



# Phosphorus Forms and Associated Properties along an Urban–Rural Gradient in Southern China

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**Abstract:** Urbanization is widely assumed to degrade soil ecosystem services, but the changes in the urban soil phosphorus (P) status due to urbanization and the associated environmental implications have rarely been studied. The objective of this study was to investigate the P forms and associated soil properties in urban soils. Thirty sites were selected along an urban–rural gradient in Nanchang, China, to examine the effects of urbanization on soil P fractions. Residual P and NaOH-extractable P (NaOH-Pi and NaOHPo) were the major P forms in the 0–30 cm of urban soils, comprising on average 37% and 43% of the total P pool, respectively, similar to the suburban and rural soils. Compared with non-urban soils, urban soil had higher contents of total P and P fractions (i.e.,  $P_{H2O}$ ,  $P_{KCI}$ , NaOH-Pi,  $P_{HCI}$ , and residual P), as well as higher contents of related soil P-retentive properties, especially soil pH and Mehlich 3-extractable Ca and Mg. Phosphorus enrichment in the urban soils may become a source of aquatic pollution because the soil labile P content (the sum of  $P_{H2O}$  and  $P_{KCI}$ ) was positively related to total P,  $P_{HCI}$ , NaOH-Pi, and residual P, which implied that the labile P can be replenished by these P pools. This study increased the understanding of P stabilization characteristics (e.g., the specific P forms) of urban soils and has further implications for urban environmental management.

Keywords: technosol; phosphorus; urbanization; eutrophication; land use

# 1. Introduction

At present, more than half of the world's population resides in cities and towns. Migration from rural to urban areas is continuing at an alarming rate in developing countries, and the percent urban population is projected to increase to 66% by 2050 [1]. The rapidly expanding urban areas have caused large areas of agricultural, pasture or forest soil to be changed to urban soil [2–4]. As a significant component of urban ecosystems, urban soil has become increasingly important with regard to human health and wellbeing [5]. Urban soil is more strongly perturbed by human activities (e.g., mixing, land filling, compaction, and soil sealing) than other soils, resulting in negative impacts on soil functions and the urban environment [3,6,7]. Therefore, a growing body of literature focuses on environmental issues associated with urban soils, particularly contaminants such as heavy metals and organic pollutants (e.g., [8–10]). However, the characterization of the forms of urban soil phosphorus (P), which is usually enriched in urban areas and may cause aquatic eutrophication, and the soil properties associated with P retention in urban areas require further investigation [11].

In certain geographical regions, P build-up in the soil due to fertilization or other land management has become a concern, as it can represent a threat to water quality when transported into water bodies via surface runoff or leaching [12–14]. Previous studies have suggested that strong enrichment of P occurs in urban soils [11,15], which can originate from household ash and various anthropogenic



waste deposits in urban areas [11]. Phosphorus accumulation in urban soils may become more severe in the future because of the low mobility of soil P and the abundant external P input in urban areas. Human activities may also alter soil properties related to P accumulation such as soil pH [16]. Thus, it is critical to investigate the P retention status (e.g., changes in soil P forms) in urban soils to gain a better understanding of P behavior in the urban environment. However, the specific P forms and associated properties in urban soils remain largely unknown.

In soils, P is present in several forms or pools, and different forms are often designated as either inorganic or organic and are commonly further distinguished as P dissolved in soil water, P sorbed to surfaces of clay or Fe and Al oxides, P in phosphate minerals, and P in organic substances and living organisms [17,18]. These descriptions imply differences among P forms with respect to reactivity in the environment. Accounting for these various P pools, different approaches to extract P from the soil are available, and numerous soil P extraction methods have been developed (e.g., [17,18]). Sequential fractionation schemes are widely used to identify different soil P forms; they extract P from the soil with selective solvents that isolate P pools of different solubility. Generally, the extractants are designed to solubilize groups of minerals usually defined as P associated with Al, Fe, Ca or residual forms [19]. In this case, sequential fractionation schemes can also be employed to investigate the characteristics of P retention in urban soils.

Urban areas in China have been expanding rapidly in recent decades, and it has become urgent to obtain a comprehensive understanding of the effects of this land-use change, including P forms in urban soils. Determination of the P forms of surface urban soils will help to identify processes of P accumulation and the prediction of P mobility within such soils. The aim of our study was to investigate the specific P forms and associated P-retentive properties of urban soils in Nanchang, Southern China. We hypothesized that with abundant external P input and severe anthropogenic disturbance, urban soil would show different P forms and P-retentive properties compared with suburban or rural soils.

#### 2. Materials and Methods

## 2.1. Studied Site and Soils

This study was conducted in Nanchang, China (28°09′–29°11′ N, 115°27′–116°35′ E), covering a total area of 7400 km<sup>2</sup> and with a population of 5.24 million. The study area is situated on the plain of the middle course of the Yangtze River and has a humid, subtropical monsoon climate. The mean annual temperature is 17.5 °C, and the mean annual precipitation ranges from 1600 to 1800 mm, with a mean annual relative humidity of approximately 77%. Nanchang in Southern China provides an excellent location for studying the effects of urbanization on P accumulation in urban soils as it has been at its current position for at least 2000 years and has recently experienced rapid urbanization and has low P contents in its natural soils [20].

In total, 30 sites were selected along an urban–rural transect, across which 13 sites were selected from a highly urbanized area, 9 sites from a suburban area (a residential area on the outskirts of Nanchang, with lower population density than the adjacent inner city), and 8 sites from a rural area (Figure 1, full raw data can be found in Zenodo.org, doi: 10.5281/zenodo.3548178). The 30 sites were mainly located in urban parks, university campuses, and rural forest parks, in which the predominant plant communities were Masson's pine trees (*Pinus massoniana*) and camphor trees (*Cinnamomum camphora*). All sites are underlain by Ferralsols [21] (local name is red soil), which are primarily derived from Quaternary red clay with abundant kaolinite or from saprolite and colluvium underlain by phyllite and/or gneiss rocks in hilly areas. Generally, the soils in our study area had loam, silt loam, sandy loam, clay loam, and sandy clay loam textures.



**Figure 1.** Soil sampling sites along an urban–rural gradient in Nanchang, China (n = 30). At each site, soil samples were collected from 0–5, 5–15, and 15–30 cm depths, and a total of 90 bulked soil samples were obtained.

At each site, five separate soil cores (5 cm in diameter) were collected from three depths (0–5, 5–15, and 15–30 cm) and mixed to obtain one sample for each depth per site (a total of 90 samples). We sampled soils at depths <30 cm because the mobility of P is limited, and it is supposed to accumulate in the surface soil layer. All soil samples were air-dried. After the removal of visible plant residues, the soil samples were sieved through a 2 mm mesh, mixed, and stored at room temperature prior to chemical analysis.

# 2.2. Soil Phosphorus Characterization

Soil P was partitioned by sequential chemical extraction using a modified fractionation procedure that differentiated six forms of P [18,22] and consisted of four extractants: distilled deionized water, 1.0 M KCl, 0.1 M NaOH, and 0.5 M HCl. Briefly, 0.5 g dry weight soil was combined with 25 mL of each extraction solution in 50 mL centrifuge tubes to maintain a soil:solution ratio of 1:50. The tubes were successively shaken on a reciprocating shaker for 1, 2, 17, and 24 h for distilled deionized water, KCl, NaOH, and HCl, respectively [22]. Subsequently, sample solutions were centrifuged at 5000 rpm for 15 min, and supernatants were filtered through a 0.45 µm membrane filter. The remaining soil residues after successive extraction were analyzed for total P (i.e., residual P) after perchloric acid digestion [23]. Phosphorus measured in filtrates was assumed to be soluble inorganic P (Pi) and was determined colorimetrically using the method of Murphy and Riley [24]. Sodium hydroxide solutions were also measured for total P by persulfate digestion. Organically-bound P in the NaOH solution (NaOH-Po) was determined to be the difference between the Pi and total P in solution.

Following these analyses, the P forms were interpreted as follows: (1) P extracted by distilled deionized water ( $P_{H2O}$ ) and by KCl ( $P_{KCl}$ ) was defined as labile P; (2) P dissolved in NaOH solutions (NaOH-Pi) was considered to be Al- or Fe-bound P; (3) the NaOH-Po fraction was attributed to humic and fulvic acid P; (4) acid-extractable P ( $P_{HCl}$ ) was presumed to be Ca- or Mg-bound P; and (5) the composition of the residual P fraction was considered unknown, but is commonly inferred to contain recalcitrant P associated with minerals and organic matter [18].

Soil texture was determined using the hydrometer method [25]. Soil pH was measured in a 1:2.5 (soil: water ratio) mixture using a glass electrode. Amorphous Fe and Al hydroxides (denoted as Fe<sub>ox</sub> and Al<sub>ox</sub>, respectively), which are important P-stabilizing agents in acidic soil, were extracted by

shaking 1:40 soil:ammonium oxalate extractant solution (pH 3.0) for 2 h in the dark [26]. In addition, Mehlich-3 (M3)-extractable Ca and Mg (Ca<sub>M3</sub> and Mg<sub>M3</sub>, respectively), which can precipitate soil P, were extracted by shaking a 1:10 soil:M3 extractant solution for 5 min [27]. The sample solutions treated with oxalate and M3 extractant solutions were centrifuged at 5000 rpm for 15 min and filtrated; the supernatants were analyzed for Fe, Al, Ca, and Mg by ICP-OES (Optima 8000, PerkinElmer, Waltham, MA, USA).

# 2.3. Statistical Analysis

All analyses were performed in the statistical program R [28]. Differences in the soil sequential P fractions and soil properties regulating P retention along the urban–rural gradient or among different soil depths were analyzed statistically using two-way analysis of variance (p < 0.05). Multiple comparisons of means were performed by Tukey's honestly significant difference test using the agricolae package in R software [29]. Data were log<sub>10</sub> transformed (pH, Al<sub>ox</sub>, Ca<sub>M3</sub>, and P fractions except NaOH-Po) or square root transformed (NaOH-Po) to stabilize the variance of individual properties where necessary. Regression analysis and Spearman's correlation analysis were employed to investigate the specific relationships among the soil P fractions and associated soil properties. Redundancy analysis (RDA) was employed to reveal the crucial soil properties accounting for the variations of P fractions along the urban–rural gradient or among different soil depths using the vegan package [30].

# 3. Results

#### 3.1. Soil P Fractions

In general, soils in urban areas showed a higher total P content compared to soils in suburban and rural areas (p < 0.001, Table 1). In fact, the total P content of urban soil was 789, 942, and 835 mg kg<sup>-1</sup> at 0–5, 5–15, and 15–30 cm, respectively, which was more than twice that of the corresponding depths of rural soil (Figure 2). In addition, the total P content of 0–5 and 5–15 cm soil layers was significantly higher than that of 15–30 cm (p = 0.02). Sequential chemical P fractionation revealed that NaOH-extractable P (NaOH-Pi and NaOH-Po) and residual P were the major P forms in the 0–30 cm of urban soil, comprising an average of 43% and 37% of total P, respectively. Similarly, NaOH-extractable P and residual P were also the dominant P fraction in the soils from suburban and rural areas. Compared with rural soils, urban soils generally had significantly higher contents of P<sub>HCl</sub>, NaOH-Pi, residual P, and P<sub>KCl</sub>, and especially P<sub>HCl</sub>. Although P<sub>HCl</sub> was not the dominant P fraction in these soils, the increases in P<sub>HCl</sub> contributed nearly 80% of the total P increases in 15–30 cm urban soils compared with rural soils (Figure 2).

**Table 1.** Analysis of the effects of the urban–rural gradient and soil depth on the contents of soil total P and P fractions.

	P <sub>H2O</sub>	P <sub>KCl</sub>	NaOH-Pi	NaOH-Po	P <sub>HCl</sub>	Residual P	Total P
Location	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Depth	NS	NS	NS	0.002	NS	< 0.001	0.02
$Location \times Depth$	NS	NS	NS	NS	NS	0.03	NS

NS = not significant.

Water-extractable P and P<sub>KCl</sub>, cumulatively labile P, accounted for a very small portion of the total P pool of the three types of soils, i.e., less than 2% of the total P of the soils studied. Both P<sub>H2O</sub> and P<sub>KCl</sub> were significantly affected by the urban–rural gradient (p < 0.001, Table 1). The mean value of 0–30 cm soil labile P (P<sub>H2O</sub> + P<sub>KCl</sub>) contents in urban areas was 15.0 (±2.12, ±SE) mg kg<sup>-1</sup>, while it was only 5.12 (±2.39, ±SE) and 0.024 (±0.003, ±SE) mg kg<sup>-1</sup> in suburban and rural areas, respectively. In addition, soil labile P was strongly positively correlated with NaOH-Pi (r = 0.73, p < 0.001), P<sub>HCl</sub>

(r = 0.46, p < 0.001), residual P (r = 0.55, p < 0.001), and total P (r = 0.64, p < 0.001) across the urban–rural gradient and different soil depths (Figure 3).



**Figure 2.** Effects of the urban–rural gradient on soil P forms. Values are means ±1 standard error. Different letters for each P fraction at the same soil depth indicate significant differences among the urban, suburban, and rural gradients.

## 3.2. Soil Properties Associated with P Stabilization

We further measured the variations of some P-retentive soil properties (including soil pH, soil organic carbon,  $Al_{ox}$ ,  $Fe_{ox}$ ,  $Ca_{M3}$ ,  $Mg_{M3}$ , and clay) in response to the urban–rural gradient and soil depths. These properties (except clay) were significantly affected by the urban–rural gradient (Table 2). The urban soil contained the highest amount of  $Ca_{M3}$  (2397 mg kg<sup>-1</sup>) and  $Mg_{M3}$  (139 mg kg<sup>-1</sup>), followed by suburban ( $Ca_{M3}$  1430 mg kg<sup>-1</sup>,  $Mg_{M3}$  87.5 mg kg<sup>-1</sup>) and rural ( $Ca_{M3}$  209 mg kg<sup>-1</sup>,  $Mg_{M3}$  19.8 mg kg<sup>-1</sup>) soils. However, the urban soils contained a significantly lower amount of  $Al_{ox}$  than the suburban and rural soils. Accordingly, soil pH was significantly higher in urban soils than in suburban and rural soils as a result of significant Ca accumulation. In general, soil depth had a weaker effect on P-retentive soil properties relative to the urban–rural gradient. Only SOC and  $Mg_{M3}$  significantly differed among soil depths, in which the 0–5 cm depth had the highest contents of SOC and  $Mg_{M3}$ .



**Figure 3.** Spearman correlations between labile P (sum of water-extractable P and KCl-extractable P) and (**A**) NaOH-extractable inorganic P (NaOH-Pi), (**B**) HCl-extractable P (P<sub>HCl</sub>), (**C**) residual P, and (**D**) total P.

### 3.3. Relationships between P Fractions and P-Retentive Soil Properties

Phosphorus fractions and associated soil properties from 30 sites (total 90 samples) were subjected to RDA to reveal the crucial parameters regulating the variations in P fractions along the urban–rural gradient or among different soil depths. Two principal components were extracted; the first principal component explained 80.66% of the total variation and was mainly correlated with  $P_{HCl}$ , pH, and  $Ca_{M3}$ , and the second principal component accounted for 16.13% of the total variance and was mainly correlated with  $Mg_{M3}$  (Figure 4). Therefore, pH and  $Ca_{M3}$  were the crucial properties responsible for the variations in P fractions along the urban–rural gradient or among different soil depths. In addition, soil pH and  $P_{HCl}$  were significantly (p < 0.01) increased by  $Ca_{M3}$  in all the samples, especially when the  $Ca_{M3}$  content was higher than 300 mg kg<sup>-1</sup> (Figure 5).

Location	Depth	pН	SOC	Alox	Feox	Ca <sub>M3</sub>	Mg <sub>M3</sub>	Clay
			g kg <sup>-1</sup>		mg	kg <sup>-1</sup>		g kg <sup>-1</sup>
Urban	5	6.25	18.51	1001	2261	2035	160	205
	15	6.95	11.96	1356	2594	2806	155	223
	30	6.72	11.25	1559	2946	2351	103	205
Suburban	5	5.99	20.95	1256	2168	1732	116	157
	15	6.02	10.23	1243	1995	1481	86	213
	30	6.06	7.15	1270	1873	1077	60	239
Rural	5	4.65	12.30	1673	2248	279	28	240
	15	4.60	8.11	1720	2324	174	16	250
	30	4.56	6.90	1761	2505	175	16	202

Table 2. Soil properties associated with P retention in response to the urban–rural gradient or soil depth.

ANOVA							
Location	< 0.001	0.009	< 0.001	0.02	< 0.001	< 0.001	NS
Depth	NS	< 0.001	NS	NS	NS	0.003	NS
Location × Depth	NS	NS	NS	NS	NS	NS	NS

SOC, soil organic carbon;  $Al_{ox}$ , ammonium oxalate-extractable Al;  $Fe_{ox}$ , ammonium oxalate-extractable Fe;  $Ca_{M3}$ , Mehlich 3-extractable Ca;  $Mg_{M3}$ , Mehlich 3-extractable Mg. NS, not significant.



**Figure 4.** Biplot of P fractions with associated soil properties from redundancy analysis (RDA) for the 90 soil samples from the city of Nanchang and the surrounding vicinity. The percentage variation explained by each factor is presented in parentheses.



**Figure 5.** Relationships between Mehlich 3-extractable Ca ( $Ca_{M3}$ ) and (**A**) soil pH and (**B**) HCl-extractable P ( $P_{HCl}$ ).

#### 4. Discussion

We found that urban soils had significant P accumulation compared with suburban and rural soils. Similarly, some previous studies have also reported that P was enriched in urban soils of a number of cities, such as Nanjing [11], Hangzhou [15], Beijing [31], and Nanchang [20] in China and London [32] in the UK. Generally, P enrichment in urban soils increases with the urban development process, including the population size and level of urban infrastructure [33]. The source and influx of P to urban soil were not investigated in our study. In general, solid waste and waste water are the main P-containing materials in urban ecosystems [34]. Some urban soils historically functioned as waste water and household treatment sites unintentionally, especially those with long urban-use histories, resulting in P enrichment in these soils. In addition, management practices in urban parks selected in our study (e.g., fertilization and irrigation) can result in P in urban soil.

Similar to the suburban and rural soils, NaOH-extractable P was a major P form in the urban soils. In addition, NaOH-Pi was significantly correlated with Feox contents for the three types of soils (r = 0.38, p < 0.01, data not shown). Since the natural soils in Southern China have a considerable amount of Fe and Al oxides [35], the parent materials and original soils appeared to control the major P fractions of the urban soils. Soil P<sub>HCl</sub> is usually present at a very low level or is absent in highly weathered acidic soils, as weathering mainly affects Ca or Mg P minerals. However, urban soils showed a significantly higher content of soil  $P_{HCl}$  (238 mg kg<sup>-1</sup>) than the other two types of soils, and the increases in P<sub>HCl</sub> contributed a major part of total P increases. Other studies also reported that urban soils had higher P<sub>HCl</sub> contents than soils in suburban and rural areas [15,20]. This is probably due to the inputs of materials with abundant Ca and/or Mg, as indicated by the higher content of Ca<sub>M3</sub> (Table 2). In fact, Ca<sub>M3</sub> accounted for 52% of the variations in the P<sub>HCl</sub> content across all soil samples and 72% of those of urban soils (Figure 5). The dissolution of calcareous materials in cement and concrete that are ubiquitous in built environments may elevate the Ca and/or Mg contents of urban soils [36,37] and further precipitate (or adsorb) P. Soil management in urban areas may also need to consider the enrichment of Ca, as some plant species that adapt to acidic soils may be sensitive to Ca enrichment.

Urban soils with relatively high total P contents may release more labile P, as labile P increased with the total P increase in urban soils. In addition, the strong relationships between labile P and NaOH-Pi,  $P_{HCl}$ , and residual P suggest that labile P that is lost from the soil via runoff, leaching or plant use may be replenished by these P pools. For example, soil  $P_{HCl}$  is probably not stable and may easily dissolve into soluble P due to soil acidification acceleration with N and acid deposition in urban ecosystems. Zhang et al. found groundwater P was significantly correlated with soil NaHCO<sub>3</sub>-extractable P in Nanjing city, suggesting that enriched P in urban soil has the potential to be released from the soil into the water system in urban areas [11]. Thus, P enrichment in urban soil may contribute partly to lake eutrophication in urban areas, which is usually caused by P enrichment of water [38].

Soil pH can regulate P reactions in soils, i.e., P sorption by Fe or Al oxides mainly occurs in acidic soils, whereas in neutral and alkaline soils, Ca or Mg can precipitate with P in the form of Ca- or Mg-P minerals [39]. The enrichment of Ca and Mg in urban soil along with increased soil pH suggested that P sorption characteristics may be changed in urban soils. Although Ca minerals can contribute to P sorption even in acidic soil [40], the enrichment of Ca (and Mg) in urban soil along with increased soil pH will decrease P sorption in urban soil, which in turn will facilitate P loss because P is held with greater bonding strength in acidic soil than in neutral or calcareous soils [39]. Further studies on the export flux of P from urban soils to adjacent ecosystems will advance our understanding of the consequences of P enrichment in urban soils.

# 5. Conclusions

A phosphorus fractionation scheme revealed that NaOH-extractable P (NaOH-Pi and NaOH-Po) and residual P were the dominant components of total P in urban soils, which is similar to rural and suburban soils. However, urbanization increased the contents of soil total P, P<sub>H2O</sub>, P<sub>KCI</sub>, NaOH-Pi,

residual P, and especially  $P_{HCl}$ . Soil pH and the  $Ca_{M3}$  content were the crucial properties responsible for the variations in P fractions along the urban–rural gradient or among different soil depths. In other words, the enrichment of Ca and/or Mg materials in urban soils, thought to be impacted by human activities, changed the soil P forms in the urban areas. Our study highlights the urgent need to extend the evaluation of P accumulation in urban soils and the associated environmental consequences to other cities and countries, especially those with long habitation histories.

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