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Assessing carbon lability of particulate organic matter from $\delta^{13}C$ changes following land-use change from C_3 native vegetation to C_4 pasture

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Abstract. Land-use change from C_3 vegetation ($\delta^{13}C$ values, –30‰ to –24‰) to C_4 vegetation ($\delta^{13}C$ values, –14‰ to –11‰) provides a useful quantitative technique for estimating organic C turnover in soil, even when total organic C changes are negligible. We utilised this technique to estimate C turnover in physically fractionated soil organic matter, particulate organic matter C (POM C >250 μm fraction and POM C 250–53 μm fraction), and the <53 μm fraction. There were small changes in total soil organic C (SOC) after 23 years of land-use change from native vegetation (mixed vegetation of *Acacia harpophylla* and *Casuarina cristata*) to buffel grass (*Cenchrus ciliaris* L. cv. Biloela) pasture grown on Vertosol–Dermosol–Sodosol soil types. The SOC values (t/ha) under native vegetation were: 31 ± 3 for the 0–0.1 m depth, 21 ± 1 for the 0.1–0.2 m depth, 15 ± 3 for the 0.2–0.3 m depth, and 16 ± 2 for the 0.3–0.4 m depth; the corresponding SOC values under pasture were 25 ± 2, 19 ± 2, 14 ± 2, and 13 ± 1 t/ha. The respective $\delta^{13}C$ values in 0–0.1 m depths of the whole SOC and POM C >250 μm fraction changed from -25.5 ± 0.1‰ and -25.5 ± 0.3‰ under native vegetation to -20.1 ± 0.5‰ and -19.4 ± 0.2‰ under pasture. Similar, although smaller, differences were observed for other depths and SOC fractions. The SOC turnover periods (years) were 31 ± 6 for the 0–0.1 m depth, 60 ± 5 for the 0.1–0.2 m depth, 55 ± 15 for the 0.2–0.3 m depth, and 63 ± 20 for the 0.3–0.4 m depth; the corresponding turnover periods for the POM C >250 μm fraction were 13 ± 2, 19 ± 5, 14 ± 4, and 12 ± 5 years. The turnover periods of SOC in the POM C 250–53 μm and <53 μm fractions were similar to, or longer than, for the whole SOC at all depths studied. Thus, the lability of the SOC and SOC pools was in the order: POM C >250 μm fraction > POM C 53–250 μm fraction = POM C <53 μm fraction = whole SOC.

Additional keywords: C_3, C_4, $\delta^{13}C$, labile C, particulate organic matter C, soil organic C.

Introduction

Land-use change from native vegetation to (introduced) pasture has shown inconsistent impact (increase, decrease, or no change) on soil organic C (SOC) stocks (Murty *et al*. 2002). Harms *et al*. (2005) studied the effect of land-use change from native vegetation to pasture at 32 paired sites in southern and central Queensland, Australia. Although SOC stocks declined by ~7% across all sites, significant losses in SOC stocks were found mostly in coarse-textured soils such as Kandosols but not in Sodosols and Vertosols. These observations were confirmed for other Kandosols by Dalal *et al*. (2005). In Vertosol–Dermosol–Sodosol soil types, Radford *et al*. (2007) observed no change in SOC from land-use change under native vegetation to that developed for buffel pasture, even after 21 years, thus corroborating the observations of Harms *et al*. (2005). Chevallier *et al*. (2006) suggested that SOC is protected against decomposition with pasture establishment on a Vertosol.

Since SOC is heterogeneous in nature, it is likely that the amounts of labile fractions of SOC may change due to changes in the amount and nature of C inputs, even though the total amount of SOC remains essentially unaffected. Cambardella and Illiott (1992) suggested that the soil organic matter C fraction >53 μm could be used as the labile fraction in grasslands and cultivated soils since it was more sensitive to change in management than the whole soil organic matter C. Thus, Skjemstad *et al*. (2004) used the >53 μm fraction as the particulate organic mater (POM) C in the Roth-C model to simulate organic C turnover in cropping soils. However, Zach *et al*. (2006) found that the POM C >53 μm fraction was no more sensitive as an indicator to change in management than the whole soil in both pasture and cultivated soils. This was also confirmed by Dou *et al*. (2008) for cultivated soils, where they found that no labile SOC pool, including the POM C >53 μm fraction, was more sensitive than the whole SOC for soils subjected to different tillage practices.

Six *et al*. (2002) proposed a comprehensive and detailed soil physical fractionation scheme which isolates >10 fractions, some of which contain relatively more labile C than others, but it is laborious and tedious to use routinely to isolate the POM C labile fraction. However, Tisdall and Oades (1982) surmised that

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macroaggregates >250 μm may be considered as the labile C fraction since they largely depend on transient binding agents such as roots and hyphae, which decay rapidly, and hence are subject to management effects, whereas microaggregates <250 μm are stabilised by persistent binding agents and thus relatively stable against management, and land-use change. Chevallier *et al*. (2004) found that almost 50% of the mineralisable SOC in a Vertosol under pasture was present in macroaggregates, although it comprised only 10% of the total SOC. Thus, separation of SOC into macroaggregate >250 μm fraction (labile fraction) and microaggregate <250 μm fraction (non-labile fraction) is a much simpler approach than that of Six *et al*. (2002). The procedure of Cambardella and Illiott (1992) for estimating the POM C >53 μm fraction can be easily adapted to sequentially separate the POM C >250 μm fraction, in addition to the POM C 250–53 μm fraction, from soil organic matter.

Change in land use from C_3-dominant vegetation to C_4-dominant vegetation provides analytical tools to differentiate C_3-derived C (native vegetation) from C_4-derived C in the whole SOC and C fractions, since natural abundance ^{13}C isotopic signatures of the former ($\delta^{13}C$ values, –30‰ to –24‰) differ from those of the latter ($\delta^{13}C$ values, –14‰ to –11‰) (Balesdent *et al*. 1987; Skjemstad *et al*. 1994). Thus, $\delta^{13}C$ analysis can be used to estimate rates and turnover periods of SOC and SOC fractions in soil subject to land-use change from C_3 native vegetation to C_4 pasture.

The objectives of this study were to: (*i*) assess the changes in stocks of SOC and SOC fractions under native vegetation and pasture; (*ii*) estimate turnover periods of SOC and its fractions under pasture; and (*iii*) compare the lability of various SOC fractions from their turnover periods in soil under pasture as estimated by $\delta^{13}C$ values.

Materials and methods

Sampling site

The study site is at 24.81°S, 149.80°E, at an altitude of 151 m above sea level. Mean annual rainfall is 720 mm and annual potential evaporation is 2100 mm. The mean maximum temperature is 33.1°C in January and minimum temperature is 6.5°C in July (Cowie *et al*. 2007).

The main soil types at the site are Grey Vertosols/Dermosols and Sodosols (Isbell 2002) or Endosodic Calcic Vertisol and Albic Vertic Luvisol (IUSS Working Group WRB 2006). Clay contents of Vertosols varied from 36% at 0–0.1 m depth to 44% at 0.2–0.3 m depth, while those of Sodosols for the corresponding depths were 18% and 28% clay. Soil pH (1 : 5 soil : water) of Vertosols varied from 6.6 (0–0.1 m depth) to 8.1 (0.2–0.3 m depth), while soil pH of Sodosols varied from 6.8 (0–0.1 m depth) to 7.5 (0.2–0.3 m depth) across the site. The dominant native vegetation at the site is brigalow (*Acacia harpophylla*); belah (*Casuarina cristata*) and black butt (*Eucalyptus cambageana*) are the co-dominant species.

Part of the site was cleared of native vegetation by chain-pulling of trees in March 1982, allowed to dry, and then burned *in situ* in October 1982. It was sown to buffel pasture (*Cenchrus ciliaris* cv. Biloela) in the ash bed (ensuring minimum soil disturbance) in November 1982. Therefore, the native vegetation and grazing portions of the study site were essentially similar. Grazing of the pasture commenced a year later and the animal stocking rate was adjusted to maintain ground cover >85% during the experimental period (1983–2005) (Radford *et al*. 2007). Detailed description of the site is available from Cowie *et al*. (2007).

Soil sampling and particulate organic matter fractionation

Soil samples were taken in November 2005 at three monitoring sites each on native vegetation and pasture. At each site, five samples were taken down to 0.4 m depth by a hydraulic-driven corer (50 mm diameter) and divided into 0–0.1, 0.1–0.2, 0.2–0.3, and 0.3–0.4 m depths and bulked for respective depths. Soil samples were dried at 40°C and ground to pass a <2 mm sieve for soil pH, particle size analysis, and C fractionation. Intact soil cores from each depth were also taken for bulk density measurements. These samples were dried at 105°C to a constant weight, and bulk density was calculated from the oven-dry mass of soil and internal core volume.

The fractionation procedure for POM was essentially that of Cambardella and Illiott (1992), modified to separate SOC into >250, 250–53, and <53 μm fractions. Briefly, a 10-g soil sample (<2 mm size) was dispersed in 30 mL sodium hexametaphosphate solution (5 g/L) by shaking for 15 h on a reciprocal shaker. The suspension was passed through a stacked set of sieves 250 and 53 μm, rinsed with deionised water several times, and the materials retained on the sieves was collected (>250 and 250–53 μm fractions) and dried at 60°C for 3 days, weighed, and ground to <0.1 mm size for C and $\delta^{13}C$ measurement by isotope ratio mass spectrometry.

Soil analysis for carbon and $\delta^{13}C$

Total soil organic C (TOC), particulate organic C, and natural abundance ^{13}C of soil and soil fraction samples were determined using an Isoprime isotope ratio mass spectrometer (IRMS) coupled to a Eurovector elemental analyser (Isoprime-EuroEA 3000) with 10% replication. Samples containing inorganic carbonates were pretreated with HCl before analysis. The isotope ratios were expressed using the 'delta' notation (δ), with units of per mL or parts per thousand (‰), relative to the marine limestone fossil Pee Dee Belemnite standard (Craig 1953) for $\delta^{13}C$, using the relationship in Eqn 1 below:

$$\delta^{13}C\ (‰) = (R_{sample}/R_{standard} - 1) \times 1000 \qquad (1)$$

where R is the molar ratio of $^{13}C/^{12}C$ of the sample or standard (Ehleringer *et al*. 2000).

The proportion of organic C in soil derived from C_4 vegetation can be estimated by using $\delta^{13}C$ data and a mixing model (Boutton 1996; Bekele and Hudnall 2003; Dalal *et al*. 2005), as in Eqn 2 below:

$$\text{Soil } C_4\text{-derived C} = (\delta^{13}C_{\text{soil under pasture}} - \delta^{13}C_{\text{C3 soil under native vegetation}}) / (\delta^{13}C_{C4} - \delta^{13}C_{\text{C3 soil under native vegetation}}) \qquad (2)$$

where $\delta^{13}C_{\text{soil under pasture}}$ is the $\delta^{13}C$ value of SOC under pasture, $\delta^{13}C_{C4}$ is the average value of $\delta^{13}C$ of C_4 buffel shoots and roots (–12.5‰; Dalal *et al*. 2005), and

$\delta^{13}C_{C3\ soil\ under\ native\ vegetation}$ is the $\delta^{13}C$ value of soil C under C_3 native vegetation. The proportion of previous vegetation, C_3-derived C in the soil under pasture is the difference between the total SOC stock under pasture and the amount of C_4-derived C in the pasture soil. Similar calculations were made for the C_4-derived C in the pasture soil at individual depths and in different fractions, using the corresponding $\delta^{13}C$ values. Thus, no corrections were required for Rayleigh fractionation of enrichment of $\delta^{13}C$ values with soil depth.

Turnover of carbon in whole soil and soil fractions

The rate of loss of native vegetation, C_3-derived C in the whole soil and soil fractions after 23 years under pasture was calculated as follows (Dalal *et al.* 2005):

$$k = -(\ln C_t/C_o)/t \quad (3)$$

where C_o and C_t are the amounts of C_3-C initially under native vegetation and at time t, 23 years under pasture after clearing of native vegetation, respectively, and k (1/year) is the rate of loss of C_3-C from soil and soil fractions. Turnover period is the reciprocal of k (1/k, year).

Statistical analysis

The results are presented as mean values with standard errors. ANOVA was used to test the differences in SOC and SOC fractions between native vegetation and pasture soils. Fisher's protected least significant difference (l.s.d.) was used to determine significant differences in SOC and different size fractions in soil under native vegetation and pasture at $P = 0.05$.

Results and discussion

Soil bulk density was higher under 23-year-old pasture soil ($1.30 \pm 0.01\ Mg/m^3$) than under native vegetation ($1.19 \pm 0.02\ Mg/m^3$) in the top 0–0.1 m depth. Below this depth, bulk density of soil was similar under both land uses. The cumulative amounts of SOC for 0–0.1, 0–0.2, 0–0.3, and 0–0.4 m depths were, therefore, corrected for equivalent soil mass to that under native vegetation, using the polynomial relationship ($r^2 > 0.99$) between the amount of soil carbon and soil mass (soil depth × bulk density) (Dalal *et al.* 2005). Therefore, soil depths reported here refer to those of native vegetation.

Carbon stocks in whole soil and soil fractions

The amounts of SOC under native vegetation were 31 t/ha at 0–0.1 m depth, 21 t/ha at 0.1–0.2 m depth, 15 t/ha at 0.2–0.3 m depth, and 16 t/ha at 0.3–0.4 m depth; SOC stocks under pasture were slightly lower but not significantly different from those under native vegetation (Table 1). The cumulative amounts of SOC at the 0–0.3 m depths (Fig. 1) were similar under both land uses. Harms *et al.* (2005) measured SOC stocks from 34 to 77 t C/ha in the 0–0.3 m depths in fine-textured Vertosols/ Dermosols under *A. harpophylla* and *A. cambagei* vegetation; SOC stock (67.7 t C/ha) under native vegetation in the present study, therefore, is within the range of these values. Similarly, SOC stocks under pasture varied from 30 to 59 t C/ha in the study by Harms *et al.* (2005); again, the mean SOC stock under pasture in our study (58.3 t C/ha) is within the range of these values for similar soils.

Table 1. Soil organic carbon (SOC) stocks and $\delta^{13}C$ values in soil under native vegetation, and after 23 years of perennial pasture (corrected for equivalent soil mass) ($n = 3$)

Values are compared at a given depth between native vegetation and pasture soil for SOC or $\delta^{13}C$. Means followed by the same letter are not significantly different at $P = 0.05$

Depth (m)	SOC (t/ha)		SOC $\delta^{13}C$ (‰)	
	Native vegetation	Pasture	Native vegetation	Pasture
0–0.1	31.0 ± 2.9a	25.1 ± 1.7a	–25.5 ± 0.1a	–20.1 ± 0.5a
0.1–0.2	21.4 ± 1.2a	18.9 ± 2.4a	–22.7 ± 0.2a	–20.2 ± 0.7b
0.2–0.3	15.3 ± 2.7a	14.3 ± 1.9a	–22.4 ± 0.4a	–19.4 ± 0.7b
0.3–0.4	16.3 ± 1.8a	13.3 ± 0.7a	–18.7 ± 0.4a	–16.8 ± 1.5a

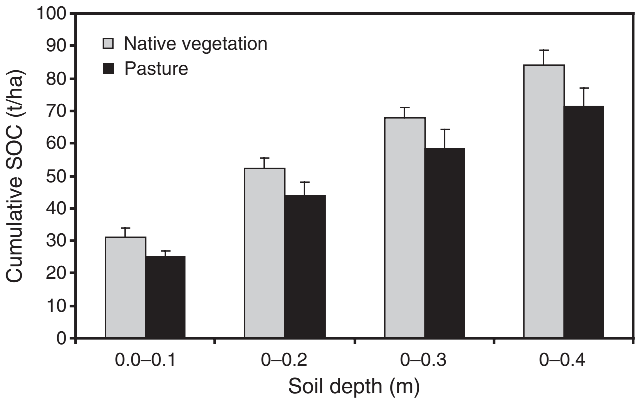

Fig. 1. Cumulative soil organic carbon (SOC) under native vegetation and after 23 years of pasture ($n = 3$). SOC stocks for pasture were calculated on equivalent soil mass basis of native vegetation for all depths according to the procedure of Dalal *et al.* (2005).

We found no significant effect of land-use change on SOC stocks between native vegetation and pasture. Earlier at this site, Radford *et al.* (2007) also found no significant difference in SOC under native vegetation and buffel grass pasture after 21 years, although the buffel grass pasture contained slightly lower SOC concentrations throughout the experimental period, similar to that found in our study. Harms *et al.* (2005) observed no significant difference in SOC under brigalow or gidgee (*Acacia cambagei*) vegetation and adjacent pasture in central Queensland. This also confirms the meta-analysis of 109 studies, where the average change in SOC due to land-use change from native vegetation to pasture across all sites did not differ significantly from zero although the SOC values varied by 10% (Murty *et al.* 2002). Even small increases in SOC stocks are reported in soils converted to pasture after deforestation in the Brazilian Amazon (Desjardins *et al.* 2004), especially after phosphorus (P) fertiliser application to originally P-deficient soils.

The proportion of POM C in the >250 µm fraction was >3-fold higher under native vegetation than in the pasture soil (43% *v.* 12%), but in the <53 µm fraction it was lower under native vegetation than in pasture soil (41% *v.* 70%) at 0–0.1 m depth (Fig. 2). The proportion of POM C in the 250–53 µm

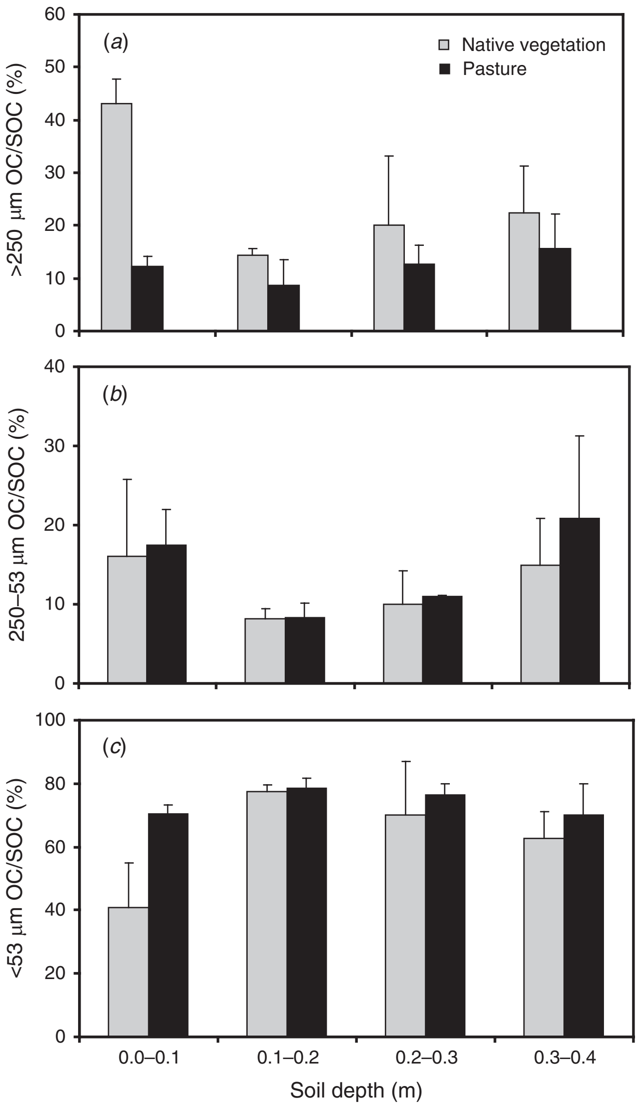

Fig. 2. Proportion of soil organic carbon (SOC) in >250, 250–53, and <53 μm fractions under native vegetation and after 23 years of pasture (n = 3). SOC stocks for pasture were calculated on equivalent soil mass basis of native vegetation for all depths.

fraction was similar under both native vegetation and the pasture at all depths (Fig. 2). Martin *et al*. (1990) found that POM C in the >250 μm fraction was decreased by 97%, compared to <50% from the <50 μm fraction, after 16 years of vegetation change in a tropical Ferrosol. Lobe *et al*. (2005) also found that the >250 μm fraction of POM C was most affected due to land-use change from savanna to arable farming. Conversely, Feller *et al*. (2001) observed that a greater proportion of the labile SOC was present in >200 μm aggregates in Vertosols. Apparently, SOC in the >200 μm fraction was present primarily as plant debris characterised by cellulosic sugars (Larré-Larrouy *et al*. 2003).

The δ^{13}C values of the whole SOC were enriched by 4.4‰ under pasture at 0–0.1 m depth and tended to be enriched at all the soil depths compared with that under native vegetation, even though total SOC stocks were similar under both land uses (Table 1). Desjardins *et al*. (2004) found that δ^{13}C values of the whole SOC increased by 5.5‰ in Central Amazonia and 7.5‰ in Eastern Amazonia after 15 years of pasture establishment, with only a small increase in SOC stock, especially in Eastern Amazonia.

The δ^{13}C values of POM C in the >250 μm fraction under pasture were more enriched than those in whole SOC, by 4–7‰, at all depths compared with those under native vegetation (Table 2). Similar trends were observed for the POM C 250–53 μm fraction and <53 μm fraction in the top 0–0.1 m depth, but less so at 0.3 –0.4 m depth (Table 2). The proportion of C_4-C (buffel pasture derived C) was the highest at 0–0.1 m depth (41.5 ± 4.2%), but it was still substantial below 0.1 m depth. At 0.3–0.4 m depth, it varied considerably (30.1 ± 25.0%). Dalal *et al*. (2005) found a C_4-derived C contribution to SOC from 23% at 0.2–0.3 m depth to 31% in the 0.05–0.1 m depth after 20 years of pasture in a Kandosol in southern Queensland, the values being similar to those measured in this study below 0.1 m depth. The higher C_4-derived C contribution to SOC in the top 0.1 m depth in this study than that measured by Dalal *et al*. (2005) on a Kandosol was possibly due to better pasture growth and hence higher C input to soil in the higher rainfall zone in this study (720 mm) compared with their site in a drier region (516 mm). Desjardins *et al*. (2004) also measured up to 45% pasture-derived C in Eastern Amazonia after 15 years of pasture establishment, a value similar to our study (41.5%).

Turnover periods of carbon in whole soil and soil fractions

The turnover period of SOC in the whole soil at 0–0.1 m depth was 31 years and it almost doubled at 0.1–0.2 m depth, and then remained similar at 0.2–0.3 and 0.3–0.4 m depths (Table 3). Dalal *et al*. (2005) estimated similar turnover

Table 2. δ^{13}C (‰) of particulate organic matter (POM) C >250 μm, POM C 250–53 μm, and <53 μm fraction C under native vegetation, and after 23 years under perennial pasture (n = 3)

Values are compared at a given depth between native vegetation and pasture soil for each soil fraction. Means followed by the same letter are not significantly different at P = 0.05

Depth (m)	δ^{13}C of POM C >250 μm		δ^{13}C of POM C 250–53 μm		δ^{13}C of soil fraction <53 μm	
	Native	Pasture	Native	Pasture	Native	Pasture
0.0–0.1	–25.5 ± 0.3a	–19.4 ± 0.2b	–25.1 ± 0.2a	–19.9 ± 0.2b	–25.4 ± 0.2a	–20.2 ± 0.7b
0.1–0.2	–25.2 ± 0.5a	–21.4 ± 1.0b	–24.3 ± 0.5a	–20.6 ± 0.4b	–22.0 ± 0.3a	–19.8 ± 1.1b
0.2–0.3	–21.5 ± 2.5a	–14.3 ± 3.0b	–23.4 ± 0.8a	–19.9 ± 1.1b	–21.9 ± 0.8a	–19.5 ± 0.5b
0.3–0.4	–20.0 ± 3.5a	–16.3 ± 2.5a	–21.1 ± 2.1a	–18.5 ± 1.5a	–20.2 ± 3.0a	–16.4 ± 3.3a

Table 3. Turnover periods (years) of soil organic carbon (SOC) and SOC fractions under pasture ($n = 3$)

POM, Particulate organic matter. At a given depth, means followed by the same letter are not significantly different at $P = 0.05$

Depth (m)	Whole soil	POM C >250 µm	POM C 250–53 µm	<53 µm fraction
0–0.1	31.1 ± 5.9a	11.5 ± 1.4b	45.7 ± 22.9ac	53.5 ± 8.6c
0.1–0.2	60.2 ± 5.4a	18.6 ± 5.6b	46.7 ± 28.4a	66.9 ± 13.5a
0.2–0.3	55.4 ± 15.3a	13.7 ± 5.4b	74.2 ± 38.4a	78.2 ± 18.3a
0.3–0.4	62.8 ± 19.8a	12.3 ± 4.9b	93.8 ± 52.4ac	105.4 ± 22.2c

periods of SOC under buffel pasture in a Kandosol (29 years) at 0–0.05 m depth.

The POM C in the >250 µm fraction turned over fastest at all depths (12–19 years), some 3–5 times faster than the whole soil (Table 3). Dalal *et al*. (2005) measured similar turnover periods of the light fraction (<1.6 Mg/m^3) in a Kandosol (14–24 years). This is not surprising, considering that POM and light fraction organic matter are essentially similar in their rates of turnover (Cambardella and Illiott 1992). Further, Dalal and Mayer (1986) found that SOC in the light fractions was lost 2–11 times faster than in the heavy fractions (>2 Mg/m^3) of five Vertosols and a Kandosol subjected to continuous cultivation for up to 70 years.

The turnover periods of POM C in the 250–53 µm fraction varied from 46–47 years at 0–0.1 and 0.1–0.2 m depths, to 94 years at 0.3–0.4 m depth. The turnover periods of SOC in the <53 µm fractions were similar to, or slightly longer than, in the 250–53 µm fraction: 54 years at 0–0.1 m depth and 105 years at 0.3–0.4 m depth (Table 3). Liao *et al*. (2006) found rapid turnover of SOC in the >250 µm fraction not protected within aggregates, while SOC in the 250–53 µm fraction, whether within macroaggregates or microaggregates, turned over almost 50% slower than the former following woody plant invasion of grassland. Martin *et al*. (1990) also found that the turnover of SOC in the >250 µm fraction was much greater than in the <20 µm fraction and other intermediate-size SOC fractions after 16 years of vegetation change in a tropical Ferrosol. Similarly, Chevallier *et al*. (2004) found that almost 50% of the mineralisable SOC in a Vertosol under pasture was present in macroaggregates >200 µm, although it comprised only 10% of the total SOC. Therefore, considering the extent of lability of SOC fractions based on its turnover periods, POM C in the >250 µm fraction was the most labile faction, while the lability of POM C in the 250–53 µm fraction was similar to that of SOC in the <53 µm fractions and the whole soil at all depths examined.

Conclusion

Land-use change from native brigalow vegetation (C_3) to buffel pasture (C_4) had a small effect on SOC stocks in the 0–0.4 m depths after 23 years. However, over this period, the contribution of buffel pasture C to the total SOC stock was 42% in the top 0–0.1 m depth and up to 30% in the 0.1–0.4 m depths. Although total SOC stocks were essentially similar under both land uses, proportion of POM C in the >250 µm fraction under pasture was almost 30% that of the native vegetation in the top 0–0.1 m depths, while that in the 250–53 µm fraction was similar under both land uses. Moreover, POM C in the >250 µm fraction turned over 3–5 times faster than the whole soil and 4–8 times faster than the POM C in the 250–53 µm and <53 µm fractions. Therefore, the lability of the SOC and SOC pools in the Vertosol–Dermosol–Sodosol soil types at the study site was in the order: POM C >250 µm fraction > POM C 53–250 µm fraction = POM C <53 µm fraction = whole SOC.

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