Phosphatase activities and available nutrients in soil aggregates affected by straw returning to a calcareous soil under the maize–wheat cropping system

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The objective of this study was to investigate the effects of different rates of straw returning on soil aggregate stability, phosphatase activities, and the available nitrogen (N) and phosphorus (P) within different soil aggregate sizes. The experiment included five treatments: 1) no straw returning and no chemical fertilizer, 2) chemical fertilizer only (150 kg N ha^{-1}, 75 kg P ha^{-1}, and 75 kg K ha^{-1}), 3) 20% straw returning with chemical fertilizer, 4) 60% straw returning with chemical fertilizer, and 5) 100% straw returning with chemical fertilizer. Soil samples were collected 3.5 years after the start of the experiment and separated into four aggregate sizes (<0.25 mm, 0.25–1 mm, 1–2 mm, and 2–7 mm) using the dry sieving method. Soil acid phosphomonoesterase (AcP) and alkaline phosphomonoesterase (AlP); phosphodiesterase (PD); pyrophosphatase (PrA) activities; and soil NO_3^-–N, NH_4^+–N, and resin-P were determined within soil aggregates. The results showed that straw returning rates did not significantly impact soil aggregate distribution. However, straw returning increased soil AcP, AlP, and PD in <2 mm aggregates, and high rates of straw returning led to high enzyme activities. Soil phosphatase activities were also higher in 1–2 mm aggregates. All straw returning and chemical fertilization treatments increased soil NO_3^-–N and resin-P concentrations but had much less effect on soil NH_4^+–N concentrations. Additionally, the study revealed that soil pH, the concentrations of NH_4^+–N, NO_3^-–N, resin-P, and $CaCO_3$ significantly influenced soil phosphatase activities, but their impact varied across different sizes of aggregates.

1 Introduction

The intensive winter wheat–summer maize cropping system in the central North China Plain is known for high inputs of chemical fertilizers, inadequate organic matter, high crop yields, and large amounts of crop residues (Zhang et al., 2016; Lu et al., 2020). Traditionally, both wheat and maize straws were used as fuel for cooking or burned on the farm to clean the field for planting the next crop. Since the on-farm straw burning practice was banned in 2008, a large quantity of straw must be incorporated into the soil. Straw returning has been shown to increase soil organic matter (SOM) and available soil nutrients (Tan et al., 2015), improve soil porosity and structure (Yao et al., 2015), reduce soil bulk density, and enhance soil enzyme activities (Wang et al., 2018). However, the straw return was challenging due to the lack of appropriate field equipment in some hilly areas, where straws are currently removed from fields. As a consequence, intensive farming has led to soil quality degradation (i.e., lack of SOM, low soil fertility, poor nutrient use, subsequently low yield, and degraded soil structure) (Liu and Diamond, 2005), which greatly influences the long-term productive capacity of the soil (Vitousek et al., 2019).

Soil aggregates are vital for soil structure and fertility (Mrquez et al., 2019), and the associated soil characteristics within and between aggregates are essential for soil quality (Lehmann et al., 2017). Straw returning has been shown to increase the proportion of soil macroaggregates larger than 0.25 mm (Alidad et al., 2012; Zhang et al., 2018; Zhao et al., 2018; Cao et al., 2021). Additionally, the distribution of soil aggregates and the mean weight diameter (MWD) also differed from the straw returning mode (Ma et al., 2020). However, the effects of straw returning on phosphorus (P) distribution among soil aggregates remain unclear. For instance, soil P could be enriched in soil macroaggregates or microaggregates due to different soil types (Ahmed et al., 2017). Studies have shown that 80% of soil total P was contained in soil macroaggregates (>2 mm) (Zhang et al., 2021), while soil total P or available P concentrations were highest in <0.053 mm soil aggregates (Cheng et al., 2019; Deng et al., 2021). Additionally, different sizes of soil aggregates also affect P loss from soil (Garland et al., 2018; Li et al., 2020; Cao et al., 2021). Therefore, the input of exogenous organic materials can potentially impact the structure of soil aggregates and P distribution among aggregates.

Phosphatase enzymes are responsible for mineralization of organic matter to release phosphate ions ($H_2PO_4^-$ and HPO_4^{2-}) in soil (Criquet and Braud, 2008). Soil acid phosphomonoesterase (AcP), alkaline phosphomonoesterase (AlP), phosphodiesterase (PD), and pyrophosphatase (PrA) are specific enzymes that facilitate this process. Studies have shown that straw returning significantly increased soil alkaline phosphatase activity (Wang et al., 2018), and phosphatase activity decreased with decreasing aggregate size (Tian et al., 2022), but the effect of straw returning on soil phosphatase activities may not be long term (Stegarescu et al., 2021). Furthermore, the turnover of nutrients is closely related to soil aggregates and soil structure (Six et al., 2000; Galantini et al., 2004) because soil aggregates can provide physical protection for nutrients from microbial decomposition (Six

et al., 2004; Barthès et al., 2008), and the availability of nutrients would conversely influence phosphatase activities. Thus, straw returning may cause a difference in soil phosphatase activities and P availability among soil aggregates.

The existing research on soil aggregates following straw returning to the field has mainly focused on water-stable aggregates (Meng et al., 2014; Garbuz et al., 2016). Although studies have primarily centered on soil carbon status among aggregates (Wang et al., 2015; He et al., 2018; Wang et al., 2018), few have investigated the concentration of available P and phosphatase activities after straw returning to the field. The study was conducted in a typical Yimeng mountainous area in Northern China, where cinnamon soil (Calcaric Cambisol, FAO) accounts for up to 50% of the land area. Straw returning is not common practice in this area, and the soils often suffer from erosion and low P nutrients. The objective of this study was to determine the effects of straw returning on the structure and internal available P and other nutrients and phosphatase activities of soil aggregates in a calcareous soil under a maize–wheat cropping system.

2 Materials and methods

2.1 Site description and experimental design

The field experiment was conducted on cinnamon soil (Calcaric Cambisols in FAO classification) at Fei County (117°54′ E and 35°13′ N), Shandong Province, Northern China. The region has a temperate continental monsoon climate; the annual mean temperature and precipitation are 14.1°C and 849 mm, respectively; and 60%–80% of the annual precipitation occurs during the maize-growing season in summer. The initial soil properties in the study area were 8.05 pH, 1.12% TC, 0.16% TN, and 548 mg kg^{-1} TP. Winter wheat was planted in early or mid-October with a seeder and harvested in early June of the following year. Then, summer maize was planted after the wheat harvest and harvested in early October. Five treatments were as follows: 1) CK (control - no fertilizer and no straw), 2) C (chemical fertilizer only), 3) SR20 (20% straw returning with chemical fertilizer), 4) SR60 (60% straw returning with chemical fertilizer), and 5) SR100 (100% amount of straw returning with chemical fertilizer). The chemical fertilizer treatment consisted of 150 kg N ha^{-1} urea and with about 5.48 kg N from $NH_4H_2PO_4$, 45 kg P ha^{-1} as $NH_4H_2PO_4$, and 45 kg K ha^{-1} as KCl. Each plot was 3 m × 4 m in size with a 1-m buffer zone between plots. All treatments were replicated four times, except CK (three times due to limited experimental area), and plots were arranged according to a single-factor, completely randomized experiment design. Prior to the initiation of this experiment, no straw returning was performed to the soil in this field previously. For this study, only maize straw was returned to the field, and the amount of the maize straw that was returned based on the average straw weight (8,700 kg/ha dry weight) in the field. The nutrient contents of straws returned to the fields were 687 g TC kg^{-1}, 9.16 g TN kg^{-1}, and 2.83 g TP kg^{-1}. Maize straw was chopped into 1-cm-long pieces and disked into 0–20 cm soil layer.

2.2 Soil sampling

Intact soil core samples were collected from each plot by pushing a plastic frame (10 cm long, 10 cm wide, and 20 cm deep) into the soil after the wheat harvest, 3.5 years after the initiation of the experiment. After being transported to the laboratory, the soil cores were gently broken into small clods according to the natural texture and dried at 4 °C in a refrigerated cabinet until the soil water content was around 8%. After being sieved through a 7-mm sieve, soil aggregates were measured with the dry screening method (Zhang et al., 2013). The dry sieving method was chosen in our study as aggregates divided by the wet-sieving method can hardly be used for the determination of soil nutrient contents and soil phosphatase activities. A sub-sample was sieved (<2 mm) and stored at 4 °C for analysis of enzyme activities within a week. Another subsample was air-dried, sieved, and analyzed for basic soil properties.

2.3 Soil aggregate fractionation measurement

Cold air-dried soil samples (100 g) were agitated by a mechanical shaker (OA SS203, Ortoalresa, Spain) at 50 Hz frequency for 2 min on a stack of sieves (2–7 mm, 1–2 mm, 0.25–1 mm, and <0.25 mm) until soil aggregate samples were enough for further analysis. The aforementioned procedure was repeated several times. All soil aggregate samples were weighed and stored at 4 °C until further analysis. The MWD of soil aggregates was calculated using the following formula (Kemper and Rosenau, 1986):

$$MWD = \sum_{i=1}^{n} X_i W_i.$$

where X_i is the mean diameter (mm) and W_i is the weight proportion of each size fraction.

2.4 Soil properties

Soil pH was measured using a glass electrode (soil/water ratio of 1.0:2.5). Soil carbonate content was measured with a sieved air-dried sample (<100 mesh) using the pressure calcimeter method: 1.0 g of soil and 2 mL of 6 M HCl containing 3% by weight of $FeCl_2 \cdot 4H_2O$ are placed at the bottom of a 100-mL sealing reaction vessel; the acid was mixed with soil by turning the vessel sideways; an 18-gauge hypodermic needle was inserted, which is attached to the pressure transducer and voltage meter, the results were recorded; and then CO_2 concentration was calculated using a calibration curve to obtain carbonate content (Loeppert and Suarez, 1996). Soil organic carbon (SOC) content was measured with sieved air-dried soil (<100 mesh)) using the dichromate oxidation method, and SOM content was calculated based on OM = 1.724 × SOC (Nelson and Sommers, 1996).

Soil available nitrogen (N) and P concentrations were determined for all grades of soil aggregates. Ammonium N and nitrate N concentrations were determined by the colorimetric method after being extracted with 1: 5 2 M KCl solutions (Mulvaney, 1996). The content of available P in soil was determined by the anionic exchange resin strips combined with molybdenum blue method, and the blue color was detected at 700 nm using the Agilent Cary 100 UV–Vis spectrophotometer (Sharpley, 2000).

2.5 Soil phosphatase assay

Soil acid phosphomonoesterase activities (EC 3.1.3.2, AcP), alkaline phosphomonoesterase activities (EC 3.1.3.1, AlP), and the phosphodiesterase activities (PD) were determined by the method of Tabatabai et al. (1994). Briefly, soil acid phosphomonoesterase activities were determined as follows: a fresh soil sample (1.0 g) was mixed with 1 mL of 50 mM disodium phenyl phosphate in 4 mL of modified universal buffer at pH 6.5 and 0.2 mL of toluene before incubating for 1 h at 37 °C. The alkaline phosphomonoesterase activities were determined using the same procedure, except the universal buffer pH was adjusted to 11. Universal buffer was prepared by mixing 12.1 g of Tris (hydroxymethyl) aminomethane, 11.6 g of succinic acid, 14.0 g of citric acid, and 6.3 g of boric acid in 488 mL of sodium hydroxide solution [C (NaOH) = 1 mol L^{-1}], then diluting to 1 L, and storing at low temperature for future use. Modified universal buffer can be obtained by diluting the universal buffer from 200 mL to 1 L and modifying it to the needed pH of 6.5 for AcP and 11.0 for AlP. After incubation, 1 mL 0.5 mol L^{-1} $CaCl_2$ and 4 mL mol L^{-1} NaOH were added to terminate the reaction. Phosphodiesterase activities (PD) were determined by incubating 1.0 g fresh soil with 1 mL of 50 mM sodium bis (p-nitrophenyl) phosphate and 4 mL of pH 8.0 buffer prepared by dissolving 6.1 g of Tris-base, adjusting to pH 8.0 with sulfuric acid solution, and then fixing the volume to 1 L. To stop phosphodiesterase activities, 1 mL 0.5 mol L^{-1} $CaCl_2$ and 4 mL of $CaCl_2$-Tris (hydroxymethyl aminomethane) were used. Fluorescence produced by the aforementioned enzymatic reactions was measured colorimetrically at 410 nm using the Agilent Cary 100 UV–Vis spectrophotometer. For the aforementioned enzyme assays, controls were included for each soil sample. Pyrophosphatase activities (EC 3.6.1.1, PrA) were also assayed following the method of Tabatabai et al. (1994). Fresh soil (1.0 g) was mixed with 3 mL of 50 mM sodium pyrophosphate, and after incubating for 1 h at 37 °C, 25 mL of 0.5 M H_2SO_4 and 3 mL of pH 8.0 modified universal buffer (the same universal buffer as phosphomonoester activities modified with pH 8.0) were added and shook using a horizontal oscillator for 3 min, and then centrifuged to obtain the supernatant. About 2 mL of the supernatants was taken in 50-mL volumetric flasks, the developers were added, the volume fixed, and the color compared at 700 nm using the UV–Vis spectrophotometer (Agilent, Santa Clara, CA) after 15 min of color development.

2.6 Statistical analyses

The differences among treatments or grades were tested by one-way ANOVA using SPSS 16.0 (SPSS Inc., Chicago, Ill., United States). Mean separation was determined using Tukey's or Dunnett's T3 test based on the results of

TABLE 1 Effects of different rates of maize straw returning on soil chemical properties, aggregate distribution, and MWD.

Treatment	pH	SOM	CaCO₃	Soil aggregate distribution (%)				MWD
		g kg⁻¹	g kg⁻¹	2–7 mm	1–2 mm	0.25–1 mm	<0.25 mm	(mm)
CK	7.98 ± 0.11a	16.8 ± 0.77 ab	30.1 ± 1.31a	30.6 ± 1.13aAB	27.6 ± 1.00aB	33.2 ± 1.37aA	8.59 ± 1.41aC	2.02 ± 0.07a
C	7.77 ± 0.17a	16.0 ± 0.15 b	27.0 ± 1.42a	32.5 ± 0.80aA	26.9 ± 0.79aB	34.5 ± 0.97aA	6.05 ± 0.68aC	2.09 ± 0.03a
SR20	8.03 ± 0.08a	19.1 ± 0.70 ab	27.3 ± 0.87a	28.4 ± 1.80aB	28.4 ± 0.53aB	35.7 ± 1.61aA	7.55 ± 1.31aC	1.94 ± 0.07a
SR60	7.97 ± 0.05a	18.7 ± 0.85 ab	12.6 ± 0.53b	32.2 ± 0.57aA	27.8 ± 0.67aB	34.6 ± 0.60aA	5.37 ± 0.79aC	2.09 ± 0.02a
SR100	7.68 ± 0.04a	19.1 ± 0.89 a	13.2 ± 0.44b	31.8 ± 0.80aA	27.1 ± 0.36aB	34.6 ± 1.01aA	6.46 ± 0.38aC	2.06 ± 0.03a

Note: Values are shown as mean ± standard error. Different lowercase letters represent the difference between the different treatments within the same aggregate size, and the different capital letters indicate differences in particle size distribution within the same treatment. Treatments were CK (no chemical fertilizer and no straw), C (chemical fertilizer), SR20 (20% straw returning with chemical fertilizer), SR60 (60% straw returning with chemical fertilizer only), and SR100 (100% full amount of straw returning with chemical fertilizer).

homogeneity of variances (p-value < 0.05). A detrended correspondence analysis (DCA) was applied, and the lengths of the gradients were less than 3. Then, a redundancy analysis (RDA) was used to identify the effects of soil properties on soil phosphatase activities among treatments and soil aggregates using Canoco Software 4.5 (Microcomputer Power, Ithaca, NY, United States). The soil factors were standardized by error variance when RDA was performed. All soil properties included in the RDA analysis were D standardized as Z-scores to remove the unit's influence. Soil phosphatase activity scores were "divided by standard deviation," soil phosphatase activity data chose the "do not transform" item, soil samples were "standardized by norm," and the phosphatase activities were "standardized by error variance" when RDA was carried out. The Monte Carlo permutation test (999 permutations) was used to select factors that significantly influenced soil phosphatase activities. Figures were prepared using SigmaPlot 10 (SYSTAT, Point Richmond, CA, United States).

3 Results

3.1 Effect of different rates of maize straw returning on soil pH, SOM, carbonate, and soil aggregate distribution

Compared to the use of chemical fertilizer alone, the maximum straw returning treatment (SR100) significantly increased SOM by 19% and reduced carbonate concentration by 51% (Table 1). The second highest straw returning treatment (SR60) significantly reduced carbonate concentration but did not significantly increase SOM. The low rate of straw returning (SR20) neither significantly affected the contents of organic matter nor those of carbonate. In addition, no significant changes in soil pH were found among treatments.

The soil aggregates in the CK sample were almost equally distributed among small macroaggregates (33.2%, 0.25–1 mm), medium macroaggregates (27.6%, 1–2 mm), and large macroaggregates (30.6%, 2–7 mm); the microaggregates (<0.25 mm) were relatively small (8.6%). As for 2–7 mm aggregates, results showed that SR20 treatment significantly

decreased the proportion of 2–7 mm aggregates compared to other treatments. Except for that, no significant differences were observed among treatments within the same size of aggregate. In addition, different treatments did not affect the soil MWD.

3.2 Effects of straw returning rate on the available N, P concentration among soil aggregates

All sizes of soil aggregates under CK treatment had significantly lower NO_3^-–N concentrations than those of other treatments, while medium macroaggregates (1–2 mm) showed significantly higher NO_3^-–N concentrations than those of other sizes of aggregates under the same treatments (Figure 1A). The high concentrations of NH_4^+–N were found in microaggregates (<0.25 mm), while the low concentrations were from medium macroaggregates (1–2 mm) for all treatments (Figure 1B). The NH_4^+–N concentration in <0.25 mm soil aggregates showed that SR100 treatment had the highest and SR20 treatment had the lowest NH_4^+–N contents, significantly different with C and CK treatments. For 0.25–1 mm aggregates, only the SR100 treatment had significantly higher NH_4^+–N contents than the other treatments. No significant difference was observed for NH_4^+–N contents associated with 1–2 mm aggregates among treatments. For 2–7 mm aggregates, the CK sample had the highest NH_4^+–N contents, which were significantly higher than those of the SR20 and SR100 treatments. Under the same treatment, NH_4^+–N contents ordinarily followed the sequence from high to low as < 0.25 mm > 2–7 mm > 0.25–1 mm > 1–2 mm. Only the SR20 treatment showed that NH_4^+–N was significantly high in 2–7 mm, followed by < 0.25 mm and 1–2 mm, and at last, 1–2 mm.

The order of resin-P concentration was <0.25 mm aggregates >0.25–1 mm aggregates > 1–2 mm aggregates = 2–7 mm aggregates under CK, C, SR20, and SR100 treatments (Figure 1C). Only SR60 treatment showed that resin-P in 2–7 mm aggregates was significantly higher than that in 1–2 mm aggregates. Within the same aggregate, no clear trend was shown among treatments, but resin-P followed the order of SR100 > SR60 > SR20 > C > CK from high to low in
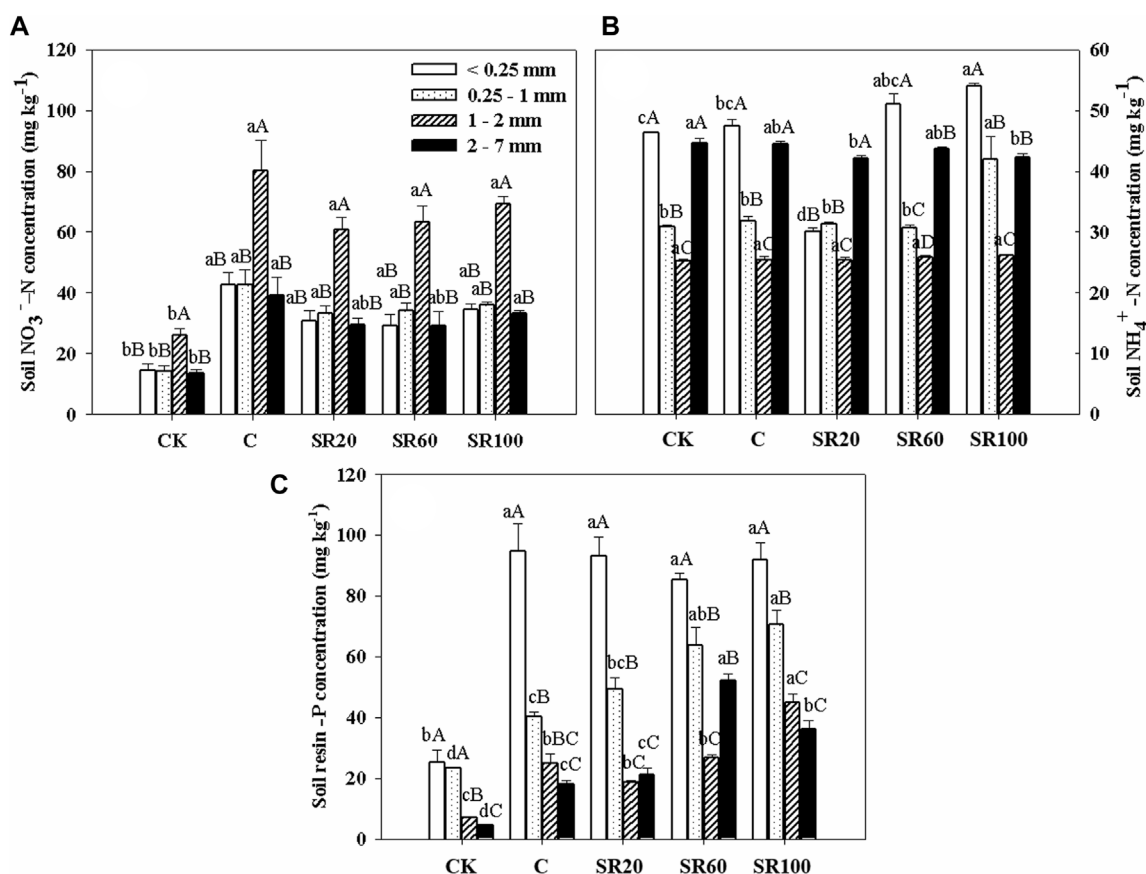
FIGURE 1
Soil aggregate-associated available NO$_3^-$−N **(A)**, NH$_4^+$−N **(B)**, and phosphorus **(C)** under different treatments [CK (no chemical fertilizer and no straw), C (chemical fertilizer only), SR20 (20% straw returning with chemical fertilizer), SR60 (60% straw returning with chemical fertilizer), and SR100 (100% full amount of straw returning with chemical fertilizer)]. Values are shown as the mean ± standard error. Different lowercase letters represent the difference among different treatments within the same aggregate size, and different capital letters indicate the difference among the grain levels of the same processing different aggregates.

0.25–1 mm and 1–2 mm aggregates. Furthermore, the higher returning rate of straw had positive effects on soil resin-P concentration.

3.3 Effects of maize straw returning rate on soil aggregate-associated AcP, AlP, PD, and PrA

As shown in Figure 2A, AcP in <0.25 mm microaggregates under CK and SR20 treatments was significantly lower than those under SR100 and SR60 treatments. Within 0.25–1 mm aggregates, only SR100 treatment significantly increased AcP. As for 1–2 mm aggregates, SR20 treatment had the lowest AcP, which was significantly lower than that in SR100. For AcP in 2–7 mm aggregates, there was no significant difference among treatments. As seen from CK treatment, 1–2 mm aggregates showed the highest AcP, whereas SR60 treatment showed the highest AcP in both 1–2 mm and <0.25 mm aggregates. The rest of the treatments showed that <0.25 mm aggregates had the highest AcP. All

treatments showed that aggregates of 0.25–1 mm had the lowest AcP.

Soil AlP was higher in 1–2 mm aggregates than in 2–7 mm aggregates under C and CK treatments (Figure 2B). Straw returning increased soil AlP associated with 1–2 mm aggregates, while only SR100 and SR60 treatments increased AlP associated with 0.25–1 mm and 2–7 mm aggregates. As for AlP within <0.25 mm aggregates, only SR100 had higher AlP than CK and C treatments. The soil AlP associated with 1–2 mm and 0.25–1 mm aggregates were higher than that associated with <0.25 mm and 2–7 mm aggregates under SR100, SR60, and SR20 treatments, while soil AlP within 1–2 mm aggregates were even more higher than within 0.25–1 mm aggregates under SR60 treatment. Furthermore, soil AlP in 1–2 mm aggregates was only higher than that in 2–7 mm aggregates under CK and C treatments.

As shown in Figure 2C, PD of <0.25 mm, 0.25–1 mm, and 1–2 mm aggregates under SR100 treatment was significantly higher than that under C, CK, and SR20 treatments. Soil PD in 2–7 mm aggregates under SR100 treatment was significantly higher than that under C treatment, and there was no significant difference
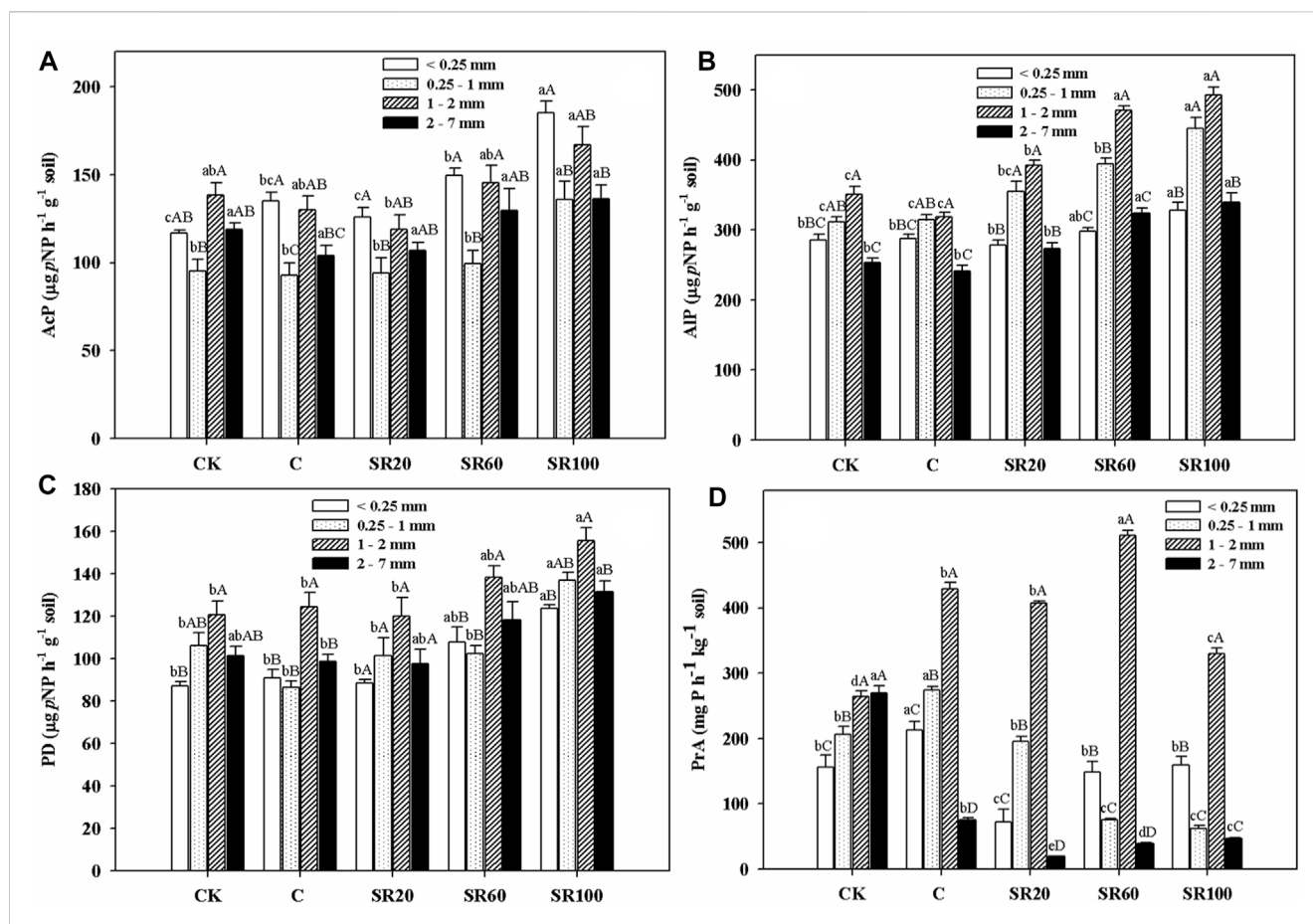
FIGURE 2
Soil aggregate-associated AcP **(A)**, AlP **(B)**, PD **(C)** and PrA **(D)** under different treatments [CK (no chemical fertilizer and no straw), C (chemical fertilizer only), SR20 (20% straw returning with chemical fertilizer), SR60 (60% straw returning with chemical fertilizer), and SR100 (100% full amount of straw returning with chemical fertilizer)]. Values are shown as mean $\pm$ standard error. Different lowercase letters represent the difference among the different treatments within the same aggregate size, and the different capital letters indicate the difference among the grain levels of the same processing different aggregates.

compared with other treatments. Considering the same treatment, PD under SR20 treatment had no significant difference among aggregates. Under other four treatments, 1–2 mm aggregates had higher PD than other aggregates, and <0.25 mm or 0.25–1 mm showed the lowest value.
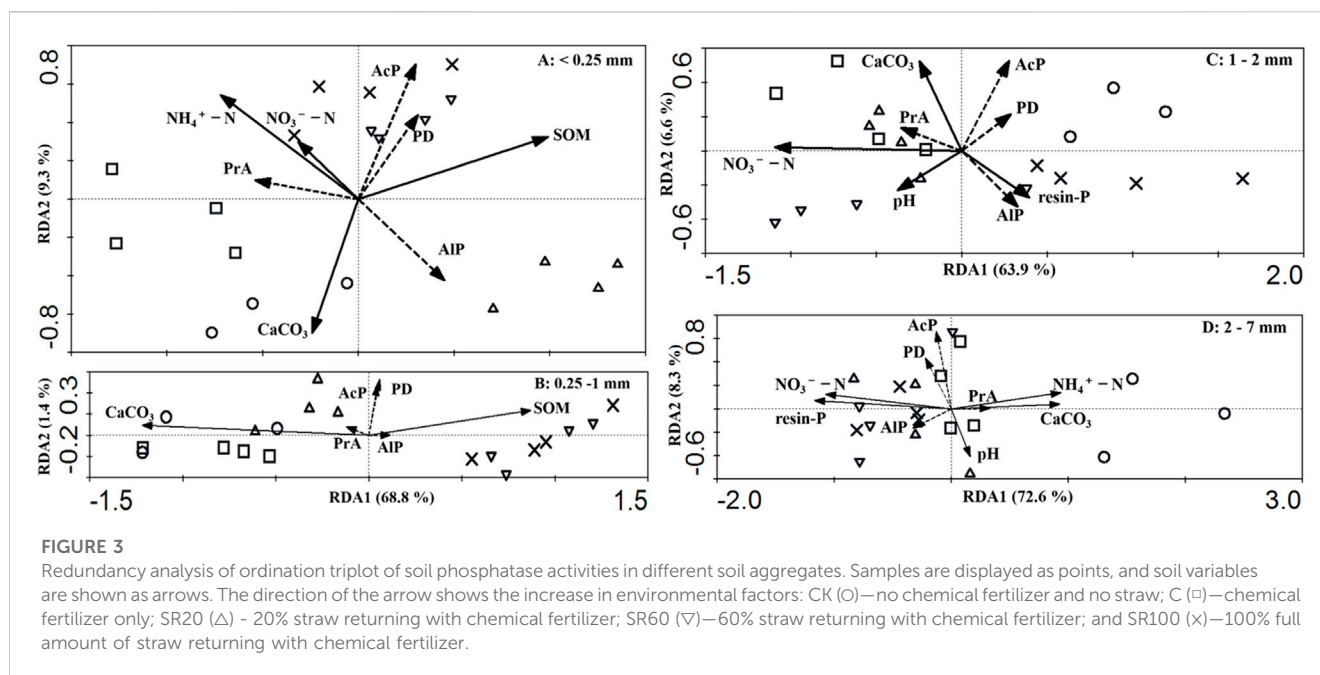
Unlike other soil phosphatase activities, soil PrA was significantly affected by treatments and among aggregates (Figure 2D). Soil PrA associated with <0.25 mm and 0.25–1 mm aggregates was significantly higher under C treatment than under other treatments. Soil PrA in 0.25–1 mm aggregates was highest under SR60 treatment, followed by C and SR20 treatments, and then SR100, and CK treatments. It is worth noting that CK treatment had the highest PrA in 2–7 mm aggregates, followed by C, SR100, SR60, and SR20 treatments. Under the same treatment, soil macroaggregates had higher PrA than microaggregates.

3.4 Multivariate analysis of soil phosphatase activities and soil properties

Redundancy analysis was conducted to investigate the relationships between soil phosphatase activities in different soil aggregates. In aggregates <0.25 mm, soil phosphatase activities were significantly related to soil available nitrogen (N) concentration, soil organic matter (SOM), and carbonate contents (Figure 3A). The soil properties explained 77.5% of the variation in soil phosphatase activities (F = 12.3, P = 0.001), with the first (RDA1) and second (RDA2) axes explaining 57.0% and 66.3% of the accumulated variation in soil phosphatase activities, respectively. The RDA1 explained 73.6% of the variation in the relationship between the soil phosphatase activities and soil properties (F = 18.1, P = 0.001), while the RDA2 explained 98.4% of the accumulated variation (F = 20.6, P = 0.001).

In aggregates 0.25–1 mm, soil phosphatase activities were significantly related to SOM and carbonate contents (Figure 3B). The soil properties explained 70.2% of the variation in soil phosphatase activities (F = 21.5, P = 0.001), with the first (RDA1) and second axes (RDA2) explaining 68.8% and 70.2% of the accumulated variation in soil phosphatase activities, respectively. The RDA1 explained 98% of the variation in the relationship between the soil phosphatase activities and soil properties (F = 40.6, P = 0.001), while the RDA2 explained 100% of the accumulated variation (F = 2.09, P = 0.312).

FIGURE 3
Redundancy analysis of ordination triplot of soil phosphatase activities in different soil aggregates. Samples are displayed as points, and soil variables are shown as arrows. The direction of the arrow shows the increase in environmental factors: CK (○)—no chemical fertilizer and no straw; C (□)—chemical fertilizer only; SR20 (△) - 20% straw returning with chemical fertilizer; SR60 (▽)—60% straw returning with chemical fertilizer; and SR100 (×)—100% full amount of straw returning with chemical fertilizer.

In 1–2 mm aggregates, soil phosphatase activities were significantly related to NO_3^-–N, resin-P, $CaCO_3$, and pH (Figure 3C). The soil properties explained 73.4% of the variation in soil phosphatase activities (F = 9.68, P = 0.001), with the RDA1 and RDA2 axes explaining 63.9% and 70.5% of the accumulated variation in soil phosphatase activities, respectively. The RDA1 explained 87.1% of the variation in the relationship between the soil phosphatase activities and soil properties (F = 24.9, P = 0.001), while the RDA2 explained 96% of the accumulated variation (F = 6.28, P = 0.111).

In 2–7 mm aggregates, soil phosphatase activities were significantly related to NO_3^-–N, resin-P, $CaCO_3$ contents, and pH (Figure 3D). The soil properties explained 81.3% of the variation in soil phosphatase activities (F = 10.5, P = 0.001), with the RDA1 and RDA2 explaining 72.6% and 80.9% of the accumulated variation in soil phosphatase activities, respectively. The RDA1 explained 89.3% of the variation in the relationship between the soil phosphatase activities and soil properties (F = 29.9, P = 0.001), while the RDA2 explained 99.4% of the accumulated variation in the aforementioned relationship (F = 11.5, P = 0.001).

4 Discussion

4.1 Soil pH, SOM, and carbonate concentration

Unlike previous studies that found returning straw could decrease (Sahrawat, 2005; Bai et al., 2013; Cheng et al., 2023) or increase (Wang et al., 2013) soil pH, this study showed no significant change in soil pH during 3.5 years of the experiment. This could be attributed to the high soil-buffering capacity due to the high concentration of $CaCO_3$ in the soil (Wang et al., 2013; Zamanian and Kuzyakov, 2019). Straw returning treatments with high

percentages (60% and 100%) led to a reduction of approximately 50% in carbonate concentration within 3.5 years. Continuation of the straw returning practice might eventually neutralize all or most carbonate, leading to a reduction in soil pH. The increase in soil organic matter (SOM) resulting from straw returning may take longer and require larger amounts of straw. Although many studies reported an increase in SOM (0–20 cm soil depth) due to straw returning (Ma et al., 2020), significant results may take several years to emerge, as reported by Zhao et al. (2018) who observed a positive influence on SOC after 7 years. Chemical fertilization, mainly with N inputs, might cause reduction in SOM (Zhao et al., 2018), likely the same situation as observed under the C treatment. Additionally, straw returning can lead to priming effects, increasing microbial activity and causing SOM depletion (Fang et al., 2018). The decrease in $CaCO_3$ content in SR60 and SR100 can be attributed to two reasons. First, organic acids released during the decomposition of the high rate of straw residue (Sahrawat, 2005; Cao et al., 2021) through root exudates and microbial respiration (Sahrawat, 2005; Wang et al., 2013) can contribute to the decrease. Second, the high rate of straw returning treatments can improve soil water permeability (Alidad et al., 2012; Gorokhova and Chursin, 2021), and the presence of H_2O and CO_2 could accelerate $CaCO_3$ dissolution (Gorokhova and Chursin, 2021).

4.2 Soil aggregate distribution and associated available N and P concentrations

The mean weight diameter (MWD), an important index for assessing soil aggregate size distribution, can reveal the physical structure of soil and reflect soil quality (Ma et al., 2020). Previous studies have shown that straw returning increased MWD (Huang et al., 2017; Ma et al., 2020; Cao et al., 2021), but we found no significant change in MWD in our study, likely due to the use of the

dry sieving method, which differs from the wet sieving method used in previous studies. As seen in Table 1, 2–7 mm soil aggregates were increased by the C, SR60, and SR100 treatments, but not the SR20 treatment. High rates of straw returning increased the proportion of >2 mm soil aggregates, consistent with previous reports (Wang et al., 2015; Ma et al., 2020), showing that fresh SOC can stimulate microbial activity to produce soil binders (Six et al., 2000), so it is easy for soil aggregate agglomeration. The C treatment also increased the proportion of >2 mm soil aggregates, which may be caused by the same reason, but the carbon source for microbial activity came from the depletion of soil original SOM. The low proportion of >2 mm soil aggregates under SR20 treatment showed high microbial activity but inadequate soil fresh carbon input.

The concentration of NO_3^-–N within all sizes of aggregates in C and straw returning treatments was higher than that in the CK treatment, likely due to the chemical fertilization of N (Nagatake et al., 2018). Furthermore, straw returning could increase soil cation exchange capacity (Cheng et al., 2023) and decrease N leaching in soil (Yang et al., 2016), which could also increase NO_3^- adsorption to soil aggregates and NO_3^-–N concentration. Our study also found that within the same treatment, NO_3^- was strongly associated with 1–2 mm soil aggregates.

Previous studies have suggested that straw returning could increase soil N availability due to N input from straw (Wang et al., 2018), immobilizing soil mineral N, and releasing N from straw decomposition (Takahashi et al., 2003; Thuy et al., 2008). However, few studies have investigated NO_3^-–N and NH_4^+–N distribution among different soil aggregates. We found that the SR100 treatment significantly increased NH_4^+–N concentration in <0.25 mm and 0.25–1 mm aggregates. The reasons might be that the SR100 treatment increased soil organic carbon content, which decreased NH_4^+ adsorption to the soil caused by reduced exchange sites for NH_4^+ (Zhang et al., 2022). All treatments showed that NH_4^+–N concentrations were higher in <0.25 mm and 2–7 mm aggregates but lower in 0.25–1 mm and 1–2 mm aggregates. Soil aggregates of <0.25 mm showed the highest NH_4^+–N concentration, likely due to their large surface area, which has been reported in previous studies to accumulate nutrients (Adesodun et al., 2007; Wang W. et al., 2011; Mitran et al., 2018). NH_4^+–N concentration was also high in 2–7 mm aggregates, possibly due to $CaCO_3$ content shown by RDA analysis (Figure 3D). $CaCO_3$ content has been reported to have positive effects on soil macroaggregates (Ge et al., 2019), which provide physical protection for soil organic carbon content from microbial decomposition (Wei et al., 2013), which was proved to be important for NH_4^+ retention in soil (Baldock and Nelson, 2000).

The application of chemical fertilizer alone or combined with straw returning was shown to increase soil available P due to P release from the fertilizer (Wang W. et al., 2011; Ahmed et al., 2017) or organic matter mineralization (Chen et al., 2018; Cheng et al., 2019; Cao et al., 2021). Previous studies reported that a high rate of straw returning has positive effects on soil available P in aggregates, indicating that more nutrients are contained in straw (Ma et al., 2020). This study found that resin-P concentrations generally increased as aggregate size decreased, which is consistent with previous findings and might be caused by the increasing surface area with decreasing size of aggregates (Mitran et al., 2018; Cheng et al., 2019).

4.3 Soil aggregate-associated phosphatase activities

Phosphatase activities play a crucial role in soil P availability. This study found that straw returning had positive treatment effects on soil acid phosphomonoesterase activities (AcP) in aggregates <2 mm and alkaline phosphomonoesterase activities (AlP) and phosphodiesterase activities (PD) in all aggregates. These findings were consistent with those of previous studies, which suggested that the increasing substrate availability caused by straw returning (Li et al., 2022), and cumulative effects on soil enzyme activities from annual straw incorporation, might contribute to the significant effects of a high rate of straw returning (Wang et al., 2018; Li et al., 2022). The high AcP, AlP, and PD in 1–2 mm aggregates indicate those aggregates provide a more suitable environment for microbial population, and soil enzymes due to the suitable environment probably had higher phosphomonoesterase and phosphodiesterase activities.

Pyrophosphatase in soil plays a role in catalyzing pyrophosphate to orthophosphate (Tabatabai et al., 1994). Pyrophosphatase activities (PrA) are negatively related to soil pH (Tabatabai and Dick, 1979), but no significance of soil pH was found in this study. The study also found that the activities of pyrophosphatase are closely related to substrate (pyrophosphate) content, which correlated positively with microbial P (Reitzel and Turner, 2014), especially with soil fungal communities (Bünemann et al., 2004; Makarov et al., 2005; Koukol et al., 2008). The previous studies showed that straw returning affects fungal communities, resulting in more fungal communities under medium and high levels of straw returning (Wang et al., 2021). The high activities of PrA in 1–2 mm aggregates under C, SR20, SR60, and SR100 treatments might show more fungal microbial biomass, which provided more pyrophosphate as substrates.

4.4 Relationships among straw returning, soil properties, soil aggregates, and soil phosphatase activities

Only four significant factors could influence <0.25 mm soil aggregates associated with phosphatase activities. Soil AcP, AlP, and PD positively related to soil organic matter, and PrA positively correlated with NH_4^+–N and NO_3^-–N concentration. Soil AcP, AlP, and PD positively related to soil organic carbon, which had been found previously (Saha et al., 2008; Wang J. B. et al., 2011; Wei et al., 2015) due to substrate effect and more favorable environmental factors for enzymes (Zhang et al., 2012; Zhang et al., 2016).

For 0.25–1 mm soil aggregates associated with phosphatase activities, only SOM and $CaCO_3$ contents significantly influenced enzyme activities. SOM positively influences AlP, PrA, and soil $CaCO_3$. Soil organic carbon increasing phosphatase activities were extensively reported (Tabatabai and Dick, 1979; Saha et al., 2008; Wang J. B. et al., 2011; Wei et al., 2015). Unlike the report of soil $CaCO_3$ negatively related to soil PrA (Tabatabai and Dick, 1979), this study found a positive relationship between $CaCO_3$ and PrA (also in 1–2 and 2–7 mm aggregates, Figures 3C, D). This was possible because the soil pH in this study still ranged in the optimum pH for soil PrA (Dick and Tabatabai, 1978), which covered the effect of $CaCO_3$.

Soil pH, NO_3^-–N, resin-P, and $CaCO_3$ were considered significant factors influencing soil phosphatase activities in 1–2 mm aggregates. Soil pH negatively correlated with soil AcP and PD, while soil AlP was attributed to soil resin-P concentration. Through the data analysis, the study found that alkaline phosphomonoesterase was a determining factor for P availability in 1–2 mm aggregates in calcareous soils and a high rate of straw returning had higher alkaline phosphatase activities.

The study found that soil pH, NH_4^+–N, NO_3^-–N, resin-P, and $CaCO_3$ are significant factors that influence soil phosphatase activities in 2–7 mm aggregates. Similar to those in 1–2 mm aggregates, soil pH negatively correlated with soil AcP and PD, while soil AlP positively related to soil resin-P and NO_3^-–N concentration. NH_4^+–N and $CaCO_3$ had a positive effect on soil PrA. Soil available nitrogen concentration positively correlated with soil PrA or soil AlP, except for 0.25–1 mm aggregates. This can be explained by soil biota secreting more enzymes to enhance the supply of N and P when soil nutrition is limited (Zhang et al., 2012). Soil pH was considered to have a negative effect on soil PrA, but the effect would be weak in neutral and alkaline soil (Tabatabai and Dick, 1979); thus, we found a slight influence between soil pH and PrA (Figures 3C, D). In addition, the soil pH in our study had no difference among treatments.

5 Conclusion

Straw returning with chemical fertilizer could increase soil phosphatase activities, and a high rate of straw returning treatment could be more desirable. The phosphatase activities within <0.25 mm, 0.25–1 mm, 1–2 mm, and 2–7 mm aggregates were influenced differently by soil properties under different treatments, with significant factors including soil pH, NH_4^+–N, NO_3^-–N, resin-P, SOM, and $CaCO_3$. These factors can be classified into 1) SOM, which influences the substrate of phosphatase, and 2) soil pH, available N and P, and $CaCO_3$, which influence the environment and soil biota. More research should be conducted on straw returning to calcareous soils, especially on soil aggregates and their associated enzyme activities, as they have not been extensively studied.

In conclusion, the short term (3.5 years) of maize straw returning to the field had no significant influence on the calcareous soil aggregate distribution. However, a high rate of straw returning significantly increased SOM and activities of soil enzymes (acid phosphomonoesterase, alkaline phosphomonoesterase, and phosphodiesterase). The soil pyrophosphatase activities showed a different trend from the other three enzymes and were significantly influenced by soil available N and $CaCO_3$ concentration. Therefore, a high rate of straw returning is recommended to improve soil nutrition and phosphatase activities in calcareous soils.

Data availability statement

The original contributions presented in the study are included in the article/Supplementary Material; further inquiries can be directed to the corresponding authors.

Author contributions

X-JL: conceptualization, data curation, and original draft preparation. G-NZ: design of methodology and supervision. ZW: writing—review and editing. Q-DH: validation. PL: resources and supervision. All authors contributed to the article and approved the submitted version.

Conflict of interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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