2.19	0.938	197	0.0218	0.967
WSA (151.05 km)	333	1.45	0.912	349	0.0065	0.946
WSB (139.65 km)	268	2.11	0.931	277	0.0143	0.960
WSC (130.63 km)	205	2.40	0.936	211	0.0225	0.968
ZGA (36.57 km)	233	1.13	0.720	251	0.0055	0.770
ZGB (5.74 km)	215	0.252	0.768	232	0.00188	0.848

increase with elevation in WLFZ soils at different elevations (Fig. 5b). Soil dry-wet cycles strongly increase mineralization and can increase the availability of plant nutrients, but they are also accompanied by leaching losses. Mechanisms of nutrient release due to soil dry-wet cycles include physical disruption of soil structure, desorption of substrate from the surface, and death and lysis of soil microorganisms (Gu et al., 2018). Periodic dry-wet cycles also accelerate P depletion in soils (Brödlin et al., 2019; Wang et al., 2020b). Vegetation also affects the P content of the WLFZ soils. Plants grow in the soil to absorb effective phosphorus, and dead or dying branches, leaves, etc. will replenish some of the soil phosphorus and release another part into the water (Wang et al., 2020a). The availability of soil phosphorus depends on the total phosphorus content of the soil and soil properties such as soil minerals (Fe and Al content and composition), and organic matter also affects the amount of unstable phosphorus in the soil (Hou et al., 2018b; Sun et al., 2017). In this study, the contents of organic carbon and Fe were higher in the artificially restored WLFZ than in the natural WLFZ (Fig. 2), which would make the artificially restored WLFZ more vulnerable to P loss.

### 3.4. Adsorption of P onto WLFZ soils

The phosphorus sorption characteristics of WLFZ soils were evaluated by sorption and isotherm experiments to explore the effect of WLFZ on phosphorus changes in Three Gorges Reservoir during periodic dry-wet cycles. The phosphorus adsorption and isothermal processes at different distances, altitudes, and in artificially restored versus natural WLFZ soils are shown in Fig. 6. The parameters of the pseudo-first-order kinetic model and the pseudo-second-order kinetic model are shown in Table 3. The maximum amount of phosphorus adsorbed by the WLFZ soil basically showed a gradual increase in the direction toward the dam (Table 3). From different elevations, the pseudo second-order kinetic model ( $0.829 < R^2 < 0.923$ ) is more suitable for describing the kinetics of phosphate adsorption by WLFZ soils (Fig. 6a-e). The P adsorption increased rapidly with time within 10 h. After 10 h, the adsorption almost reached equilibrium, the kinetics of adsorption were similar for all samples, and the results were consistent with previous reports (Zhang et al., 2012a).

The pseudo-second-order kinetic model is mainly used to describe chemisorption kinetic processes. The adsorption process of sorbent on adsorbent is mainly controlled by chemisorption (El-Khaiary et al.,

**Table 2**

Equation of adsorption isotherm isothermal curves of phosphate adsorption by WLFZ soil.

Sample	Langmuir function	R <sup>2</sup>
Artificially restored	C/Q = 0.0269 + 0.00210C	0.985**
Natural	C/Q = 0.0851 + 0.00200C	0.989**
145 m	C/Q = 0.0208 + 0.00159C	0.991**
150 m	C/Q = 0.0244 + 0.00180C	0.984**
155 m	C/Q = 0.0208 + 0.00196C	0.987**
160 m	C/Q = 0.0212 + 0.00184C	0.979**
165 m	C/Q = 0.0218 + 0.00175C	0.992**
170 m	C/Q = 0.0321 + 0.00236C	0.976**
175 m	C/Q = 0.0316 + 0.00246C	0.977**

\*\* Indicates significant difference at p xxx level.

2010). Therefore, the main mechanism of phosphate adsorption by WLFZ soils is chemisorption. Overall, the adsorption kinetic adsorption equilibrium concentrations of P in the WLFZ soils far from the dam were lower than those closer, with the average maximum adsorption reduced from 230 mg/kg to 182 mg/kg. The WLFZ soil adsorption equilibrium concentrations were lower at high elevations than at low elevations, with the average maximum adsorption reduced from 271 mg/kg to 192 mg/kg. Natural WLFZ (229 mg/kg) showed higher mean maximum adsorption than artificially restored WLFZ (210 mg/kg). As can be seen from Fig. 6, the adsorption of P by the soil basically tends to plateau around 16 h and almost reaches a maximum.

The Langmuir isothermal adsorption model is based on the assumption that the adsorbent surface is homogeneous and that a limited number of adsorption sites exist. The adsorbent is adsorbed on the adsorbent surface to form a single molecular layer, and the adsorption sites will not continue to adsorb after being occupied by the adsorbent (Langmuir, 1916). The isothermal adsorption of P by WLFZ soils was fitted using the Langmuir isothermal model, and the R<sup>2</sup> (0.735 to 0.997) of the thermodynamic fit varied widely for different distances (Table 2). The R<sup>2</sup> of the P isothermal adsorption fitted at different elevations was above 0.976, indicating that the Langmuir isothermal model fits well for the experimental data of adsorption on WLFZ soils at different elevations (Tang et al., 2014; Wu et al., 2021).

The maximum P adsorption by WLFZ soils at different distances from the dam ranged from 197 mg/kg to 530 mg/kg. This is similar to the variation in P content, where high soil P content results in low P adsorption, and more P may be sorbed by the WLFZ soils upstream of the reservoir, resulting in less P downstream. The maximum adsorption of P at different elevations ranged from 328 mg/kg to 488 mg/kg, with the highest adsorption occurring at 145 m elevation and the lowest adsorption at 175 m elevation. This may be related to the duration of inundation, which was shorter at 175 m and resulted in less phosphorus loss, and longer at 145 m, which resulted in more phosphorus loss. Artificially restored in natural WLFZ, the maximum P adsorption was 476 mg/kg in artificially restored WLFZ and 499 mg/kg in natural WLFZ. The phosphorus uptake of natural WLFZ soils is higher than that of artificially restored ones, which indicates that natural WLFZ has a higher phosphorus adsorption capacity. However, the TP<sub>i</sub> content of artificially restored WLFZ (748 mg/kg) was higher than that of natural WLFZ (679 mg/kg), which was mainly due to the fact that the HCl-P<sub>i</sub> content of artificially restored WLFZ (349 mg/kg) was much higher than that of natural WLFZ (200 mg/kg), while the Res-P content (316 mg/kg) was lower than that of natural WLFZ (395 mg/kg). This suggests that there may be a risk of phosphorus loss from the natural fallout zone, as HCl-P<sub>i</sub> and Res-P are biologically difficult to use for P.

### 3.5. Correlations of various factors in WLFZ soils

Statistical analysis showed that elevation was an important factor controlling Ca and Fe, and that Ca ( $R^2 = -0.30^*$ ) and Fe ( $R^2 = -0.24^*$ ) contents decreased significantly with increasing elevation, which is



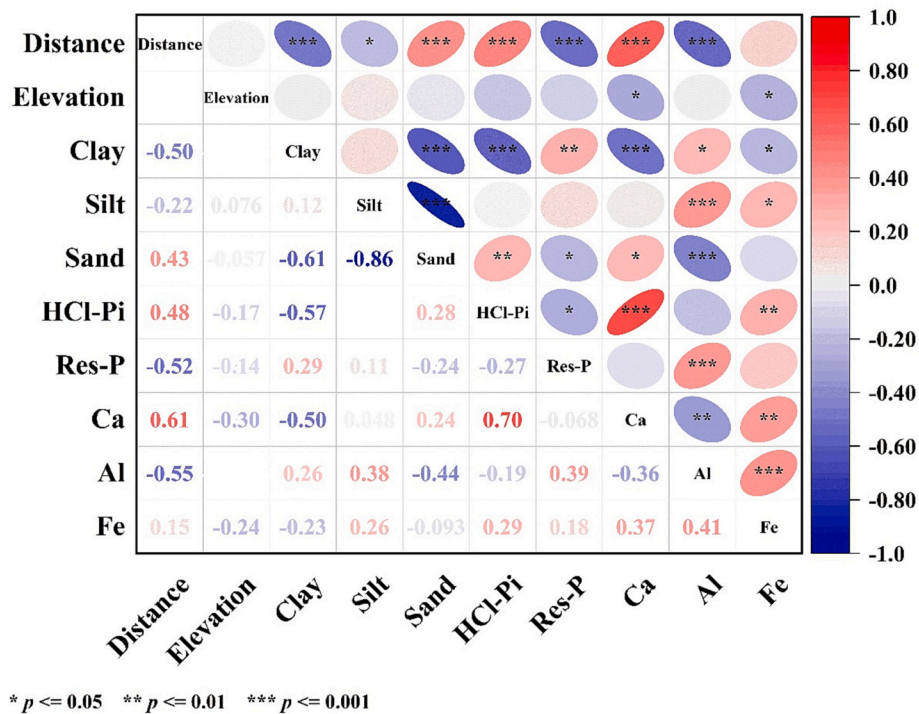


Fig. 7. Relationship between soil particle size (clay, silt, and sand), metal (Ca, Al, and Fe) element content, and P form (HCl-P<sub>i</sub> and Res-P) content.

likely related to the duration of periodic inundation of soils located at different elevations, water depth, and the resulting changes in redox conditions at the water-soil interface.

According to the experimental results, from Fuling to Yichang, The HCl-P<sub>i</sub> concentration of the WLFZ soil was decreasing in the direction toward the dam ( $R^2 = 0.48^{***}$ ), and the Ca content was also decreasing. In contrast, the change of Res-P is opposite to that of HCl-P<sub>i</sub>, whose content is increasing toward the dam ( $R^2 = -0.52^{***}$ ). The correlation analysis showed that the HCl-P<sub>i</sub> content is significantly and positively correlated with Ca content ( $R^2 = 0.70^{***}$ ), while Fe content showed a significant negative correlation with clay ( $R^2 = -0.23^*$ ). Res-P concentration showed a significant positive correlation with Al ( $R^2 = 0.39^{***}$ ) and clay and a significant negative correlation with sand ( $R^2 = 0.24^*$ ). This indicates that HCl-P<sub>i</sub> is mainly enriched in coarse particles, which usually have a higher Ca-P content compared to other particles with smaller particle sizes (Dam et al., 2021; Zhang et al., 2022). Coarse particles settle faster than fine particles such as clay due to their larger size and greater density, and coarse particles are mainly distributed in the direction away from the dam, while HCl-P<sub>i</sub> is thus settled down. The Res-P is mainly enriched in fine particles, which can move farther along the water flow, and the residual phosphorus content is higher the closer to the dam direction (Fig. 7).

#### 4. Conclusion

This study investigated the effects of periodic inundation-emergence, different elevations, long-range, and artificial restoration on P speciation in WLFZ soils to understand the migration and enrichment pathways of P in WLFZ soils. Results showed that at different elevations, the P concentrations of all forms, except for H<sub>2</sub>O-P<sub>i</sub>, exhibited an increasing trend and then a decreasing trend, with the highest concentration occurring at 160 m elevation. The migration of HCl-P<sub>i</sub> and Res-P, the most abundant of the different P forms, was controlled by the sand and clay content of the soil, respectively. In the direction away from the dam, the adsorption of phosphorus by the WLFZ soil decreases, which is consistent with the variation of phosphorus content in the soil. The area upstream of the reservoir was the main enrichment area for P. While the

natural WLFZ has a higher P uptake than the artificially restored zone, the artificially restored zone has a higher P content, which could pose a potential risk of P release into the water. The WLFZ is an important regulatory zone for P in the TGR area, and the high P content in the upstream area of the reservoir can be reduced by anthropogenic means to reduce its over-enrichment in the WLFZ. The natural WLFZ has a better buffering capacity for P, so it is important to protect the WLFZ area and minimize its damage. We initially aimed to explore the changes of P in WLFZ soils to reveal its change phenomena, but the intrinsic mechanisms still need further investigations. Further study is required to elucidate the P migration routes from different perspectives that provide a management basis and foundation for P regulation in the TGR.

#### 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

Data will be made available on request.

#### Acknowledgments

This study was sponsored jointly by the National Key Research and Development Plan of China (2021YFC3201000), the Strategic Priority Research Program of CAS (No. XDB40020400), the Chinese NSF project (No. 4227253, 42273083, 42077313), the Central Leading Local Science and Technology Development Fund Project (20214028), the Science and Technology Service Plan of CAS (KFJSTSQYD202124001), and the Youth Innovation Promotion Association of CAS (No. 2019389).

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