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ORIGINAL ARTICLE

Effects of 15 years of manure and inorganic fertilizers on soil organic carbon fractions in a wheat-maize system in the North China Plain

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Abstract Soil organic carbon (SOC) and its labile fractions are strong determinants of chemical, physical, and biological properties, and soil quality. Thus, a 15-year experiment was established to assess how diverse soil fertility management treatments for winter wheat (*Triticum aestivum* L.) and summer maize (*Zea mays* L.) cropping system affect SOC and total N (TN) concentrations in the North China Plain. The field experiment included three treatments: (1) unfertilized control (CK); (2) inorganic fertilizers (INF); and (3) farmyard manure (FYM). Concentrations of SOC, TN, and different labile SOC fractions were evaluated to 1-m depth. In comparison with INF and CK, FYM significantly increased SOC and TN concentrations in the 0–30 cm depth, and also those of dissolved organic C (DOC), microbial biomass C (MBC), hot-water extractable C (HWC), permanganate oxidizable C ($KMnO_4$–C), and particulate organic C (POC) in the 0–20 cm depth. Despite the higher crop yields over CK, application of INF neither increased the SOC nor the labile C fractions, suggesting that by itself INF is not a significant factor affecting SOC sequestration. Yet, POC (18.0–45.8% of SOC) and HWC (2.0–2.8%) were the most sensitive fractions affected by applications of FYM. Significantly positive correlations were observed between SOC and labile organic C fractions in the 0–20 cm depth. The data support the conclusion that, wherever feasible and practical, application of FYM is important to soil C sequestration and improving soil quality under a wheat/maize system in the North China Plain.

Keywords Farmyard manure · Inorganic fertilizer · Soil organic C and total N · Labile soil organic C · North China Plain

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Introduction

Soil organic carbon (SOC) plays a crucial role in determining physical, chemical and biological properties, and the overall soil quality and fertility (Gregorich et al. 1994). Restoration of SOC in arable lands also represents a potential sink of atmospheric CO_2 (Lal and Kimble 1997; Banger et al. 2009). Therefore, the amount and quality of SOC in agricultural soils benefit both soil productivity and environmental quality, making it a truly "win–win strategy" (Lal 2004). Some common strategies for enhancement of SOC include conservation tillage which reduces decomposition (Six et al. 2000; Chen et al. 2007, 2009), and optimal fertilization which increases C input by enhancing biomass production. In general, application of organic fertilizers and especially manure, either alone or in combination with inorganic fertilizers, increases SOC concentration (Blair et al. 2006a, b; Rudrappa et al. 2006; Manna et al. 2007; Purakayastha et al. 2008; Gong et al. 2009b). In contrast, applications of inorganic fertilizers have often produced contradictory effects on SOC concentrations and/or its fractions: enhancement (Gong et al. 2009b), suppression (Ghani et al. 2003; Wu et al. 2004), or no effect (Šimon 2008). This uncertainty is partly attributed to the specific processes governing C sequestration under management practices, and which vary with soil type, climate, and crop rotation. Therefore, it is important to assess C sequestration for specific climate/soil/crop systems in order to draw site-specific conclusions.

Ironically, losses or gains in SOC over short- and medium-term are difficult to detect because of high antecedent amounts and large temporal and spatial variability (Bosatta and Ågren 1994). In contrast, labile C fractions [i.e., microbial biomass C (MBC), particulate organic C (POC), dissolved organic C (DOC), hot-water extractable C (HWC), and permanganate oxidizable C ($KMnO_4$–C)] respond more quickly to changes in management practices than SOC, and are thus used as early and sensitive indicators of SOC changes (Powlson et al. 1987; Cambardella and Elliott 1992; Blair and Lefory 1995; Haynes 2000; Ghani et al. 2003). In general, the labile C fraction is decomposed relatively easily, has a greater turnover rate, and a smaller amount compared with recalcitrant fractions (McLauchlan and Hobbie 2004). Thus, labile C fractions are important to study, as these fractions fuel the soil food web and greatly influence nutrient cycles and many biologically related soil properties (Weil et al. 2003). Since soil is a complex system, the measurement of a single labile C fraction does not adequately reflect management-induced changes in soil quality. In contrast, the simultaneous measurement of several labile fractions is typically required for a better assessment of the effects of management on soil properties (Iovieno et al. 2009).

The North China Plain is the largest and most important agricultural region of China. It covers an area of 31 million ha (Mha), of which 18 Mha are used for agriculture (18.6% of the national total), and support a population of 203 million. The predominant cropping system is a winter wheat (*Triticum aestivum* L.)—summer maize (*Zea mays* L.) rotation, covering up to 60% of arable land in this area. The North China Plain supplies more than 50% of the nation's wheat and 33% of its maize (China Statistics Bureau 2001). Thus, sustainability of agriculture in this region could affect China's food security and may influence the environment.

China has undergone dramatic changes in the agricultural sector over the past 30 years since 1980. Prior to 1950s, organic fertilizers, such as farmyard manure (FYM), were major amendments to maintain soil fertility. Along with the rapid growth in demand for food, driven by the ever increasing population between 1960s and 1990s coupled with the fast economic development since the 1980s, the organic fertilizers were gradually replaced by synthetic (inorganic) fertilizers. Thus, a long-term fertilizer management experiment established in 1993 in Quzhou (located in the centre of the North China Plain) was used to assess the effects of fertilizers and manuring on changes in SOC and its labile fractions. The objectives of this study were to determine the effects of application of FYM and inorganic fertilizers over 15 years on: (1) SOC and total N (TN) concentrations; (2) labile fractions of SOC including MBC, DOC, HWC, POC and $KMnO_4$–C, and their sensitivity to management-induced changes; and (3) relationships between SOC concentration and the labile C fractions. The strategy is to identify the best fertilizer management practices for SOC sequestration under a winter wheat-summer maize double cropping system in the North China Plain.

Materials and methods

Description of sites and soil sampling

The study was conducted on an on-going long-term fertilizer experiment (since 1993) at China Agricultural University's Quzhou agricultural experiment station (115°01′E, 36°52′N, 40 m a.s.l.) in Hebei province, Northern China. The station is located in a warm, semi-humid and continental temperate monsoon zone, with mean annual temperature of 13.1°C and mean annual precipitation of 556 mm. Approximately 60% of the annual precipitation is received between July and September. The annual frost-free period is 200 days. The antecedent soil properties (in 1993) for 0–20 cm depth were as follows: pH (soil : water ratio of 1:5) of 8.77, and concentration (g kg^{-1}) of 10.03 of soil organic matter, 0.59 of TN, 0.58 of total P, 26.13 of available P, and 65.37 of available K. Wheat and maize yields at the beginning of the experiment were 1,985 and 3,828 kg ha^{-1}, respectively. The soil, with silty loam texture (Table 1) is classified as a Cambisol according to the FAO/WRB (FAO 1998).

A double-cropping rotation consisting of winter wheat and summer maize is the typical cropping system practiced in this region. Three treatments selected for the present study were: (1) control (CK, without any FYM or inorganic fertilizers); (2) inorganic fertilizers (INF: 600 kg ha^{-1} $CO(NH_2)_2$, 1,125 kg ha^{-1} NH_4HCO_3 and 1,125 kg ha^{-1} $Ca(H_2PO_4)_2{\cdot}H_2O$, corresponding to the NP rates of 362 kg N ha^{-1} and 272 kg P ha^{-1} for each crop); (3) FYM (15 Mg ha^{-1} for each crop). The application rates of fertilizer and manure have been constant since the start of the experiment. The treatments were allocated in 10.5 × 3 m plots, replicated thrice, and allocated in the randomized block design. The FYM contained 60% straw (wheat or maize straw), 30% live-stock dung, and 10% cottonseed (*Gossipium hirsutum*)-press mud. On average, it contained 22.8% C, and 0.67% N (Hu et al. 2008). The entire doses of inorganic fertilizers and manure were uniformly broadcast by hand as basal dose and incorporated by a rotary cultivator to a depth of 18 cm immediately prior to the sowing of wheat and maize. Winter wheat is seeded on October 19th and harvested on June 13th. Summer maize is seeded immediately following the wheat harvest on September 29th. Winter wheat received three irrigations: 70 mm before winter, 80 mm at tillering and 90 mm at the initial grain filling stages. The summer maize received only two irrigations: 70 mm at emergence and 90 mm at the initial grain filling stages. Besides the three fertilizer treatments, all other agronomic management was identical. After harvest, all the aboveground residues were mowed and incorporated into soil in INF (14,704 kg ha^{-1}) and FYM

Table 1 Texture and pH of soil of the experimental site at Quzhou

Soil characteristics	Soil depth (cm)						
	0–10	10–20	20–30	30–40	40–60	60–80	80–100
Texture[a]	Silty loam	Silty loam	Silty loam	Silty loam	Silty loam	Silty loam	Silty loam
Sand (g kg^{-1})	104.8	26.4	28.3	53.4	53.4	76.3	55.3
Silt (g kg^{-1})	691.8	770.8	769.2	765.4	725.1	763.1	804.0
Clay (g kg^{-1})	203.5	202.8	202.4	181.3	221.6	160.6	140.7
pH_{CaCl2} (1:2.5)							
CK	7.63 (0.06) a	7.75 (0.02) a	7.77 (0.06) a	7.76 (0.05) a	7.72 (0.02) a	7.73 (0.06) a	7.72 (0.03) a
INF	7.49 (0.06)ab	7.73 (0.04) a	7.80 (0.02) a	7.78 (0.02) a	7.76 (0.03) a	7.79 (0.08) a	7.80 (0.03) a
FYM	7.43 (0.10) b	7.58 (0.08) b	7.79 (0.03) a	7.74 (0.04) a	7.78 (0.03) a	7.77 (0.03) a	7.78 (0.05) a

[a] *Sand* 2–0.05 mm, *Silt* 0.05–0.002 mm, *Clay* < 0.002 mm

Values are means with the standard deviation in parenthesis (n = 3). Values within a column followed by different lowercase letters are significantly different ($P < 0.05$). *CK* control, without any farmyard manure or inorganic fertilizers, *INF* inorganic fertilizers, *FYM* farmyard manure

(15,483 kg ha^{-1}), but they were removed from the CK treatment.

Soil samples were collected at 0–10, 10–20, 20–30, 30–40, 40–60, 60–80 and 80–100 cm depths from each plot with a 10 cm diameter soil core sampler after the summer maize harvest in October 2008. Four soil cores within each plot were well mixed and composited by depth. There were a total of 63 composite samples representing three fertilizer treatments, seven depths, and three field replicates.

Each soil sample was separated into two parts. One part was air-dried, ground with wooden blocks and passed through a 2-mm sieve for physical and chemical analyses. The other part was sieved through a 2-mm sieve and frozen for biochemical analysis. Identifiable crop residues, root material, and stones were removed during sieving.

Soil analysis

Labile soil organic C fractions

Labile SOC fractions were determined only for the upper soil layers (0–10, 10–20 and 20–30 cm depths), as in general they did not differ significantly among the three fertilizer treatments below 30 cm depth.

The MBC was determined by the chloroform fumigation-extraction method on fresh soils (Vance et al. 1987). Each replicate was divided into two equivalent portions, one was fumigated for 24 h with ethanol-free chloroform and the other was the unfumigated control. Both fumigated and unfumigated soils were shaken for 30 min with 0.5 M K_2SO_4 (1:4 soil: extraction ratio) and centrifuged and filtered. Extracts were analyzed for DOC on a Multi 3100N/C TOC analyzer (Analytik Jena, Germany). Soil MBC was calculated by dividing the differences of extractable C between fumigated and nonfumigated soils with a conversion factor of 0.45 (Joergensen 1996). The unfumigated samples were used to estimate the background DOC values.

The HWC was determined by a modified method of Ghani et al. (2003). Briefly, 10 g of fresh soils were extracted with 40 ml of distilled water in 50 ml polypropylene centrifuge tubes. The tubes were shaken for 30 min on an end-over-end shaker and then were left in a hot-water bath for 18 h at 80°C. The tubes were then shaken for 10 min to ensure that the extracted C was fully suspended. The tubes were centrifuged for 15 min at 8,000 rev min^{-1}, and the supernatants were vacuum filtered through 0.45 μm filters. Extracts were analyzed for total organic C (TOC) on a Multi 3100N/C TOC analyzer (Analytik Jena, Germany).

The $KMnO_4$–C was determined according to Vieira et al. (2007). Finely ground air-dried soil samples (equivalent to 15 mg of SOC) were oxidized by 25 ml of 333 mM $KMnO_4$. The suspensions were horizontally shaken at 60 rev min^{-1} for 1 h and centrifuged at 2,000 rev min^{-1} for 5 min. The supernatants were diluted and measured at 565 nm with a spectrophotometer (UV2300).

The POM was separated from soil following the wet sieving method of Cambardella and Elliott (1992). Briefly, 20 g of air-dried soil (<2 mm) was dispersed in 100 ml of 5 g l^{-1} sodium hexametaphosphate solution by shaking overnight. The dispersed materials were passed through a 53 μm sieve. The materials left on the sieve (>53 μm) (POM) were oven-dried at 60°C and ball-milled. The C concentrations in POM fractions were determined by dry combustion method.

Soil organic C and total N, pH and texture

Samples for SOC measurements were pretreated with 0.5 M HCl to remove carbonates (Chen et al. 2009) and then ball-milled. Concentrations of SOC and TN for bulk soil and POC were determined by dry combustion (vario Macro CNS Analyzer, elementar, Germany).

Soil pH was determined with a PHS-3C pH meter (REX Instrument Factory, Shanghai, China) by mixing 10 g of soil with 25 ml of 0.01 M $CaCl_2$ solution. Soil texture was obtained by the pipette method (Gee and Bauder 1986).

Calculations and statistical analysis

The effects of fertilizer treatments on SOC, TN, and labile SOC fractions (MBC, DOC, HWC, $KMnO_4$–C, POC) within each depth were analyzed using one-way ANOVA. Differences were considered significant at $P < 0.05$. Pearson linear correlation was used to evaluate the relationships between the parameters. All statistical analysis was performed with SPSS for windows, version 11.0.

The sensitivity index (SI) related to fertilizer treatments for labile SOC fractions was calculated using Eq. 1:

$$\text{SI} = (\text{labile SOC fractions in FYM or INF treatments} - \text{labile SOC fractions in CK}) \times 100/\text{labile SOC fractions in CK} \quad (1)$$

Results and discussion

Soil pH and crop yields

Soils were neutral to slightly alkaline in all the three treatments, with pH values ranging from 7.43 to 7.80 (Table 1). In comparison with CK, application of FYM significantly lowered pH values in the 0–10 and 10–20 cm depths, while INF tended to lower pH in the 0–10 cm depth (Table 1). Decrease in pH of the surface layer in the INF might be attributed to the nitrification and acidification processes stimulated by continuous application of fertilizers as well as by H^+ released by roots. Similar results have been reported by Wang and Yang (2003) and Guo et al. (2010).

The decrease in pH of the soil amended with FYM might be due to the presence of organic acids in the manure. Chang et al. (1990) also reported that soil pH in the top 15 cm of calcareous soils (pH 7.8) amended annually with cattle manure for 11-year declined by 0.3 to 0.7 units. In contrast, however, application of manure in an acidic soil can increase its pH (Hue 1992; Whalen et al. 2000). Presence of carbonates and bicarbonates in manure can increase pH of an acidic soil. Also manure has organic acids with carboxyl and phenolic hydroxyl groups which buffer soil acidity and increase the pH of acid soils (Hue 1992; Wong et al. 1998; Whalen et al. 2000). Thus, the effects of manure on soil pH depend on the manure source and soil characteristics.

Application of FYM and INF significantly increased the yields of wheat and maize compared to that in the CK, but did not differ among each other (Fig. 1). Long-term application of FYM improves soil fertility including concentrations of SOC and of macro- and micronutrients and physical properties. In contrast, application of INF increases crop yields by directly supplying plant nutrients required for crop growth. Fan et al. (2008) reported that FYM and NPK treatments increased wheat and maize yields significantly compared to no fertilizer control in a 26-year fertilizer management experiment in China's Loess Plateau. Zhang et al. (2009) reported that application of pig manure and NP fertilizers significantly enhanced grain yields of wheat and maize compared to that in CK from 1990 to 2006 in a red soil of Southern China. Similar effects of the applications of FYM and INF on crop yields have also been reported from the Rothamsted classical experiments (Jenkinson 1991).

Soil organic carbon and total nitrogen

Concentrations of SOC and TN in the 0 to 30 cm depth were significantly affected by the soil fertility management treatments (Fig. 2). Compared to CK, SOC in the FYM was 56.2, 46.3, and 14.0% more, and TN was 43.9, 29.1, and 27.6% more for 0–10, 10–20, and

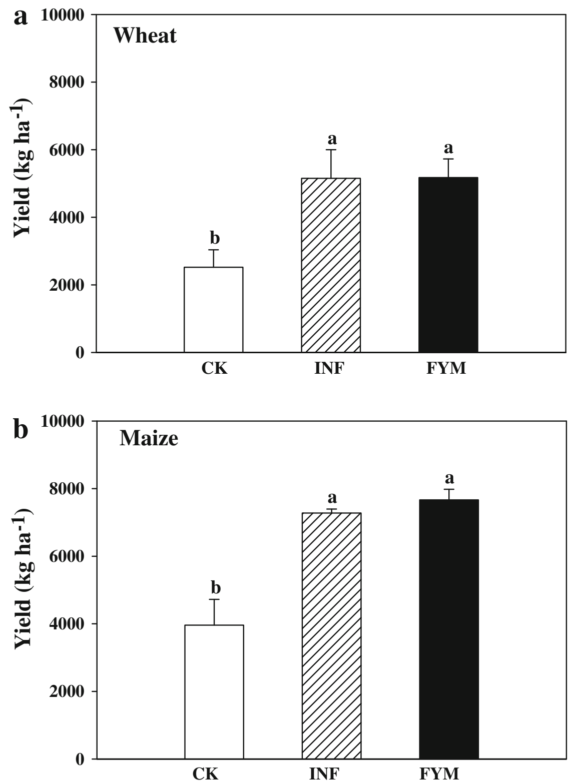

Fig. 1 Effect of farmyard manure (*FYM*) and inorganic fertilizers (*INF*) on the yields of wheat (**a**); maize (**b**) during 2007–2008. *Error bars* represent standard deviation. Values followed by different *lowercase letters* within depth are significantly different between tillage treatments ($P < 0.05$)

20–30 cm depths, respectively. These trends are attributed to more C being sequestered in the soil amended with FYM than in other treatments.

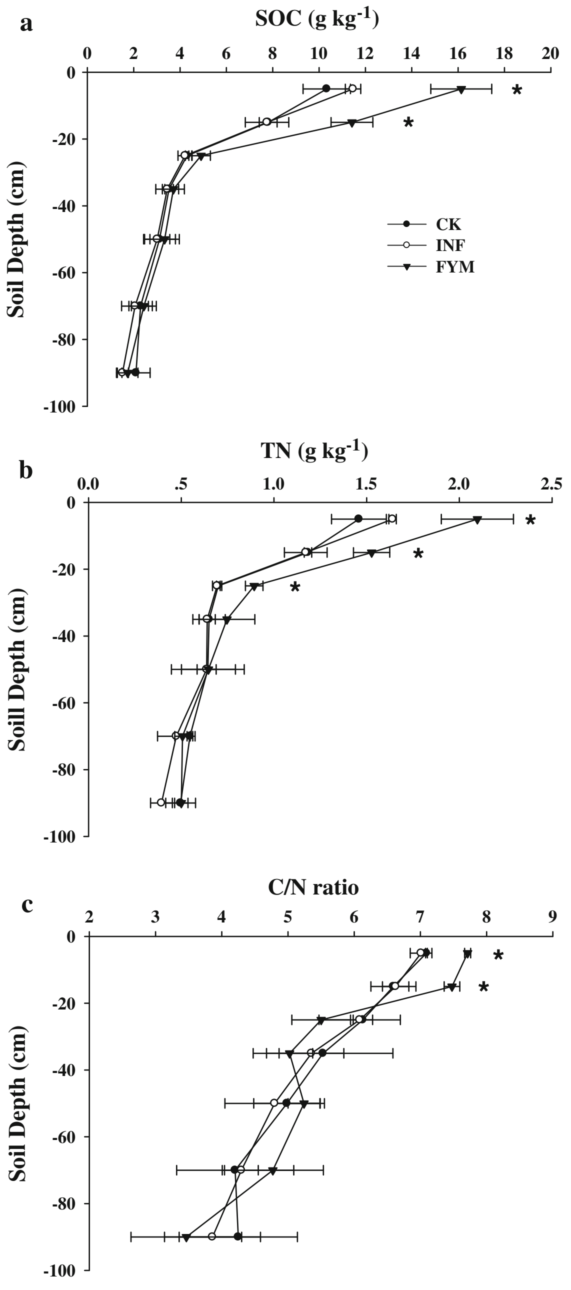

Fig. 2 Effect of farmyard manure (*FYM*) and inorganic fertilizers (*INF*) on the distribution of soil organic C (*SOC*) (**a**); total N (TN) (**b**); and C/N ratio (**c**). Soil depths were 0–10, 10–20, 20–30, 30–40, 40–60, 60–80, and 80–100 cm. *Error bars* represent standard deviation. *Significant differences between FYM and INF or CK at $P < 0.05$

However, consistent differences among treatments were not observed in the subsoil layers. These results suggest that beneficial effects of manuring are primarily limited to the surface layer. Hernandez-Ramirez et al. (2007) and Chen et al. (2009) also reported that the concentration of SOC by management (e.g. crop rotation, N source, conservation tillage) is primarily enhanced in the upper 15 or 30 cm layer, and accumulation of SOC in the subsoil is unaffected for one to two decades.

Increase in SOC and TN concentrations with application of FYM could be attributed to the regular addition of organic materials for 15 years, and also to a higher crop productivity and hence to return of more crop residues in the forms of roots and stubbles. Blair et al. (2006a) reported that FYM increased concentrations of SOC by 165% and TN by 151% compared to those in the control in the Broadbalk Wheat Experiment at Rothamsted, UK, established in 1843 on an Aquic/Typic Paleudalf. Banger et al. (2009) observed that a sandy soil amended with FYM contained 36.1% more SOC and 24.4% more TN concentrations than those in the CK in the 0–15 cm depth under a 16-year rice-cowpea cropping system in semi-arid tropics. Similar beneficial effects of FYM on SOC have been observed in other long-term experiments elsewhere (Blair et al. 2006b; Yan et al. 2007; Šimon 2008; Gong et al. 2009b). The magnitude of the effects, however, varies depending on the application rate of manure, soil texture, cropping system, climate, and duration of the experiment.

In general, inorganic fertilizers indirectly influence SOC concentration by increasing crop yields and thereby increasing the return of crop residues to the soil. In the same context, application of INF also enhances SOC sequestration (Manna et al. 2007; Purakayastha et al. 2008; Banger et al. 2009; Gong et al. 2009b). In contrast, the data for the present study indicate no differences in the concentrations of SOC and TN among INF and CK treatments throughout the 1-m depth (Fig. 2). The lack of significant differences in SOC concentration may be due to the low C input and the enhanced fertilizer-induced decomposition of SOC (Wu et al. 2004).

Furthermore, the application of INF did not significantly increase the TN concentration in comparison with that in the CK. This trend suggests that a part of mineral N applied may have been lost via ammonia volatilization (44.1% of applied N), leaching (14.8%), and denitrification (4.4%) in the wheat/maize system on the North China Plain, as was reported by Ju et al. (2009). Over and above the N contained in FYM, increase in TN concentration in the FYM treatment may also be attributed to a slow release of N from manure and thus lower losses of N, and higher biological N-fixation stimulated by FYM (Kundu et al. 2007).

There was a strong depth-dependency of SOC and TN concentrations in all three treatments (Fig. 2). The SOC and TN concentrations were much higher in the upper 20 cm layer (7.8–16.1 g C kg^{-1}, 1.17–2.10 g N kg^{-1}) than in the lower layers (20–100 cm) (1.52–4.91 g C kg^{-1}, 0.893–0.393 g N kg^{-1}). Higher SOC and TN concentrations in the top soil may be explained by the fact that they are strongly related to root C inputs, and that FYM and crop residues are often accumulated in the surface soils. Different inputs (kg ha^{-1} $year^{-1}$) in the present study for CK, INF and FYM treatments, respectively, were as follows: (1) crop residue input of 0, 14,704, and 15,483, (2) root biomass input of 3,430, 4,351, and 4,564, (3) crop residue C of 0, 6,416, and 6,757, (4) crop residue N of 0, 118, and 122, (5) root C of 1,364, 1,727, and 1,811, and (6) root N of 31, 35, and 36. The manure C and N inputs for FYM treatment were 6,840 kg C ha^{-1} $year^{-1}$ and 201 kg N ha^{-1} $year^{-1}$, respectively. Therefore, crop residues and manure contributed most towards the increase in SOC and TN concentrations in the top soil, followed by that through root C and N inputs.

The C:N ratio is indicative of the capacity of the soil to store and recycle nutrients (Šimon 2008). In general, the soil C:N ratio decreases with increase in depth irrespective of the treatments, ranging from 7.7 in the 0–10 cm depth to 3.5 in the 80–100 cm depth (Fig. 2). The C:N ratio of SOM is usually around 10–12 (Schlesinger 1995). The narrow C:N ratios in the present study suggest that frequent tillage operations might accentuate decomposition of SOM (Chen et al. 2007). As decomposition proceeds, C is released during respiration and some of the mineralized N is lost through leaching or gaseous emissions while some is reincorporated into the SOM pool (Chapin et al. 2002). The C:N ratios were significantly affected by fertilizer treatments only in the top 20 cm, and were significantly higher in the FYM than those in INF and CK in the 0–10 and 10–20 cm depths but did not differ among each other.

Labile soil organic carbon fractions

Fertility management treatments significantly affected the concentration of the labile SOC fractions in the top 20 cm, but not in the 20–30 cm depth (Fig. 3). In general, and for both 0–10 and 10–20 cm depths, FYM had significantly higher DOC, MBC, HWC, POC, $KMnO_4$–C concentrations than those in either INF or CK, which did not differ among each other (Fig. 3). Concentrations of labile C fractions in all treatments tended to decrease with depth (Table 2). Proportions of labile C fractions in SOC did not differ significantly among treatments at all three depths, except HWC/SOC at 10–20 cm, MBC/SOC and POC/SOC at 20–30 cm depths (Table 2). There were large differences in proportions among the five labile C fractions. Concentration was 1.5–3.0% for MBC, 0.7–1.1% for DOC, 2.0–2.8% for POC, 18.0–45.8% for HWC and 22.7–28.8% of SOC for $KMnO_4$–C (Table 2). These results are in accord with those reported by Powlson (1994), Blair and Lefory (1995), Haynes (2000), Šimon (2008), and Chen et al. (2009).

The DOC concentration was considerably smaller than those of other labile C fractions in the present study. Although DOC constitutes a small fraction of SOC, it plays an important role in numerous soil chemical and biological processes. It is the primary energy source for soil microorganisms, controls the nutrient turnover, and the development of microbial populations (Gong et al. 2009a). It is also the most mobile fraction of SOC and can reach almost all soil components by diffusion and convection (Lützow et al. 2007). The DOC percentage increased with increase in soil depth in all treatments (Table 2), which is in accord with the findings of others (Zhang et al. 2006; Wright et al. 2007). The illuviation or leaching may have increased the relative proportion of DOC in the subsurface layers. At 0–30 cm depth, DOC concentration was significantly higher in soil amended by FYM than those in INF or CK treatments (Fig. 3). Increases in DOC concentration with application of FYM have also been reported by Banger et al. (2010) and Gong et al. (2009b), probably because the FYM contains decayed soluble SOM (Liang et al. 1997).

Soil MBC is the living component of SOM, and it plays a critical role in nutrient cycling and SOM decomposition and transformation. Being highly dynamic, it is strongly influenced by soil management.

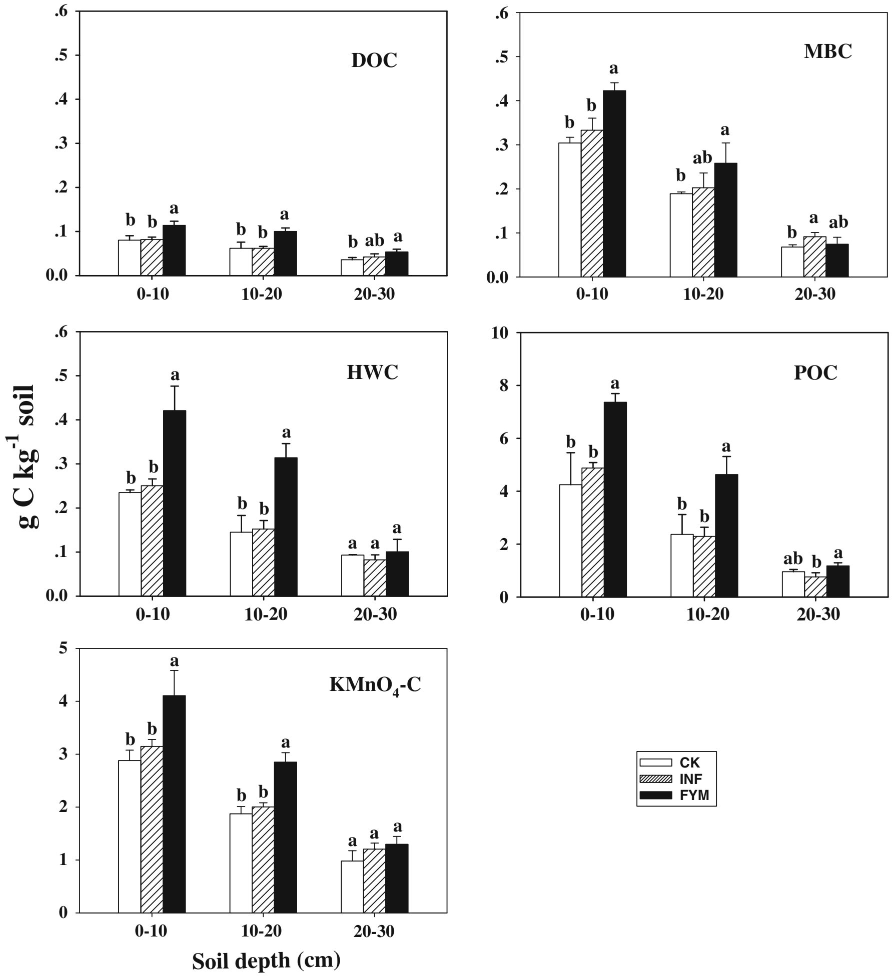

Fig. 3 Labile soil organic carbon (g C kg^{-1} soil): microbial biomass C (*MBC*), dissolved organic C (*DOC*), hot-water extractable C (*HWC*), permanganate oxidizable C (*$KMnO_4$–C*) and particulate organic C (*POC*) as effected by fertilizer treatments at 0–10, 10–20 and 20–30 cm depths. *CK* control, *INF* inorganic fertilizers, *FYM* farmyard manure. *Error bars* represent standard deviation. Values followed by different *lowercase letters* within depth are significantly different between tillage treatments ($P < 0.05$)

Thus, application of FYM for 15 years increased MBC concentration by 39% in the 0–10 cm depth, and by 37% in the 10–20 cm depth, as compared to that in the CK (Fig. 3). However, application of INF did not increase the concentration of MBC compared to that in the CK in the 0–20 cm depth. Apparently, the steady supply of the readily metabolizable C and N in FYM is likely to be the most influential factor contributing to the MBC increase, while microbial activity is limited by the reduced supply of organic substrates in the INF and CK treatments. In general, application of manure increases MBC (Yan et al. 2007; Šimon 2008; Banger et al. 2009; Gong et al. 2009b). However, effects of INF on MBC are inconsistent. Banger et al. (2010) and Gong et al. (2009b) reported that application of INF increased MBC compared to no fertilizer control. Similar to the present study, Šimon (2008) and Grego et al. (1998) also reported that addition of INF does not change MBC. In contrast, Okano et al. (1991) and Ghani et al. (2003) reported that application of N had an adverse impact on MBC concentration.

The hot-water extractable organic matter is supposed to contain MBC, soluble soil carbohydrates and amines (Ghani et al. 2003). It is frequently used as a measure for potentially bioavailable SOC and as an index of soil quality. Application of FYM significantly increased HWC concentration compared with those in either INF or CK in the 0–20 cm depth. The increase of HWC concentration by application of FYM is attributed to a higher plant biomass as well as added FYM. Similarly, Böhme and Böhme (2006) reported that application of FYM significantly increased HWC compared to the NPK or control in a loess-derived Chernozem in a 1902-initiated long-term ‘Static Fertilisation Experiment’, Bad Lauchstädt.

Table 2 Fertilizer induced changes in the percentage of labile soil organic carbon fractions (in % of SOC) in 0–10, 10–20 and 20–30 cm depths

Depth/treat.	MBC/SOC (%)	DOC/SOC (%)	HWC/SOC (%)	$KMnO_4$–C/SOC (%)	POC/SOC (%)
0–10 cm					
CK	2.96 (0.27) a	0.78 (0.03) a	2.29 (0.21) a	27.93 (0.99) a	40.81 (8.70) a
INF	2.91 (0.31) a	0.71 (0.03) a	2.18 (0.07) a	27.43 (0.76) a	42.58 (2.64) a
FYM	2.63 (0.25) a	0.71 (0.06) a	2.61 (0.28) a	25.46 (2.45) a	45.77 (3.66) a
10–20 cm					
CK	2.42 (0.13) a	0.79 (0.15) a	1.87 (0.38) b	24.00 (0.70) a	30.10 (7.95) a
INF	2.61 (0.26) a	0.80 (0.06) a	1.97 (0.12) b	26.05 (2.63) a	29.51 (1.03) a
FYM	2.25 (0.22) a	0.88 (0.09) a	2.75 (0.13) a	25.07 (2.57) a	40.61 (5.66) a
20–30 cm					
CK	1.58 (0.08) b	0.84 (0.11) a	2.16 (0.04) a	22.72 (4.20) a	22.28 (1.59) ab
INF	2.18 (0.37) a	1.02 (0.24) a	1.97 (0.43) a	28.80 (4.59) a	17.98 (2.97) b
FYM	1.51 (0.20) b	1.09 (0.06) a	2.03 (0.42) a	26.57 (3.86) a	24.12 (3.32) a

Values are means with the standard deviation in parenthesis (n = 3). Values within a column followed by different lowercase letters are significantly different ($P < 0.05$). *CK* control, without any farmyard manure or inorganic fertilizers, *INF* inorganic fertilizers, *FYM* farmyard manure, *MBC* microbial biomass C, *DOC* dissolved organic C, *HWC* hot-water extractable C, *$KMnO_4$–C* permanganate oxidizable C, *POC* particulate organic C

The POM fraction is dominated by plant-derived remains and microbial and micro faunal debris, and acts as an energy source for microorganisms and as a reservoir of relatively labile SOM and plant nutrients (Janzen et al. 1992; Christensen 2001). It is highly sensitive to management practices (Six et al. 1999; Chen et al. 2009). In the present study, the soil amended by FYM contained significantly higher POC in the top 20 cm than that in the INF or CK treatments (Fig. 3). The POC represents the recent C inputs, the left-over crop residues and FYM might contribute to this labile SOC pool in the FYM treated soil. Banger et al. (2010) also reported that FYM treatment was the most effective in increasing POM when compared with chemical fertilizer treatments (N, NP, NPK) and CK, after 16 years of fertilization to rice—cowpea rotation system in a sandy loam Typic Rhodalfs in semi-arid tropics.

The measurement of C released by the oxidation of soil with 333 mM $KMnO_4$ is a rapid and an economical method to quantify labile SOC fraction (Blair and Lefory 1995; Weil et al. 2003). The $KMnO_4$–C is a sensitive indicator of changes in the SOC caused by different management practices (Weil et al. 2003). In the present study, $KMnO_4$–C concentration in 0–20 cm depth was significantly higher in the soil amended with FYM than those by INF and CK treatments (Fig. 3). Application of FYM and enhanced crop production might be responsible for increased $KMnO_4$–C. Similarly, Gong et al. (2009b) reported that application of FYM was more effective in increasing $KMnO_4$–C compared to INF. The concentration of $KMnO_4$–C ranged in the order FYM > 1/2 FYM-N > balanced fertilizer NPK > unbalanced fertilizers (NP, PK, NK) > CK, after 18 years of fertilization under a wheat-maize cropping system in Henan province, China. Rudrappa et al. (2006) and Purakayastha et al. (2008) also reported that application of FYM along with NPK resulted in the highest accumulation of $KMnO_4$–C while the lowest value was observed in the control in a long-term field experiment in maize-wheat-cowpea cropping system established in 1971, in semi-arid sub-tropical region of India.

Sensitivity of labile C fractions for evaluating changes in soil organic carbon

The data on labile C fractions can be used to distinguish any fertilizer-induced changes in SOC concentration. The results of present study show that concentrations of MBC, DOC, HWC, $KMnO_4$–C, and POC were significantly higher in the FYM treatment than those in INF or CK in the upper 20 cm depth (Fig. 3). Thus, the labile fractions are sensitive indicators of detecting changes in SOC in the medium term—within 1–2 decades. However, the magnitude of changes in the labile C fractions differed among FYM and CK treatments. The HWC (79.1–116.6%) and POC (73.4–95.6%) fractions were the most sensitive to the effects of fertilizer than was SOC which increased by only 46.4–56.2% (Table 3). The lower sensitivity of MBC and DOC compared to that of POC and HWC might be due to their smaller sizes, highly labile nature, and the mobility of DOC (Janzen et al. 1992; Chen et al. 2009). While the lower sensitivity of $KMnO_4$–C might be due to the presence of slowly cycling SOC in addition to that of the labile C pool (Weil et al. 2003). Similar results have been reported by other researchers indicating that HWC and POC are more sensitive than SOC to changes in the tillage regimes (Chen et al. 2009), and in fertilizer treatments (Yan et al. 2007; Šimon 2008).

Table 3 Sensitivity index of the labile soil organic C fractions in 0–10 and 10–20 cm depths

Treatments	SOC	MBC	DOC	HWC	$KMnO_4$–C	POC
0–10 cm						
INF	11.0	9.5	1.7	6.6	9.2	14.8
FYM	56.2	39.1	41.7	79.1	42.7	73.4
10–20 cm						
INF	-0.6	7.1	0.0	5.0	6.9	-3.2
FYM	46.4	36.5	61.8	116.6	52.1	95.6

INF inorganic fertilizers, *FYM* farmyard manure, *MBC* microbial biomass C, *DOC* dissolved organic C, *HWC* hot-water extractable C, *$KMnO_4$–C* permanganate oxidizable C, *POC* particulate organic C

Correlations between SOC and labile C fractions

The entire data collected from each treatment, depth and replication were pooled together to compute the correlations among SOC fractions. The SOC concentrations were significantly and positively correlated with labile fractions: MBC, DOC, HWC, $KMnO_4$–C, and POC in the 0–10 and 10–20 cm layers (Table 4). In general, there were no significant correlations between these selected parameters in the 20–30 cm layer (data not shown). These correlations indicated that SOC was a major determinant of the labile C fractions present. On the other hand, the improvement or depletion in the labile C fractions could also provide an effective early warning of changes in SOC. The labile fractions MBC, DOC, HWC, POC, and $KMnO_4$–C were significantly and positively correlated with each other in the 20 cm layer (Table 4), suggesting that they are closely interrelated. Chen et al. (2009) also observed that MBC, POC, DOC, $KMnO_4$–C, HWC and SOC were all positively correlated with each other. Gong et al. (2009b) reported that MBC, water-soluble organic C, $KMnO_4$–C, C mineralization and SOC were significantly correlated with each other. Significant positive

Table 4 Pearson correlation among selected parameters at 0–10 and 10-20 cm depths

	SOC	TN	MBC	DOC	HWC	$KMnO_4$–C	POC
0–10 cm							
SOC	1						
TN	0.991**	1					
MBC	0.888**	0.866**	1				
DOC	0.941**	0.918**	0.780*	1			
HWC	0.950**	0.922**	0.833**	0.950**	1		
$KMnO_4$–C	0.942**	0.938**	0.788*	0.959**	0.975**	1	
POC	0.933**	0.925**	0.861**	0.923**	0.870**	0.878**	1
10–20 cm							
SOC	1						
TN	0.991**	1					
MBC	0.883**	0.903*	1				
DOC	0.910**	0.866**	0.720*	1			
HWC	0.962**	0.950**	0.850**	0.930**	1		
$KMnO_4$–C	0.921**	0.893**	0.706*	0.947**	0.932**	1	
POC	0.937**	0.906**	0.763*	0.975**	0.916**	0.957**	1

* Significant at the 0.05 level; ** Significant at the 0.01 level

MBC microbial biomass C, *DOC* dissolved organic C, *HWC* hot-water extractable C, *$KMnO_4$–C* permanganate oxidizable C, *POC* particulate organic C relationships of HWC, MBC, water-soluble C and SOC were also reported by Ghani et al. (2003).

Application of FYM increases SOC, and it also exacerbates N_2O emissions (Jones et al. 2007). Based on the comprehensive consideration of soil fertility, environment and crop yields, the combined application of FYM and INF is a sustainable soil management option for the wheat-maize cropping system in the North China Plain. Several studies have indicated that long-term and balanced application of chemical fertilizers and organic manure can generally achieve higher crop yields and improve soil quality than any of these applied alone (Rudrappa et al. 2006; Manna et al. 2007; Fan et al. 2008).

Conclusions

Continuous application of FYM for 15 years significantly increased SOC and TN concentrations, and disproportionally increased DOC, MBC, HWC, $KMnO_4$–C, POC in the 0–20 cm layer compared to those under INF and CK for intensively cultivated winter wheat-summer maize cropping system in the North China plain. Further, application of inorganic fertilizer alone was not sufficient to increase SOC and labile C fractions over that of CK. Labile C fractions were highly sensitive to changes in SOC, with POC and HWC being the most sensitive fractions to applications of FYM.

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