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Original Research Article

# Variations of soil organic carbon fractions in response to conservative vegetation successions on the Loess Plateau of China



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#### ABSTRACT

Land use changes profoundly affect the equilibrium of soil organic carbon (SOC) sequestration and greenhouse gas emissions. With the current global climatic changes, it is vital to understand the influence of ecological restoration and conservation management on the dynamics of SOC under different land uses, especially in erosion-endangered Loess soils. Therefore, we investigated changes in SOC through a suit of labile fractions, namely: light fraction organic C (LFOC), heavy fraction organic C (HFOC), coarse particulate organic C (CPOC), fine particulate organic C (FPOC), and dissolved organic C (DOC), from two forests i.e., *Robinia pseudoacacia* (RP) and *Platycladus orientalis* (PO), with different ages, in comparison with farmland (FL). The SOC and STN contents significantly increased over 42 years in the RP forest where the contents of CPOC and FPOC were significantly higher than in the FL. Moreover, total SOC and its labile fractions, in the studied land use types, significantly correlated with soil CaCO<sub>3</sub>, pH, and STN contents, indicating their key roles in SOC sequestration. The results reported here from different stages of vegetation restoration. Our findings suggest that long-term natural vegetation restoration could be an effective approach for SOC sequestration and soil conservation on the Loess soil.

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Abbreviations: CPOC, coarse particulate organic carbon; DOC, dissolved organic carbon; FPOC, fine particulate organic carbon; FL, farmland; HFOC, heavy fraction organic carbon; LFOC, light fraction organic carbon; PO, Platycladus orientalis forest; RDA, redundancy analysis; RP, Robinia pseudoacacia forest; SOM, soil organic matter; STN, soil total nitrogen; SOC, soil organic carbon.

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#### 1. Introduction

Land degradation has considerably reduced crop production and altered biological diversity throughout the globe (Higginbottom & Symeonakis, 2014; Wijitkosum, 2021). Land degradation, caused by various factors, does not only deplete soil organic carbon (SOC)

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2095-6339/© 2022 International Research and Training Center on Erosion and Sedimentation, China Water and Power Press, and China Institute of Water Resources and Hydropower Research. Publishing services by Elsevier B.V. on behalf of KeAi Communications Co. Ltd. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/). stock but also reduces soil potentials to sequester C and accelerates the global greenhouse effect (Lal, 2001; Prăvălie et al., 2021), which makes the timely adoption of climate-smart and conservative practices significantly relevant. The amount of carbon (C) stored in soil is two to three times higher than that in the atmosphere and living vegetation C pools combined (Scharlemann et al., 2014; Richter et al., 2020). In fact, soil holds the third largest global C reservoir and releases 4% of C emissions each year into the atmosphere (Li et al., 2014). In addition, soil is a significant contributor of  $N_2O$  emissions, i.e., natural soil released approximately 6 Tg year<sup>-1</sup>, while agricultural soil released approximately 4.2 Tg year $^{-1}$ , which significantly affects N<sub>2</sub>O emissions (Saikawa et al., 2014). Carbon and N are the two key elements that protect ecosystem structure, function, and stability, with a crucial role in sustaining ecosystem productivity (Canedoli et al., 2020; Dai et al., 2022; Knops & Tilman, 2000; Reich et al., 2006).

Several studies on the Loess Plateau, China, have shown extensive alterations in SOC dynamics in response to climate and vegetation types (Han et al., 2018; Liu et al., 2011; Liu, Li, Liu, Wang, & Chang, 2021). The Loess Plateau, China, is particularly subjected to extreme soil erosion because of its soil poor structure (Chang et al., 2012; Wang et al., 2021). Several researchers demonstrated that erosion-related processes, such as transport, deposition, and detachment of soil materials, have shown considerable negatively impacted SOC accumulation and spatial distribution (Schiettecatte et al., 2008; Zhu et al., 2014; Liu et al., 2021; Wang et al., 2021). Afforestation and natural restoration are two key processes that can reduce soil erosion and other deleterious impacts caused by land degradation (Berthrong et al., 2012; Deng & Shangguan, 2017). However, the roles afforestation play in SOC, as well as in soil C, sequestration and soil conservation is still to be explored, particularly on the Loess Plateau, China. Nevertheless, previous studies have reported contradicting results where afforestation either accelerated SOC accumulation or showed a negligible influence on SOC sequestration (Chen, Yu, et al., 2017; Hu et al., 2021; Li et al., 2018; Rytter, 2016; Xiao et al., 2017). Thus, it is vital to understand the influence of ecological restoration and conservation management, e.g. the afforestation, on the dynamics of SOC in such erosion-endangered Loess soils.

Moreover, several studies have examined the association between SOC accumulations and soil inorganic C (SIC), and reported a positive relationship between SOC and SIC (Guo et al., 2016; Shi et al., 2017; Wang, Wang, Xu, et al., 2015; Dai et al., 2022). In contrast, there is also evidence on the negative association between SOC and SIC in soils under different land use types (Li et al., 2010; Zhao et al., 2016). Nevertheless, Zhao et al. (2016) found no correlation between SIC and SOC under diverse land use types on the Loess Plateau. However, the variation in the relationship between SOC and SIC may reveal insights on SOC and SIC responses to environmental conditions. It is clear that a higher soil pH often results in a low level of SOC accumulation, which reflects the possible instability of SOC in soils with high pH (Chen, Yu, et al., 2017; Demoling et al., 2007). Higher soil pH results in excessive releases of DOC, which affect microbial activities and C loss/ retention from agroecosystem (Shi et al., 2017; Zhang et al., 2020). Moreover, extensive evidence showed that high Ca<sup>+2</sup> availability is fundamental for SOC stabilization (Tavakkoli et al., 2015) due to greater formation of aggregates and consequent protection of SOC (Rowley et al., 2018). Apparently, higher content of  $Ca^{+2}$  in soil is due to a high level of CaCO<sub>3</sub>, i.e., SIC (Oste et al., 2002; Wang, Wang, Xu, et al., 2015).

Soil OC content is the result of a long-term and ongoing equilibrium among sequestration, mineralization and emissions (Dai et al., 2022; Hu et al., 2021). Land use changes stimulate disturbance in soil balance and cause ecological imbalances that can alter both the input and decomposition rates of organic matter and finally impact soil C and N content, e.g., the conversion of farmland to forestland accumulates SOC due to litter decomposition (Chang et al., 2011; Rennert et al., 2018; Tolbert et al., 2002; Zatta et al., 2014). Although different land use changes influence soil C differently, equilibrium occurs if environmental conditions, e.g., temperature, moisture, biota, and C inputs are comparatively stable (Qin et al., 2013; Rennert et al., 2018; Stewart et al., 2007).

Different contents of C fractions in soils are derived from different soil pedogenesis and agronomic management practices and can explain the composition, cycling pattern, and sequestration mechanisms of C in soil (Six et al., 2002; Hu et al., 2021). Soil OC primarily consists of two fractions, labile and recalcitrant C (Haynes, 2005), where the latter is a stabilized form of soil C that takes 100-1000 years to turn over, which indicates short term management practices have minimal effects on recalcitrant C concentrations (Haynes, 2005). In contrast, labile C fractions turn over very promptly as they are sensitive and responsive to management practices, which induce changes in SOC equilibrium. Labile SOC fractions are compositionally similar to plant material (Abdelrahman et al., 2016), but with less carbohydrates and a higher quantity of sterols indicating early stages of decomposition in soil (Gregorich et al., 1996). Moreover, the oxidation of SOC is the main driver of CO<sub>2</sub> flux from soil to the atmosphere, thereby, affecting nutrients cycling and ultimately soil quality and productivity (Mandal et al., 2007), which makes labile SOC fractions an early indicator of SOC quality (Haynes, 2005). Therefore, to assess land use driven changes in SOC, we used four DOC, LFOC, CPOC, and FPOC (Bolinder et al., 1999: Murata & Goh, 1997).

The DOC is a reactive SOC fraction that consists of different water dissolved organic fragments, whereas the LFOC and particulate organic C (POC) are composed of degraded plant and animal tissues with a relatively quick turn over, making them vital plant nutrients source (Wander et al., 1994). However, POC is relatively more stable than LFOC (Gregorich, 1996), and the proportion of POC in SOC is usually higher than the proportion of LFOC (Carter et al., 1998). Some land use changes, e.g., forests/grassland restoration, are effective strategies to increase C sequestration capacity of terrestrial ecosystems (UNFCCC, 2009) and with the responsiveness of the labile SOC fraction it is possible to observe changes in SOC in response to land management than could allow total SOC (Desta et al., 2021; Zhou et al., 2006).

We hypothesized that the labile SOC fraction can clearly show changes in SOC in response to different vegetation types and successional stages and consequently evaluate the conservation practices on the Loess Plateau. Therefore, the objectives of the current study are to: i) determine the effect of various vegetation types and successional stages on SOC (total and labile fractions) and STN; and ii) elucidate the influence of different vegetation successions on SOC sequestration and buildup.

#### 2. Materials and methods

#### 2.1. Experimental site description

Samples were collected from the Maliantan Farm located in the Yongshou County (34°47′58.4″N - 34°48′42.7″N, 108°05′22.6″E – 108°05′39.3″E) of the Yellow River Middle Reaches. This area is also an experimental site for the Science and Technology Strategic Program "Conservation of Water and Soil Vegetation Construction Engineering Research and Demonstration in the Loess Plateau". The area has an elevation range of 900–1300 m above sea level and average annual temperature and precipitation of 10.8 °C and 601.6 mm, respectively. Various vegetation types (Fig. S1, Supplementary Material), cultivation models, forests and natural grasses were planted in this study area during the 1980–1985 to control soil erosion and protect the environment. The main soil type at this study site is classified as Calcic Cambisols (IUSS Working Group WRB, 2015), representing 45.6% of the total area. The particle size distribution in the soils of the study sites was 30–55% sand; 20–45% silt: and 15–25% clay.

In this work, we sampled: i) arbor RP forests established in 1978–1992, where stands with the ages of 22, 28, 35, and 41 year, at the time of sampling, were sampled for the present study; ii) arbor PO forests established in 1978–1992, where stands with the ages of 10, 16, and 20 years, at the sampling time, were sampled for the present study; and compared the results of these two sampled forest sites with the iii) farmland (FL) site consisting of mono-cropping plots of wheat that was first planted 1000 years ago (Liu et al., 2021).

#### 2.2. Soil sampling

Topsoil samples (0–20 cm) were collected using a stainlesssteel shovel from five different spots from each vegetation type in October of 2002, 2008 and 2012. The samples were taken at the same elevation from various vegetation types and their succession phases at the same sampling period or at separate sampling times in the same vegetation types. However, for the C measurement, soil samples were collected from a plot size of  $25 \times 25$  cm. All collected soil samples were taken to the laboratory, air-dried at room temperature, then passed through 2-mm and 0.15-mm sieves to remove debris, stones, and any visible root.

#### 2.3. Soil chemical analyses

Soil pH was determined by pH meter in a soil extract (1:5; w/v). For the determination of CaCO<sub>3</sub>, a neutralization titration method was employed (Bao, 2000). Soil particle size distribution was measured by using the pipette method Ge et al. (2019) and the relative proportions of clay, silt and sand were used to determine soil texture classes (USDA taxonomy). Total SOC was determined by a total OC analyzer (TOC-V Series, SSM-5000 A, Japan) and STN was measured via H<sub>2</sub>SO<sub>4</sub> digestion using a H<sub>2</sub>SO<sub>4</sub>-CuSO<sub>4</sub>-Se catalyst by using the Kjeldahl method (Bremner, 1996).

#### 2.4. Fractionation of soil organic carbon

In this work, the LFOC, HFOC, CPOC, FPOC, and DOC were separated each from a single soil sample. The purpose was to compare each of these fractions among different land use systems to evaluate the land use effect on depletion/accumulation of each of SOC fractions.

A modified method after Cambardella and Elliott (1992) was utilized for the POC fractions separations. Briefly, 20-g air-dried samples and 100 mL of 5 g L<sup>-1</sup> sodium hexametaphosphate were shaken on a reciprocating shaker (90 rev min<sup>-1</sup>) for 18 h. The soil suspension was passed through stacked 250  $\mu$ m and 53  $\mu$ m sieves. All remaining material on the sieves was washed into dry clean dishes, dried in the oven for 48 h at 60 °C, and ground to analyze C content. The material >250  $\mu$ m was named coarse occluded particulate matter, and its C content was termed CPOC. Whereas, the 53–250- $\mu$ m material was named fine particulate matter, and its C content was termed FPOC. Here we considered the sum of CPOC and FPOC fractions as the total POC.

The method described by Cambardella and Elliott (1993, pp. 449–457) was used to determine the LFOC. In brief, 20 g airdried sample was placed in a centrifuge tube with 40 mL of 1.7 g cm<sup>-3</sup> NaI solution. Then, tubes were shaken using a reciprocating shaker for 60 min then centrifuged at  $1008 \times g$  for 10 min. The

suspended material was drawn off from suspension and isolated on Whatman GF/D filter paper; this process was repeated two to three times until no floating material remained. The material remaining at the bottom of the centrifuge tubes (heavy fractions; HFOC) was washed three times with distilled water, dried at 60 °C, weighed, and analyzed for C content. The organic C contents of the HFOC, CPOC, FPOC, and LFOC fractions were determined by using elemental analyzer (vario EL, Elementar Analysensysteme, Germany).

Soil DOC was measured by extracting 12.5 g of dry soil with 50 mL of 0.5 M  $K_2SO_4$ . Afterward, the mixture was placed on a shaker for 30 min at 180 rev min<sup>-1</sup>. The supernatant was decanted and collected from suspension and isolated on Whatman GF/D filter paper. Then, the concentration of extractable C was determined with an analyzer (TOC-VCSH, Shimadzu Corporation, Kyoto, Japan).

#### 2.5. Statistical analyses

One-way ANOVA was used to determine significant variations in SOC, STN, and labile SOC fractions in response to different restoration ages and land use types at  $p \le 0.05$ . For each land use type, the least significant difference (LSD) test was used to differentiate means of the different sampling periods/ages. The statistical analyses were performed using SPSS 12.0 software and data represent the means  $\pm$  standard errors (n = 3).

Correlations among the different SOC fractions and other soil parameters, such as CaCO<sub>3</sub>, STN and pH were determined by the redundancy analysis (RDA) using the VEGAN vegetation analysis package in R (Oksanen et al., 2013). Variance decomposition refers to the correlation and multivariate regression analysis among the SOC fractions and CaCO3, STN, and pH. The influential gradient force reveals the significance of CaCO<sub>3</sub>, STN and pH size to the SOC fractions. For the quantitative analysis of the influence of various dimensions of CaCO<sub>3</sub>, STN and pH on SOC sequestration, content of SOC fractions were used as the dependent (explained) variables, and CaCO<sub>3</sub>, STN and pH were used as the independent (explanatory or influencing) variables. Variance decomposition and single-factor sequencing were carried out to identify the influence extent of CaCO<sub>3</sub>, STN and pH on the SOC sequestration efficiency.

#### 3. Results

#### 3.1. Response of soil texture

All soils were characterized as loam except the PO forest sampled at age of 10 years, which was characterized as silt loam soil (Table 1). There were significant differences in soil textural particles (clay, silt, and sand content) under different vegetation succession; the proportion of sand significantly decreased with progress in restoration stages and considerably under the PO system. Moreover, clay content significantly (P < 0.05) increased in all vegetation types, especially in the PO, from 6.8% to 22%, whereas, silt content increased with the progress in the restoration stages only in the RP compared to the PO and FL.

#### 3.2. Changes in total SOC and STN

The different patterns of vegetation succession have shown significant effects on SOC with different restoration ages. Generally, with the progress in vegetation restoration SOC increased slowly and fluctuated among different vegetation restoration stages. The SOC content significantly increased in the FL between 2008 and 2012 (Fig. 1a), while in the PO forest, SOC content increased from 11.4 g kg<sup>-1</sup> in the 10-year-old PO trees to 27.2 g kg<sup>-1</sup> in the 20-year-old trees, and from 17.8 g kg<sup>-1</sup> in the 22-year-old trees to

#### Table 1

Selected soil characteristics from different land use types in the long-term soil management experiment in the Loess Plateau of China. Values represent the me	an $\pm$ standard
error (n = 3).	

Vegetation Types	Sampling time or years after conversion to forest/grass	рН	$CaCO_3 (g kg^{-1})$	Clay (%)	Silt (%)	Sand (%)	Texture
Farmland	2002	8.03 ± 0.02 b	164.0 ± 0.23a	20.37 ± 0.49 b	42.31 ± 0.32a	37.27 ± 0.18a	Loam
	2008	8.29 ± 0.00a	166.8 ± 0.31a	24.00 ± 0.27a	40.82 ± 0.28 b	35.21 ± 0.14 b	Loam
	2012	$8.29 \pm 0.04a$	167.6 ± 0.07a	$24.72 \pm 0.09a$	$40.41 \pm 1.36a$	34.87 ± 1.46c	Loam
Platycladus orientalis	10	7.58 ± 0.07a	155.2 ± 0.04a	6.75 ± 0.15 b	50.21 ± 0.27a	43.04 ± 0.13a	Silt Loam
	16	7.99 ± 0.01 b	137.2 ± 0.00 b	20.12 ± 0.77a	39.81 ± 1.79 b	$40.08 \pm 2.54a$	Loam
	20	$7.99 \pm 0.00 \text{ b}$	137.2 ± 0.00 b	22.12 ± 1.01a	39.15 ± 1.94 b	38.74 ± 2.95 b	Loam
Robinia pseudoacacia	22	8.01 ± 0.02a	133.2 ± 0.26a	21.61 ± 0.27c	$43.24 \pm 0.63a$	35.15 ± 0.90a	Loam
	28	$8.10 \pm 0.07a$	130.7 ± 0.25a	22.53 ± 0.07 b	41.88 ± 0.24 b	35.59 ± 0.18a	Loam
	32	$8.10 \pm 0.01a$	$131.2 \pm 0.06a$	22.99 ± 0.03 b	41.39 ± 0.28bc	35.63 ± 0.25a	Loam
	35	$6.90 \pm 0.00 \text{ b}$	114.4 ± 0.27 b	$22.26 \pm 0.08 \text{ b}$	$44.34 \pm 0.05a$	33.41 ± 0.13 b	Loam
	41	$7.57\pm0.02a$	$131.7 \pm 0.06a$	$23.93 \pm 0.23a$	$43.06\pm0.01a$	$33.00 \pm 0.12 \text{ b}$	Loam

Different letters within each column in each vegetation types indicate significant differences between different restoration stages (sampling or stand/grass age according to LSD at p < 0.05.

### 39.8 g $kg^{-1}$ in the 41-year-old trees in the RP forest (Fig. 1a).

Similar to SOC, STN also increased with the progress in the vegetation restoration except in the FL land use, which had a sharp increase between 2002 and 2008 but then decreased as observed in 2012 (Fig. 1b). The STN contents increased from 1.13 g kg<sup>-1</sup> in the 10 year-old PO trees to 1.92 g kg<sup>-1</sup> in the 20 year-old PO trees, from 1.10 g kg<sup>-1</sup> in the 22-year old RP to 3.60 g kg<sup>-1</sup> in the 41-year old RP (Fig. 1b). Consequently, the C:N ratio increased with different vegetation types and differed among the various stages. In the FL, the C:N ratio first decreased non significantly from 7.8 to 5.8, but then significantly increased from 10.1 to 14.2, however, in the RP the C:N declined continuously until 35 years after afforestation, but at later stages, it showed an increasing trend with the progress in successional stage from 35 to 41 years.

#### 3.3. Changes in SOC fractions

The DOC increased significantly with different vegetation types and successional stages (Fig. 2a). The largest overall mean DOC value was observed in the RP, followed by the PO, and FL vegetation systems. In contrast, DOC/SOC proportion increased in all vegetation types (Fig. 2a).

The total POC (CPOC + FPOC) decreased significantly in the FL, which was expected in comparison with the forestland systems, as the latter showed an increasing trend in the PO and RP. The content of CPOC was significantly (P < 0.05) different among the different vegetation types and restoration stages (Fig. 2b) as it increased from 4.8 g kg<sup>-1</sup> to 9.8 g kg<sup>-1</sup>, and from 3.4 g kg<sup>-1</sup> to 17.5 g kg<sup>-1</sup>, <sup>1</sup> in the PO, RP, respectively. The FPOC (Fig. 2c) showed a similar pattern as the CPOC, especially in the FL system, where FPOC significantly decreased over time. In the PO, the FPOC slightly decreased, during the 10–16-year age period, from 2.8 g kg<sup>-1</sup> to 1.8 g kg<sup>-1</sup>, but the difference was not significant. However, from the 16- to 20-year age period, FPOC increased significantly from 1.8 g kg<sup>-1</sup> to 4.5 g kg<sup>-1</sup> with the progress in restoration stages.

Although the LFOC increased in the FL from 0.69 g kg<sup>-1</sup> to 0.82 g kg<sup>-1</sup> during the 2002–2008 period, it declined to 0.72 g kg<sup>-1</sup> during the 2008–2012 period; however, the difference between 2002 and 2012 was not significant. Conversely, the LFOC significantly increased in the PO, and RP, (Fig. 2d). The HFOC showed an opposite trend to the LFOC as it continuously increased in all land use types (Fig. 2e). The HFOC/SOC proportion decreased in the early stages of PO (10 years) and RP (22 and 28 years; Fig. S2 Supplementary Material); it decreased from 76.7% to 73.8% in the first 10–16 years, but then increased from 73.8% to 76.7% in the subsequent 16–20 years in the PO system (Fig. S2, Supplementary



**Fig. 1.** Changes in a) soil total organic C (SOC) and b) soil total nitrogen (STN) observed in the farmland (FL), Robinia pseudoacacia (RP) and Platycladus orientalis (PO) vegetation types and restoration stages at the Loess Plateau of China. Values represent the mean  $\pm$  standard error (n = 3). Different letters within a land use indicate significant difference among the different restoration stages (sampling or stand/grass age) according to LSD at p < 0.05 level. Numbers after FL refers to the sampling time and to years after conversion after the PO, and RP.

#### Material).

In the FL system, the proportional LFOC carbon content (LFOC/ SOC) decreased from 9.4% to 8.0%; however, it did not change



**Fig. 2.** Changes in a) dissolved organic C (DOC); b) the coarse fraction of particulate organic C (CPOC); c) fine fraction of POC (FPOC); d) particulate organic C (POC = CPOC + FPOC); e) light fraction organic C (LFOC); and f) heavy fraction organic C (HFOC) observed in the farmland (FL), Robinia pseudoacacia (RP) and Platycladus orientalis (PO) vegetation types and restoration stages at the Loess Plateau of China. Values represent the mean  $\pm$  standard error (n = 3). Different letters within a land use indicate significant difference among the different restoration stages (sampling or stand/grass age) according to LSD at p < 0.05 level. Numbers after FL refers to the sampling time and to years after conversion after the PO and, RP.

between the first and last sampling times in the PO forest soil. In the RP forest, the LFOC/SOC increased from 7.4% to 16.1%, at later stages (35–41 years). Similarly, HFOC/SOC proportion decreased from 92.6% to 80.3% in the RP at the age of 22–35 years, but then increased from 80.3% to 83.9% at the age of 41 years (Fig. S2, Supplementary Material). However, in the FL, HFOC/SOC proportion significantly declined from 90.6% to 79.0% with the progress from 6- to 16-year vegetation period.

## 3.4. Influence of land use changes and successional stages on $CaCO_{\rm 3}$ and pH

Slight difference was observed in the  $CaCO_3$  content in the FL land use from 2002 to 2012; it increased from 164.0 g  $kg^{-1}$  to

167.6 g kg<sup>-1</sup> (Table 1). In contrast, CaCO<sub>3</sub> tended to decrease in the other vegetation restoration and with successional stages. For example, in the PO forest, CaCO<sub>3</sub> decreased from 155.2 to 137.2 g kg<sup>-1</sup> in the 16-year-old PO, but subsequently remained at 137.2 g kg<sup>-1</sup>. A similar pattern was observed in the RP forest as with the progress in the successional stage CaCO<sub>3</sub> content decreased. However, CaCO<sub>3</sub> in the RP system did not show any significant changes between the 22-year and the 32-year age period as it decreased from 133.2 g kg<sup>-1</sup> to 131.2 g kg<sup>-1</sup> and continue to decline to 114.04 g kg<sup>-1</sup> at 35-year age, but surprisingly, after 35 years it increased 114.04 g kg<sup>-1</sup> to 131.7 g kg<sup>-1</sup>. Changes in CaCO<sub>3</sub> represent the changes in SIC in response to land use and climatic conditions.

No significant differences were observed in soil pH over the study period (Table 1), however, soil pH slightly increased

numerically in the FL and PO systems. Also, there was a variation in soil pH with the successional stages in the RP forest where soil pH slightly increased at the 32-year stage, but then decreased at the 35-year and 41-year stages.

#### 3.5. Influence of soil properties on C sequestration

The decomposition of variance in the RDA was used to evaluate the influence of CaCO<sub>3</sub>, pH, and STN on the C accumulation process, where the effects of each individual dependent variable and the interacting variables were calculated (Fig. 3). Among the dependent variables, CaCO<sub>3</sub>, pH and STN explained 84% of variance in CPOC, 82% in FPOC, 89% in DOC, 62% in HFOC, 79% in LFOC, 96% in POC, and 74% in SOC. Among the dependent variables, STN could explain 8%, 9%, 19%, 24%, 23%, 11% and 28% of the variance in CPOC, FPOC, DOC, HFOC, LFOC, POC, and SOC, respectively, which indicates that STN is a key factor for SOC sequestration.

The combination of CaCO<sub>3</sub>, pH, and STN could explain 51%, 49%, 30%, and 37% of variance in CPOC, FPOC DOC, and LFOC, respectively. For the HFOC, the interaction effect of CaCO<sub>3</sub> and STN showed a more profound impact on SOC fractions, which reached 29%, and the synergetic effect of CaCO<sub>3</sub>, pH, and STN was 16%. For the POC, the interaction effect of CaCO<sub>3</sub>, pH and STN were the main factors for the sequestration of SOC, representing an effect of 57%. Specifically, STN was the main driving factor for SOC sequestration as it explained 28% of variance in SOC. The SOC sequestration ability in the Loess soil results from a combination of CaCO<sub>3</sub>, pH and STN where the LFOC and HFOC were primarily affected by STN and its combination with soil CaCO<sub>3</sub>, and pH.

#### 4. Discussion

#### 4.1. Influence of land use and vegetation succession on SOC and STN

Alterations in SOC and STN contents due to land use changes are vital drivers for C and N cycling. Higher SOC content indicates higher C input than C emission/loss from soil and implies management practices favor soil conservation. Soil OC content increased through higher C inputs when conventional agricultural land was replaced by other conservative land uses, e.g., conversion to forestland. To a certain extent, an increase in the SOC stock helps reduces CO<sub>2</sub> emission levels and global warming, however, it depends on land use type and/or vegetation cover whether SOC will increase under certain soil management and land use type (Dai et al., 2022. Different land use types affect differently soil C and N (Zhang et al., 2013) in the current study different land use types significantly influenced soil C and N contents (Fig. 1). Different vegetation can influence C and N contents by litter input variability and decomposition pathways (Zhang et al., 2013). Prietzel and Bachmann (2012) have shown that retention and accumulation of SOC and STN can be affected by various plant species, plant traits, and standing density.

Furthermore, stand age also significantly affected soil C and N content (Fig. 1). In this study, the RP forest, with an age of 41 years exhibited the largest SOC and STN contents (39.8 g kg<sup>-1</sup> and 3.6 g kg<sup>-1</sup>, respectively) among all land use types under investigation on the Loess plateau. This observation suggests that natural vegetation restoration has a greater capability for soil conservation (Du & Gao, 2020; Wen et al., 2021) and soil quality improvement by maintaining and increasing SOC and STN. The increase in SOC is possibly due to the input by plant residues and cover on soil surface, as also documented by Liu (2005) for the mountain area of Southern Ningxia. The lowest SOC found in the FL system is due to the decline in C inputs and greater C mineralization (Nyamadzawo et al., 2009). Moreover, farmers on the Loess Plateau generally practice tillage, thus, oxidizing soil organic matter (SOM), which causes a notable C loss (Lal, 2004; Nachimuthu & Hulugalle, 2016).

The key measure of a vegetation system is to maintain and improve SOC, which consequently contribute to soil conservation as SOC plays a pivotal role in maintaining soil physiochemical and biological quality. Under forestland (RP and PO), SOC increased due to a thick litter layer that decreased the loss of nutrients due to soil erosion processes, and ultimately increased SOC. These findings reveal that land use conversion and adjustment have substantial



**Fig. 3.** Effects of CaCO3, pH and soil total N (STN) on soil organic C (SOC) fractions in the long-term management experiment in the Loess Plateau of China. X1, X2, and X3 represent CaCo3, pH and STN, respectively. SOC: soil organic C, dissolved organic C (DOC), particulate organic C (POC), coarse particulate organic C (CPOC), fine particulate organic C (FPOC); soil light fraction C (LFOC), and the heavy fraction C (HFOC). Numbers inside the circles refer to the variation explained by one set of explanatory variables.

potential to improve SOC sequestration in China's Loess Plateau. Therefore, afforestation is suggested to conserve soil quality and promote C sequestration in soil.

#### 4.2. Influence of soil texture on SOC and STN

Soil texture is another crucial factor affecting SOC sequestration and stability. Previously, Tisdall & Oades, 1982 and Hassink (1997) reported that the addition of organic matter to soils results in the formation of organo-mineral associations between SOM and clay and silt particles. The fine particles of silt and clay in aggregates improve organic matter retention as they provide physical protection against microbial decomposition (Whisler et al., 2016). Theoretically, fine particles have a larger surface area compared to coarse particles and, thus, have a stronger ability to bind to organic matter and protect them from being accessed by soil microorganisms (Zhou et al., 2019). As a consequence, soil texture is expected to influence SOC sequestration after land use changes as forest systems help to improve soil structure and soil development through mineral transformations (Ferro-Vázquez et al., 2014). Furthermore, clay and silt contents are important indicators of soil structure and are used to assess soil guality (Zhang et al., 2021). Nonetheless, soil particles and structure play a critical role in soil conservation against erosion as indicated by Sun et al. (2021) who estimated that the mean sediment load, driven by splash erosion, was significantly different in the order of sandy loam > sandy clay loam > clav loam > loamv clav. All of the studied soils are loamv except the FL soil collected in 2002 and the PO sampled at 10-year age, which are rich in silt and sand. The SOC and STN content gradually increased with increased silt and clay content, especially in the PO and FL sites, as reported for other tree species (Liu et al., 2021, which indicates that silt and clay contents are positively correlated with SOC and STN. These results are in agreement with previous studies conducted throughout the Loess Plateau (Ge et al., 2019; Wang et al., 2020). The SOC content was improved greatly with increased silt content across different land use conversions and across many sites with varying soil texture (Dai et al., 2022; Ge et al., 2019), which implies that vegetation cover prevents or slows soil degradation by preventing the removal of highly erodible particles and, thus, promotes SOC accumulation.

### 4.3. Effect of land use changes on $CaCO_3$ and pH and their relation to SOC sequestration

Calcium carbonate and soil pH were positively correlated (Fig. 4), where in all vegetation types, soil pH remained around 8.0 and CaCO<sub>3</sub> were above 70 g kg<sup>-1</sup>. CaCO<sub>3</sub> acts as a strong buffering agent and keeps soil pH *quasi* constant as long as active CaCO<sub>3</sub> contents are sufficient in the soil (Raza, Miao, et al., 2020). Soil pH starts decreasing only when CaCO<sub>3</sub> stock are lost from the soil as the buffering capacity of other soil buffers is weaker than carbonate (Raza, Miao, et al., 2020).

CaCO<sub>3</sub> contents were nearly stable in all vegetation types, except in the PO forest (Table 1). CaCO<sub>3</sub> contents in the FL remained relatively stable (increased by 3.55 g kg<sup>-1</sup>) during 20 years, which could be explained by the maintained balance between CaCO<sub>3</sub> neutralization from acidification induced by N fertilizers and the formation of pedogenic carbonates facilitated through the addition of Ca<sup>2+</sup> containing fertilizers such as single and triple superphosphate (Raza et al., 2020). The decrease in CaCO<sub>3</sub> contents in the PO forest could be explained by the events of high rainfall causing CaCO<sub>3</sub> dissolution and leaching of Ca<sup>2+</sup> and possibly due to the root exudates that contain organic acids, which neutralize CaCO<sub>3</sub>.

Soil organic C contents increased with the restoration age in all vegetation types, due to higher plant biomass input over the years



**Fig. 4.** Correlation among soil total organic C (SOC), soil total nitrogen (STN), particulate organic C (PO), coarse particulate organic C (CPOC), fine particulate organic C (FPOC); soil light fraction C (LFOC), heavy fraction C (HFOC), the proportion of CPOC/SOC, FPOC/SOC, POC/SOC, LFOC/SOC and HFOC/SOC. \* Correlation was significant at  $p \leq 0.05$ ; \*\* Correlation was significant at  $p \leq 0.01$ .

from roots and plant litter (Zhong et al., 2021). Similar observation was reported in previous studies focusing on different land use systems such as the conversion of farmland to forestland (e.g., Liu et al., 2021) including, hippophae, robinia and pinus forests in Loess hilly and gully regions of China (Han et al., 2017; Li, Liu, et al., 2017). However, comparing the PO and RP land use, it is possible to postulate that the PO land use favors larger SOC accumulation in the studied sites as the labile and total SOC fractions were greater in the PO than in the PR sites at a comparable standing age (e.g., PO at 20 years vs. RP at 22 years). Still, SOC accumulation seems was facilitated by the presence of higher CaCO<sub>3</sub> contents in soil as in the PO soils (Table 1), which promote SOC stabilization process through clay-Ca<sup>2+</sup>-OM complexes (Rowley et al., 2018).

In addition, higher SOC and biological activity under natural vegetation restoration expectedly resulted in higher heterotrophic and autotrophic respiration, which possibly caused higher CO<sub>2</sub> pressure (Chang et al., 2012) that consequently promoted the dissolution of pedogenic CaCO<sub>3</sub> and the stabilization of SOM. However, as soil is complex system the accumulation of SOC under restored natural vegetation could have induced organic acids in the soil, which reduced the availability of Ca<sup>+2</sup> through cationic exchange of Ca on soil particles (Sartori et al., 2007), which in turn accelerate the dissolution and leaching of soil carbonate and consequently reduce SIC concentrations in forests (Du & Gao, 2020; Kuzyakov & Razavi, 2019). The SIC stocks generally exhibited a declining trend with increasing restoration age in the surface soil layers and were augmented in the deep soil layers as also reported by Yang et al. (2012). In our study, there was a negative correlation between SOC and SIC (Fig. 4) which was similarly reported by Liu et al. (2014).

A positive correlation between soil pH and SIC was observed (Fig. 4), which can be explained by the acidification process driven by SOM decomposition and root exudates. The acidic environment in soil accelerates the dissolution of soil carbonate and leads to a decline in SIC (Raza, Miao, et al., 2020; Zamanian & Kuzyakov, 2019). The pattern and variations in soil pH with different land conversions, in our study, are in line with previous studies (e.g., Deng et al., 2013; Falkengren-Grerup, ten Brink, & Brunet, 2006),

where soil pH showed a declining trend with woodland growth on the Loess Plateau and thus had a negative correlation with SOC.

#### 4.4. Effect of land uses and successional stages on labile SOC

Different SOC fractions exhibited different responses to land use changes (Liu et al., 2010; Yusheng et al., 2004). For instance, labile SOC was the most prominent C that is easily utilized by soil (micro) organisms, showing rapid soil response and sensitivity to land use change and indicating labile SOC is considered a key element of nutrients cycling. Total SOC showed less sensitivity to tillage than did LFOC or POC as reported by Liu et al. (2018) and Mi et al. (2019). Miller et al. (2019) showed that LFOC and POC in forest soils affect ions alteration in different land use patterns and could be used as sensitive indicators to detect soil C stock changes. The LFOC is more labile than POC (Gregorich, 1996) and is more responsive to organic amendment but to a different extent in different farmland sites (Abdelrahman et al., 2017; 2020).

Nevertheless, DOC is considered one of the most responsive fractions of SOC. Our study showed that DOC variations are much quicker than that of soil LFOC, followed by CPOC and FPOC, respectively. These observations also agree with the results reported by previous studies on labile soil C (Gregorich, 1996; Pu et al., 2017). Although DOC is considered a small fraction of soil C, it is an essential component involved in several soil processes (Chantigny, 2003) and acts as a C facilitator for soil microbes (Li, Liu, et al., 2017). A significant increase in DOC was observed in the forestland, suggesting that forests had positive effects on soil microbes and provided a source of C by the aboveground and belowground litter decomposition. In this study, the DOC/SOC proportion was higher in the FL and was positively interrelated with pH (Fig. 4). A recent study by Zhang et al. (2020) showed that a higher ratio of DOC/SOC resulted in more desorption and removal of DOC and lower SOC accumulation, which may imply the low conservation status of the FL. Also, earlier research showed that higher soil pH increases soil erodibility and reduces soil aggregate stability and SOM protection against decomposition (Kalbitz et al., 2000; Tavakkoli et al., 2015).

SOC quality is affected by land use changes, which may be evaluated by the proportion of C in various SOC fractions to total SOC (Eclesia et al., 2012; Martens et al., 2004). The proportion of labile SOC to total SOC generally reflects the impact of various vegetation types on soil C behavior more efficiently than does total SOC alone. Usually, the higher the labile SOC proportion, the more dynamic is SOM, implying that susceptibility to mineralization is also increased. Conversely, the accumulation of SOC tends to increase with a higher proportion of stabilized soil C, as was reported for the proportion of HFOC/SOC in this study and in other works (Cheng et al., 2010; Wen et al., 2017).

With the progress in vegetation stages, labile SOC proportion of SOC gradually declined, followed by an increase in the proportion of stable SOC (HFOC), which indicates that the SOC is undergoing the stabilization processes. Our observations in this regard are consistent with those of Tang et al. (2010) and Xu et al. (2019), who reported long term cultivation practices, could facilitate the accumulation of organic C in soil, particularly in silt and clay size fractions; i.e., mineral bound organic matter. The increase in stable SOC, i.e., HFOC, was likely due to the supply of aboveground and underground organic materials with the favorable characteristics, in terms of quantity and quality, through vegetation growth, which sustained and promoted the development of SOM stabilization continuum (Greogorich et al., 2006).

Long-term vegetation restoration conserves and protects soil from degradation, as it becomes less erodible with increasing restoration age. Moreover, with progress in restoration stages, SOM stock tends to be balanced due to continuous plant litter and root inputs. Furthermore, labile SOC gradually degrades where a fraction of it is steadily turned into the relatively stable forms of SOC, as illustrated by Gregorich et al. (2006). As the proportion of labile SOC showed a declining trend while increasing the total SOC, it implies a gradual increase in SOC stability, and microorganisms inaccessibility to utilize it extensively. Therefore, reestablishment of natural vegetation seems to play a critical role in enhancing longterm SOC stabilization in the study area. Labile SOC, however, plays a significant role as a short-term indicator for evaluating the ecological restoration progress of soil.

#### 4.5. Influence of soil properties on C sequestration

Soil OC sequestration is affected by soil chemical, physical and biological properties; SOC was significantly negatively correlated with CaCO<sub>3</sub> and pH in this study. Sartori et al. (2007) demonstrated that SOC sequestration as a result of vegetation restoration is likely attributed to the increase in carbonic and organic acid production, which can reduce Ca<sup>+2</sup> availability via cation exchange, ultimately increasing carbonate dissolution and leaching of Ca<sup>+2</sup> from the surface soil layer to the subsurface layer and, thus, decreasing SIC. Furthermore, our findings indicate that SOC accumulation was significantly associated with labile SOC pools (Fig. 4), which are consistent with the findings of Zhao et al. (2015) for the same study area. The increase in labile SOC may be associated with fresh organic C inputs from the plant roots and litter (Sierra et al., 2013; Zhao et al., 2015) suggesting that SOC sequestration was a result of an increase in labile C and consequent transformation to stabilized C forms. Nevertheless, conservation management helps reducing soil erosion (Du, Jian, Du, & Stewart, 2021) as soil erosion modulus varies with land use type in the order of farmland > forest as reported by Yan et al. (2018) who estimated land use/cover change effect on soil erosion response in a catchment of the Loess Plateau.

#### 5. Conclusions

The distribution of SOC among different fractions changed when the farmland was converted to forestland, which was probably due to the continuous supply of organic materials. Different vegetation restoration strategies influence soil quality by improving SOC and STN, especially their labile fractions. Additionally, the increase in total SOC indicate that soil erodibility is possibly reduced as a fraction of labile C is stored in a stable form, which is possibly due to an equilibrium between organic matter inputs and decomposition, which favors SOC accumulation driven by different vegetation restorations. These results provide a better understanding of SOC and STN variations in different land use stages following vegetation restoration. Our findings suggest that long-term restoration of natural vegetation plays a significant role in SOC pools cycling and sequestration in the study area. This information shall help the evaluation of vegetation restoration and soil C buildup at different scales, which could be an effective approach for SOC sequestration and soil conservation.

#### **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.

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#### Appendix A. Supplementary data

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