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Effects of weed control and fertilization on soil carbon and nutrient pools in an exotic pine plantation of subtropical Australia

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Abstract
Purpose Soil carbon (C) and nutrient pools under different plantation weed control and fertilizer management treatments were assessed in a 7-year-old, F_1 hybrid (*Pinus elliottii* var. *elliottii* × *Pinus caribaea* var. *hondurensis*) plantation in southeast Queensland, Australia. This research aimed to investigate how early establishment silvicultural treatments would affect weed biomass, soil C, nitrogen (N) and other nutrient pools; and soil C ($\delta^{13}C$) and N isotope composition ($\delta^{15}N$) to help explain the key soil processes regulating the soil C and nutrient pools and dynamics.
Materials and methods Soils were sampled in June 2006 in both the planting row and in the inter-planting row at three depths (0–5, 5–10, and 10–20 cm). Soil parameters including total and labile C and N pools; soil $\delta^{13}C$ and $\delta^{15}N$; total phosphorus (P); extractable potassium (K); moisture content and weed biomass were investigated.
Results and discussion The luxury weed control treatments significantly reduced weed biomass and its organic residues returned to the soil in the first 7 years of plantation development. This resulted in significant variations at some depths and positions in soil $\delta^{13}C$, $\delta^{15}N$, extractable K, hot water extractable organic C (HWEOC), hot water extractable total N (HWETN), potentially mineralizable N (PMN), and soil moisture content (MC). Luxury weed control in the absence of luxury fertilization also significantly decreased extractable K. There was a significant interaction between soil depth and sampling position for soil total C, total N, HWEOC, and HWETN. Weed biomass correlated positively with soil total N, $\delta^{13}C$, PMN, MC, HWEOC, and HWETN.
Conclusions Luxury weed control treatments significantly reduced weed biomass leading to a reduction of soil organic matter. Soil $\delta^{13}C$ and δ^{15}, together with the other soil labile C and N pools, were sensitive and useful indicators of soil C dynamics and N cycling processes in the exotic pine plantation of subtropical Australia.

Keywords C and N pools · Exotic pine plantation · Silviculture · Soil organic matter

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1 Introduction

The early establishment period of forest plantation development provides an opportunity for plantation managers to maximize growth by controlling competition and maximizing access to nutrient and water resources (Neary et al. 1990). Weed control and fertilization are important management practices that allow plantation managers to achieve production outcomes (Mead 2005; Wagner et al. 2006). The success and extent of practices applied at early establishment depend largely on the management objectives and the controlling economic factors (Keeves 1966; Wagner et al. 2006). Despite the reasons for the plantation management decisions, sustaining and investigating the soils productive capacity

has become a priority which is now formally endorsed around the world by the plantation certification, forestry standards, and global climate change research (Weil and Magdoff 2004; Xu and Chen 2006). The effects of weed control and fertilization to maximize growth and encourage the efficient use of nutrients could therefore be summarized not only by their contribution to the plantation productivity but also by how they affect soil processes during the early-age, establishment phase (Smethurst and Nambiar 1989; Woods et al. 1992), as has been done in the past with other forest management practices (Vitousek and Matson 1984; Xu et al. 2008, 2009). Contemporary research on this subject shows a complexity of results due to the nature of soils (Jobbagy and Jackson 2001). It is well accepted globally that land-use change, such as the conversion of abandoned pastures to forest plantations, or establishment of a second rotation plantation can have significant influences on soil C and nutrient dynamics (Chen et al. 2004; Echeverria et al. 2004). Plantation establishment, whether native or exotic pine plantations, has been shown to influence soil C status when established on abandoned or improved pastures (Guo and Gifford 2002; Paul et al. 2002; Chen et al. 2004). Previous land-use and management practices have also been shown to influence N transformations in soil and their associated $\delta^{15}N$ natural abundances (Watson and Mills 1998; Burton et al. 2007; Huang et al. 2008; Pan et al. 2008, 2009) while deforestation and agricultural pursuits can reduce soil organic matter (SOM) recycling and alter SOM chemical composition (Solomon et al. 2002; Mathers et al. 2003; Ussiri and Johnson 2007).

Sustainable management of plantation soils occurs where management practices maintain, increase, or slow SOM decomposition rates (Swift 2001; Weil and Magdoff 2004). For example, maintaining SOM inputs into soils ensures that soils are capable of storing nutrient resources in stable, less mineralizable forms (Swift 2001; Jandl et al. 2007). SOM is responsible in part, for the binding of soil particles, increasing their structure and porosity which leads to an increase in the soils ability to cycle nutrients and hold plant available water (Ghani et al. 2003; Chantigny 2003). One concept of SOM is that it is divided into a number of fractions which includes labile, intermediate, and passive pools. N availability, microbial biomass community structure, gross N mineralization, and C:N ratio can each be influenced by the presence of the light-fraction organic matter which forms a part of the labile C fraction (Cookson et al. 2005). Labile C fractions as measured by hot water extractable organic C (HWEOC) are a useful indicator of soil fertility because they are responsive to short-term management practices (Sparling et al. 1998; Ghani et al. 2003). Labile C has also been identified as indicators of soil productivity, microbial activity, and sustainable land management practices (Franzluebbers et al. 1996; Vance 2000). This research has focused on both total and labile C and (N, along with soil $\delta^{13}C$ and δ^{15} as indicators of soil C dynamics and N cycling processes in an exotic pine plantation of subtropical Australia. Increased SOM (plant, root, and microbial) decomposition and C_4 photosynthetic plant compositions above-ground, can also influence $\delta^{13}C$ in soils (Balesdent et al. 1987; Ehleringer et al. 2000) and so the status of soil organic C and $\delta^{13}C$ can provide an understanding of C cycling resulting from forest management practices (Xu et al. 2008, 2009). Paul et al. (2002) found from comparing a number of studies on soil C and land-use that weed control and fertilization could influence the rate of soil C decomposition when pastures or ex-cropping lands were converted to forest plantations. Simpson et al. (2004) looked at the effects of weed control and residue retention over time and found that residue retention could improve tree growth and weed control could influence soil fertility during second rotation, on coastal, sandy soils of low fertility. They also surmised that luxury weed control was neither financial nor environmentally acceptable as a current management practice.

Despite this, results presented here offer a unique opportunity to understand how soil $\delta^{13}C$ and $\delta^{15}N$ dynamics change with C and N pools as well as other nutrient parameters as a result of effects of weed control and fertilization. To build on previous studies, this study therefore aimed to investigate the manner in which weed control and fertilization practices at early establishment influenced soil C pools, including $\delta^{13}C$, N pools and $\delta^{15}N$ in a 7-year-old exotic pine plantation. This study also aimed to quantify the effects of weed control and fertilization on the other nutrient pools (extractable K and total P). With approximately 64% of the 135,000 hectares of exotic pine plantations in southeast Queensland grown on coastal soils typically low in both N and P and considering the length of time to harvest (up to 25 years), this study aimed to highlight how early establishment weed control and fertilization treatments would influence soil C dynamics and N cycling processes on these sites. The hypotheses that were tested included: (1) weed biomass and weed composition could be influenced by weed and fertilization treatments; (2) soil C and nutrient pools could be influenced by weed control and fertilization treatments; and (3) soil $\delta^{13}C$ and $\delta^{15}N$ could be influenced by changes in soil C and N processes occurring as a result of weed control and fertilization treatments.

2 Materials and methods

2.1 Site description

This experiment was established in 1999 by Forestry Plantations Queensland (FPQ). It was developed as a

complete randomized block design. The experimental plots are located across compartments 207, 208, and 217 in Toolara State Forest, in southeast Queensland, Australia (26°1.556′S, 152°48.81′E). The region has a subtropical climate with an average annual rainfall of 1,222 mm. The mean monthly rainfall and maximum temperatures for Toolara Forest Station are shown in Fig. 1. The local area experiences an average, daily relative humidity between 54% and 76%. Regional soil types are classified as kandosols and hydrosols (Isbell 1996). Localized soil types vary from gray podzolics through to yellow earths but are generally dominated by gray podzolics. Soil particle size analysis indicated that texture was predominantly sandy (78–79%) while pH was relatively acid at 4–4.3 (Table 1). The dimensions of the experimental plots were ten rows × 16 trees, at 5 m × 2.4 m spacing and planted at 888 trees/ha. Gross plots are approximately 0.19 ha. Sixteen plots were selected for this research out of the total experimental area (9.7 ha). The plots represent four treatments: (1) routine fertilizer plus routine weed control (RF+RWC); (2) routine fertilizer plus luxury weed control (RF+LWC); (3) luxury fertilizer plus routine weed control (LF+RWC); and (4) luxury fertilizer plus luxury weed control (LF+LWC) and were replicated four times each.

2.2 Site preparation and planting

All plots were strip-plowed in December 1998. The cuttings were set in October 1998 and planted out when soil moisture was suitable in May 1999. Ten high-growth performance clones (containerized cuttings) of *Pinus elliottii* (var. *elliottii*) × *Pinus caribaea* (var. *hondurensis*) F_1 hybrid were used in each plot.

2.3 Weed control and fertilizer treatments

Routine weed control treatments were applied in accordance with FPQ routine practice in coastal exotic plantations, which stipulates that weed cover should not exceed an average of 20% during the first 9 months in the planting rows. The luxury weed control treatment was applied across the whole plot. Table S1 (see Electronic Supplementary Material) shows a summary of the weed control and fertilizer treatments. Luxury weed control treatment differed from the routine application by frequency of application. The fertilizer treatments were applied as a band application in July 1999. Routine fertilizer was applied as mono-ammonium phosphate (MAP) at the rate of 226 kg ha^{-1} and was reported to provide 10% N and 21.9% P. In addition to the MAP, the luxury fertilizer treatment included a special blend of K and micro-nutrients at a distance of approximately 20 cm from the base of each tree. The luxury fertilizer treatment was intended to encourage maximum growth rates without the growth deformities as a result of excessive N fertilization (Woods et al. 1992).

2.4 Soil sampling and analyses

Soils were sampled in June 2006 in both the planting row (PR) and in the inter-planting row (IPR) to three depths (0–5, 5–10, and 10–20 cm). Soil was collected using a 10 cm diameter soil auger at five random locations within each plot and bulked for each soil depth. This equals one composite soil sample at each depth for each plot, totaling four composite soil samples at each depth for each treatment. Soil samples were refrigerated after sampling and maintained at ~4°C until processing. Field moist samples were used for ammonium (NH_4^+-N), nitrate (NO_3^--N) and potentially mineralizable N (PMN) measurements using the KCl extraction and incubation method of Keeney (1980). Analysis was carried out using a SmartChem SC200 discreet chemistry analyzer. Soil moisture content (MC) was performed by oven drying field moist samples to a constant weight (Rayment and Higginson 1992). The NH_4^+-N and NO_3^--N results were adjusted for water content. HWEOC and HWETN extracts were prepared using the

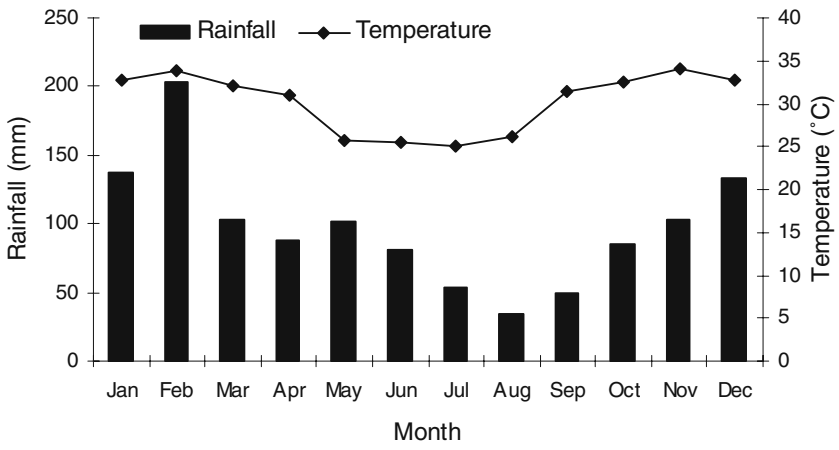

Fig. 1 Mean monthly rainfall and mean maximum temperatures for Toolara Forestry Station

Table 1 Particle size analysis and pH in the top 20 cm soil profile of the experimental site in a 7-year-old plantation of the F_1 hybrid between Slash pine and Caribbean pine in southeast Queensland, Australia

Particle size	Depth (cm)		
	0–5	5–10	10–20
Clay <2 μm (%)	11.0±(2.7)[a]	12.0±(1.9)	12.5±(2.2)
Silt 2-50 μm (%)	9.0±(0.9)	10.0±(0.6)	9.0±(2.2)
Sand 50-2000 μm (%)	79.0±(2.3)	78.0±(1.5)	78.0±(0.0)
pH (0.01 *M* $CaCl_2$)	3.98±(0.05)	4.10±(0.02)	4.30±(0.02)

Values are means (n=4)

[a] Standard errors are indicated in brackets after percentages.

method of Sparling et al. (1998) and Chen and Xu (2005) where 5.0 g (dry weight equivalent) of fresh soil was mixed with 30 mL of distilled water in polypropylene tubes and incubated in a hot water bath at 70°C for 18 h. At the completion of the incubation, the tubes were inverted on an end-over-end shaker for 5 min and then placed in a centrifuge at 2,000 rpm for 20 min. The tubes were centrifuged for another 10 min at 10,000 rpm before filtering through Whatman 42 filter papers into 70 mL containers. Finally, the extract was passed through a 0.45-μm filter membrane before 25 mL of the extract was decantered for analysis using a Shimadzu TOC-$V_{CSH/CSN}$ total organic C and total N analyzer.

All other analysis required soil sub-samples to be ground on a puck and mill grinder including total C and N, $\delta^{13}C$, $\delta^{15}N$, total P, and extractable K. Total C and N, $\delta^{13}C$, and $\delta^{15}N$ were determined on a GVI Isoprime Mass Spectrometer (Manchester, UK) with a Eurovector elemental analyzer (Milan, Italy). Total P was analyzed using a perchloric/nitric acid digestion (Olsen and Sommers 1982). The supernatant was measured by colorimetric determination on a UV-160A Shimadzu, UV Visible Recording, Spectrophotometer at 880 nm. Extractable K was carried out using an acetic acid extraction method and determined using a flame atomic absorption spectrophotometer (ASS; Avanti, GBC Sigma; Knudsen et al. 1982). A series of soil reference samples for total P and extractable K was sent for analysis to two external, independent laboratories to check the accuracy of P and K analysis and used as reference samples. All other analyses were carried out at Griffith University, Nathan, Queensland.

2.5 Understorey biomass sampling

Five 0.25-m^2 quadrates were used to collect understorey biomass from each of the four treatment replicates. The biomass was collected from the inter-row sampling position using the method of Mannetje and Haydock (1963). The samples were stored at 4°C until sub-sampling and then separated into plant types and pine litter (including debris) and dried in an oven at 65°C for 24 h prior to weighing each sample. The mass of the weed biomass and pine litter/debris from the five quadrates per plot were summed and converted from grams per sampling area to tonnes per hectare. Treatment means were then calculated and analyzed. When accounting for total understorey biomass two divisions of the understory components were made. These were (1) pine litter/debris which included leaf litter, bark and branch, cones and duff (horizon above the mineral soil layer) and (2) weed biomass sorted in generalized life forms. These life forms included dried litter (predominantly blady grass litter), herbaceous weeds (*Bidens* spp., etc), native grasses (barbed wire grass), (green) blady grass (*Imperata cylindrica*), pasture grasses (*Paspalum* spp., etc), *Lomandra* spp., native shrubs (*Acacia* spp., *Hakea* spp., *Doodenia* spp.), exotic shrubs (*Baccharis* spp.), pine seedlings (*Pinus* spp.), grass trees (*Xanthorrhoea* spp.), vines (various), and swamp grasses.

2.6 Statistical analysis

Statistical analysis was carried out using combinations of factorial and general ANOVAs and Fisher's least significant difference (LSD) for pair-wise comparisons (treatment and treatment×position). Bonferroni analysis was used where significant means were compared to more than two means (depth, depth×position, and interactions). Analysis was done using GenStat version 11.1 (VSN International Ltd. 2008). Statistical analysis included three factors, sampling positions, sampling depths, and treatments. Sampling positions were divided into planting row (PR) and inter-planting row (IPR), with three sampling depths (0–5, 5–10, and 10–20 cm) and four treatments (RF+RWC, RF+LWC, LF+RWC, and LF+LWC). Correlations were undertaken between the weed biomass and the soil parameters and were assessed for pooled depths (0–5, 5–10, and 10–20 cm) and pooled positions (PR and IPR) at each depth using the Spearman correlation coefficient. Primer 6 (Clarke and Gorley 2005) was used to summarize the patterns between the composition of weed biomass and environmental variables. This included tests such as multidimensional scaling (MDS), Anosim (permutation-based hypothesis testing between groups), and SIMPER (to assess the differences between the weed biomass compositions within each treatment). Principal component analysis (PCA) was used to identify the inter-relationships between soil parameters at the 0–5-cm depth at both positions because it was expected this would be the most dynamic soil depth. Soil parameters were range-standardized and the weed biomass data were log-transformed prior to multivariate analysis.

3 Results

3.1 Weed biomass and composition

Multidimensional scaling of weed compositions by biomass indicated that the four treatments formed two significantly different groups. Group 1 (RF+RWC and LF+RWC) consisted predominantly of a mix of dried and green blady grass (*Imperata* spp.), shrubs and native grass biomass while Group 2 (RF+LWC and LF+LWC) consisted of a mixture of native grass, pine seedlings and herbaceous weeds (Table S2, see Electronic Supplementary Material). Group 1 consisted of RF+RWC and LF+RWC treatments which had approximately 8.38 and 6.45 t ha^{-1} of weed biomass respectively (Table S3, see Electronic Supplementary Material). Group 2 consisted of RF+LWC and LF+LWC treatments which had approximately 0.03 and 0.06 t ha^{-1} of weed biomass, respectively. The Anosim global *R* test revealed that weed compositions were significantly different and that it was the LF+RWC treatment that varied in composition from the RF+LWC and LF+LWC treatments ($p<0.05$). In addition to weed biomass each treatment had a layer of pine needle litter and woody biomass (branches and cones) which attributed approximately ~6 t ha^{-1} of understorey cover. The exception was for the RF+LWC treatment which had ~11 t ha^{-1} although this was shown to be not significantly different from the other treatments (Table S3, see Electronic Supplementary Material).

Principal component analysis (PCA) was used to investigate the inter-relationships within soil parameters. In the planting row at the 0–5 cm depth, PC 1, 2, 3 and 4 explained 82.2% of the total variation in the soil parameters. PC1 at this position and the soil depth contributed to the 38.9% of the total variation and consisted of predominantly soil total C, N and NH_4^+-N. PC2 contributed to the 20.0% of the variation and consisted of NO_3^--N, HWEOC, and HWETN. PC3 contributed 12.8% and consisted of soil NO_3^--N, PMN and total P. PC4 contributed 10.5% and consisted of C:N ratio and total P. At the 0–5-cm depth in the inter-planting row, results indicated that PC1, 2, 3, and 4 contributed to the 84.3% of the total variation. Of this variation, PC1 contributed to the 37.2% of the variation in the soil parameters and consisted of predominantly of total C, HWETN, and extractable K. PC2 contributed to the 22.3% of the variation and consisted of NO_3^--N and soil total P while PC3 contributed to the 16.5% of the total variation in the soil parameters and consisted of $\delta^{15}N$, NO_3^--N, and PMN. PC4 at the 0–5 cm depth in the inter-planting row contributed 8.3% and consisted of C:N ratio and NO_3^--N.

3.2 Effect of sampling depth and position on soil parameters

There was an interaction of sampling depth and position for soil total C, total N, HWEOC, and HWETN, where each of these parameters decreased with soil depth (Table S4, see Electronic Supplementary Material). Soil $\delta^{13}C$, $\delta^{15}N$, C:N ratio, total P, extractable K, NH_4^+-N, NO_3^--N, PMN, and MC were significantly different for soil depth when sampling positions were pooled (Table S5, see Electronic Supplementary Material). Soil $\delta^{13}C$, $\delta^{15}N$, NH_4^+-N, NO_3^--N all increased with soil depth while C:N ratio, total P, extractable K, and PMN decreased with depth. Soil total P varied significantly between the two sampling positions at the 0–5 cm depth in the LF+LWC treatment ($p\leq0.05$) and at the 5–10 cm depth in the RF+RWC treatment ($p<0.05$; Table 2). In both cases, soil total P was higher in the planting row. HWEOC showed a significant effect of sampling position in the RF+RWC ($p<0.05$), LF+RWC ($p\leq0.05$), and LF+LWC ($p<0.05$) treatments at the 0–5 cm depth. HWETN showed a similar response for position and both HWEOC and HWETN were higher in the inter-planting row (see Table 2). Soil moisture content was significantly higher in the inter-planting row at 5–10 cm in the LF+RWC treatment while NH_4^+-N showed a significant effect of sampling position at the 5–10 cm depth in the RF+LWC treatment (see Table 2).

3.3 Effects of treatments on soil parameters

There were significant main effects of the weed control treatments at the 0–5 cm depth in the planting row, on soil total HWEOC and HWETN; total N and $\delta^{13}C$, and in the inter-planting row on HWEOC, HWETN and moisture content, soil $\delta^{13}C$ and $\delta^{15}N$, extractable K, PMN at the 0–5 cm depth ($p<0.05$; see Tables 2 and Table 3). There was a significant interaction between the luxury fertilizer and luxury weed control treatment for soil extractable K in the planting row at the 0–5 cm depth. There were significant main effects of the fertilizer treatments at the 0–5 and 5–10 cm depth on soil $\delta^{13}C$ in the planting row ($p<0.05$; see Table 3).Weed control treatments were significantly different at the 5–10 cm depth in the planting row for moisture content and PMN ($p<0.05$) where both MC (see Table 2) and PMN (see Table 3) were higher in the routine weed control treatments. Weed control treatments were significant at the 5–10-cm depth in the inter-planting row for HWEOC, moisture content, soil $\delta^{13}C$, $\delta^{15}N$, extractable K, and PMN ($p<0.05$). There was a significant interaction of luxury fertilization and luxury weed control for both soil C:N ratio in the inter-planting row at the 5–10 cm depth which reduced the C:N ratio and for extractable K in the planting row at 0–5 cm depth where extractable K was lowest as a result of routine fertilization and luxury weed control (see Table 3).

Table 2 Total phosphorus (P), hot water extractable total organic carbon (HWEOC), hot water extractable total nitrogen (HWETN), nitrate (NO_3^--N), and moisture content (MC) in the 0–10 cm soil profile under different management practices at early establishment of an exotic pine plantation

Treatments		Total P ($mgkg^{-1}$)		HWEOC ($mgkg^{-1}$)		HWETN ($mgkg^{-1}$)		NO_3^--N ($mgkg^{-1}$)		MC (%)	
Fertilizer	Weed control	PR	IPR	PR	IPR	PR	IPR	PR	IPR	PR	IPR
0–5 cm											
RF	RWC	89.95	71.5	313.8a B	459.5a A	10.30a B	16.30a A	6.92	9.86a	1.28	1.32
RF	LWC	68.3	61.1	259.2b	324.3b	9.08b	12.21b	6.22	7.11b	0.64	0.87
LF	RWC	76.3	49.4	347.3a B	501.2a A	13.12a B	18.37a A	7.67	9.65a	1.13	2.42
LF	LWC	76.7A	44.1B	249.5b B	314.6b A	8.04b B	12.32b A	6.47	6.76b	0.98	1.54
5–10 cm											
RF	RWC	71.1A	46.8B	268.1	312.3a	8.04B	11.07A	8.15a	8.57a	2.00	1.56
RF	LWC	66.4	56.6	272.7	236.1b	11.15	8.83	6.47b	6.96b	0.84B	1.52A
LF	RWC	67.8	50.5	317.2	293.3a	11.64	10.92	8.01a B	8.61a A	1.53	2.27
LF	LWC	66.7	41.1	222.5	240.2b	6.41	8.52	7.19b	7.43b	1.24	1.42

Treatments are: routine fertilizer plus routine weed control (RF+RWC); routine fertilizer plus luxury weed control (RF+LWC); luxury fertilizer plus routine weed control (LF+RWC); and luxury fertilizer plus luxury weed control (LF+LWC). Sampling positions are from the planting row (PR) or the inter-planting row (IPR). Values are means (n=4). Where values are followed by different lower-case letters for each soil depth, this indicates that treatment means are significantly different from each other (p<0.05). Where values are followed by different capital letters, this indicates that sampling position means are significantly different (p<0.05)

3.4 Correlations of soil parameters and weed biomass

At pooled sampling positions and soil depths, there were significant positive correlations between soil total C and N (Fig. 2a) and between HWEOC and HWETN (see Fig. 2b; see Table S6, Electronic Supplementary Material). Significant negative correlations existed between soil total C and $\delta^{15}N$ (Fig. 3a), soil total N and $\delta^{15}N$ (see Fig. 3b); between HWEOC and $\delta^{15}N$ (Fig. 4a); and between HWETN and $\delta^{15}N$ (see Fig. 4b). There were also significant correlations between soil total C and extractable K, and between soil total N, and extractable K (Fig. S1a and S1b, see Electronic Supplementary Material) when the sampling positions and depths were pooled (see

Table 3 Total nitrogen (N), carbon (C) and N isotope compositions ($\delta^{13}C$ and $\delta^{15}N$), extractable potassium (K), potentially mineralizable N (PMN) and C:N ratio in the 0-10 cm soil profile under different management practices at early establishment of an exotic pine plantation. Treatments are: routine fertilizer plus routine weed control (RF+RWC); routine fertilizer plus luxury weed control (RF+LWC); luxury fertilizer plus routine weed control (LF+RWC); and luxury fertilizer plus luxury weed control (LF+LWC). Sampling positions are from the planting row (PR) or the inter-planting row (IPR). Values are means (n=4)

		Total N (%)	$\delta^{13}C$ (‰)			$\delta^{15}N$ (‰)		Extractable K ($cmol\ kg^{-1}$)		PMN ($mg\ kg^{-1}$)		C:N ratio	
Fertilizer	Weed control	PR	IPR	PR	IPR	PR	IPR	PR	IPR	PR	IPR	PR	IPR
0–5 cm													
RF	RWC	0.039 a	0.057	−26.9 ba	−26.8 a	2.05	1.60 b	0.063 a	0.068	8.70	10.9 a	37.9	41.6
RF	LWC	0.032 b	0.043	−27.3 bb	−27.2 b	2.22	1.85 a	0.043 b	0.045	5.89	6.58 b	37.7	38.9
LF	RWC	0.044 a	0.056	−25.9 aa	−26.4 a	1.67	0.92 b	0.065 a	0.075	8.94	10.9 a	37.7	38.4
LF	LWC	0.031 b	0.044	−26.9 ab	−27.3 b	2.82	2.04 a	0.063 a	0.066	5.47	6.53 b	41.1	40.7
5–10 cm													
RF	RWC	0.035	0.048	−26.7 b	−26.5 a	2.28	1.79 b	0.056	0.058 a	8.97 a	9.41 a	36.6	41.1 a
RF	LWC	0.039	0.032	−26.8 b	−26.8 b	2.42	3.37 a	0.055	0.040 b	4.21 b	5.67 b	36.5	33.7 b
LF	RWC	0.038	0.038	−26.0 a	−25.9 a	1.72	1.77 b	0.062	0.059 a	9.28 a	10.4 a	36.1	35.4 b
LF	LWC	0.031	0.034	−26.6 a	−26.8 b	2.74	2.18 a	0.050	0.055 b	4.54 b	6.22 b	38.1	35.9 ab

Where values are followed by different lower-case letters for each soil depth, this indicates that treatment means are significantly different from each other (p<0.05)

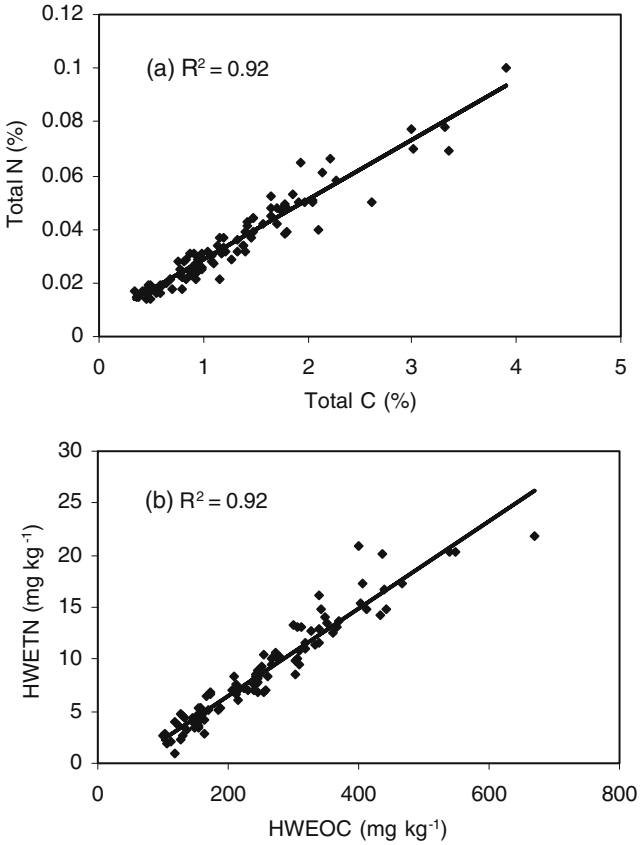

Fig. 2 Relationships between: **a** total carbon (C%) and total nitrogen (N%) (n=92, p=<0.001); and **b** between hot water extractable C (HWEOC) and hot water extractable total N (HWETN; n=92, p< 0.001) at pooled soil sampling depths and positions under different weed control and fertilization treatments

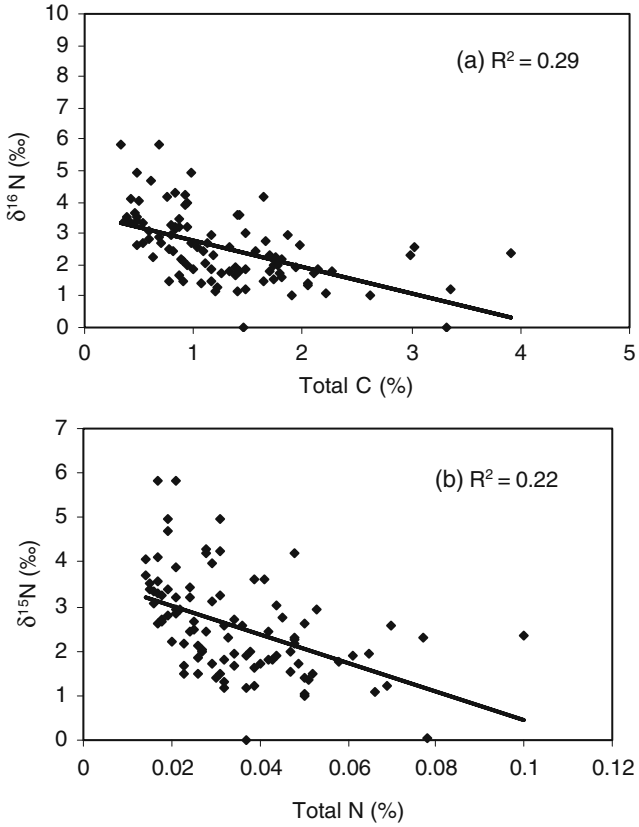

Fig. 3 Relationships between: **a** total carbon (C%) and nitrogen (N%) isotope composition (δ^{15}N‰; n=92, p<0.001); and **b** total N (%) and δ^{15}N (n=92, p<0.001) at pooled soil sampling depths and positions under different weed control and fertilization treatments

Table S6, Electronic Supplementary Material). Correlations also existed between the soil parameters at the 0–5-cm depth when sampling positions were pooled, particularly between soil moisture content and soil total C (/total N/extractable K and NH_4^+-N); PMN, soil total C and total N were each correlated to HWEOC and HWETN (Fig. 5); and soil moisture content was correlated to soil total C, N (Fig. S2, see Electronic Supplementary Material), and extractable K (Table S7, see Electronic Supplementary Material). At the 5–10-cm depth with pooled sampling positions, soil total C, and total N were highly correlated to soil extractable K (/HWEOC/HWETN) while PMN was also correlated with soil moisture content (Table S8, see Electronic Supplementary Material). At the 10–20 cm depth, soil total C and N were correlated to extractable K (HWEOC/ HWETN/ soil total P and soil moisture content) while soil δ^{13}C was correlated to soil total P(/total C/HWEOC and HWETN; Table S9, see Electronic Supplementary Material).

When weed biomass (tonnes per hectare) was correlated to the soil parameters, at pooled soil depths and sampling positions, weed biomass was significantly and positively correlated to soil total N, δ^{13}C, PMN, moisture content, HWEOC, HWETN, and extractable K, but negatively correlated to soil NO_3^--N (see Table S6, Electronic Supplementary Material). At the 0–5-cm depth with the pooled sampling positions, weed biomass was positively correlated to total N, soil δ^{13}C, PMN, moisture content, HWEOC and HWETN but was not correlated to soil NO_3^--N (see Table S7, Electronic Supplementary Material). At the 5–10 cm depth with the pooled sampling positions, weed biomass was positively correlated to PMN, moisture content, HWEOC, and extractable K, but was negatively correlated to NO_3^--N (see Table S8, Electronic Supplementary Material). At the 10–20-cm depth with the pooled sampling positions, only soil moisture content was significantly and positively correlated to the weed biomass (see Table S9, Electronic Supplementary Material).

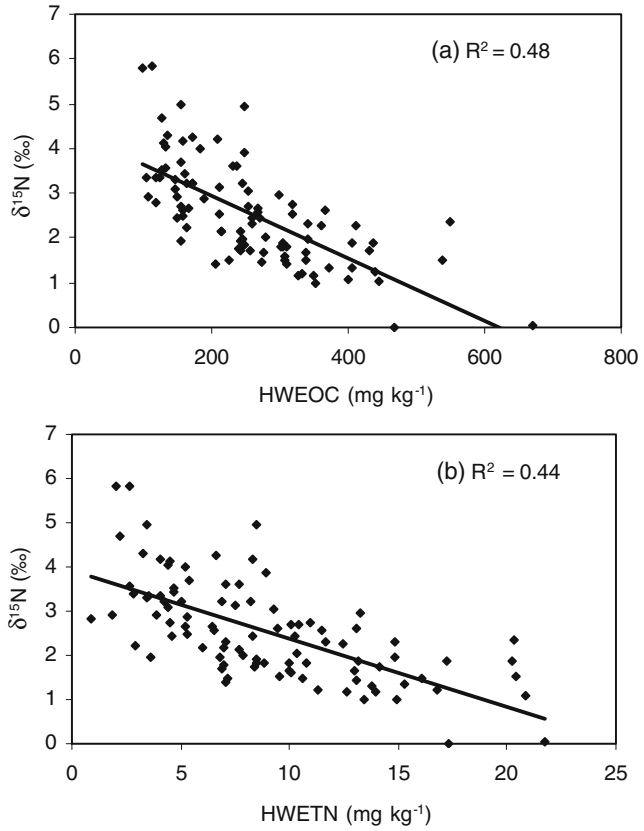

Fig. 4 Relationships between: **a** hot water extractable organic carbon (HWEOC; mg kg^{-1}) and nitrogen (N) isotope composition ($\delta^{15}N‰$) (n=92, p<0.001); and **b** hot water extractable total N (HWETN; mg kg^{-1}) and $\delta^{15}N$ treatments (n=92, p<0.001) at pooled soil sampling depths and positions under different weed control and fertilization

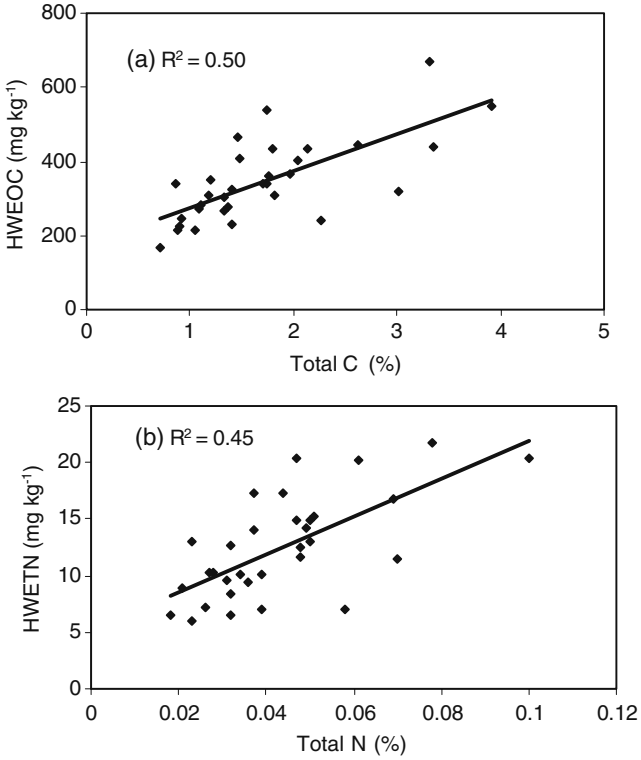

Fig. 5 Relationships between: **a** total carbon (C%) and hot water extractable organic C (HWEOC; mg kg^{-1}; n=32, p<0.001); and **b** total nitrogen (N%) and hot water extractable total N (HWETN; mg kg^{-1}; n=32, p<0.001) at the 0–5 cm soil sampling depth under different weed control and fertilization treatments

4 Discussion

4.1 Treatment effects on weed biomass and composition

Results indicated that weed biomass and weed composition were influenced by weed control and fertilization treatments as hypothesized (Hypothesis 1). The use of routine weed control treatments with luxury fertilization showed the greatest potential for biomass composition of C_4 weeds. The composition of C_4 grasses alone can result in greater SOM residues (Cheng et al. 2008) and increased $\delta^{13}C$ of plant residues recycling into the soil organic matter fractions (Balesdent et al. 1987). Cheng et al. (2008) discuss how C_4 plants have a higher C:N ratio in their biomass compared to C_3 plants due to the increased rubisco levels in the C_4 plants. The variation in C:N ratios of C_4 plants allow them to fix more C per unit weight and therefore produce more biomass for cycling to the soils. Other studies have also found that herbicide applications have the potential to reduce C and N cycling as a result of decreased residues returned to the soil and the reduction of the quality of those residues (Vitousek et al. 1982; Locke and Bryson 1997). In addition to the weed biomass, each treatment also had a layer of pine needle litter and woody biomass which would also be a significant contributor to the nutrient recycling. Although this layer was not significantly different between treatments there was a trend for it to be higher in the routine fertilizer and luxury weed control treatment which changes the predominant source of residues returned to the soils in these treatments. These results suggest that the choice of weed control management has the potential to influence the amount of weed biomass while luxury fertilization has the potential to influence the composition of weeds growing and the subsequent residues returned to the soil over the 7-year period.

4.2 Treatment and sampling effects on soil C pools, $\delta^{13}C$ and other related soil parameters

Results indicated that weed control and fertilization treatments influenced soil C pools (Hypothesis 2) and $\delta^{13}C$ (Hypothesis 3). Routine weed control, when compared to luxury weed control treatments, resulted in a significant increase in weed biomass. This increase was associated with a significant increase in soil $\delta^{13}C$ at the 0–5- and

5–10-cm depths in the inter-planting row and at the 0–5-cm depth in the planting row. A number of reasons are offered to explain why soil $\delta^{13}C$ would differ as a result of weed control treatments and sampling position. Ehleringer et al. (2000) proposed that the most regularly observed trend contributing to the progressive increase of $\delta^{13}C$ in the SOM was due to the increased soil microbial activity. Ehleringer et al. (2002) indicate that soil bacteria and fungi constitute an important component for nutrient cycling, and that they are usually enriched in $\delta^{13}C$ compared to the substrates which they decompose. This leads to the increased soil $\delta^{13}C$, where soil microbes are present. It has been established that soil microbial processes are controlled not only by pH, temperature, and soil moisture but also the quality and quantity of available substrates (Franzluebbers 2004; He et al. 2005, 2006). The presence and degree of this activity is limited primarily by the soil total C and N pools available within the substrates (Mathers et al. 2003) and this has been shown to decrease with soil depth due to the depletion of C compounds (Schlesinger 1977). The results for HWEOC and HWETN also support an increase in microbial activity in soils under these treatments. The hot water method used for labile C extraction removes a component of microbial cells and the method has been shown to correlate with microbial biomass C (Sparling et al. 1998). The HWEOC results show a similar trend to soil $\delta^{13}C$ concentrations in the 0–10 cm soil depths in the inter-planting row and in the 0–5 cm depths for the planting row. The variation at sampling depth and position could be due to the presence or absence of weed roots in the weed control treatments. The increase in HWEOC and $\delta^{13}C$ in the routine weed control treatments seems to indicate an increase of microbial activity as a result of the increased litter, roots, and detritus available for decomposition, although further investigation of microbial activity is warranted to confirm this.

On the other hand, Balesdent et al. (1987) attribute relative proportions of $^{12}C/^{13}C$ in organic matter, to the plant material it is derived from, resulting in the labeling of organic matter $\delta^{13}C$ content dependent on its origins from either C_3 or C_4 vegetation types. The less negative or $\delta^{13}C$ enrichment in soils under the routine weed control treatments could also be related to the organic matter being enriched with $\delta^{13}C$ from C_4 residues growing in these treatments. The results presented here show that the most significant contribution to soil C and N dynamics was as a result of the increased above-ground residues from the routine weed control treatments even though the experiment was only 7 years old at sampling. Wedin et al. (1995) found that $\delta^{13}C$ changes were small but significant after 2 years when four grass species were introduced into an oak savannah. The grass litter in their study reportedly lost 70% of its initial mass over the 2 years. In addition, the $\delta^{13}C$ signatures shifted for both C_4 and C_3 grasses during decomposition by −1.5‰ and +0.6‰ respectively. Wedin et al. (1995) concluded that the shifts in $\delta^{13}C$ were the result of soil organic C mixing with residual C from fungal and microbial activity formed on litters from both C_3 and C_4 sources. Oelbermann and Voroney (2007) found a shift in soil $\delta^{13}C$ from that typically recorded for C_4 vegetation (long-term pasture site) to one representative of C_3 vegetation after 13 years of inter-cropping with predominantly C_3 plants.

Cheng et al. (2008) found that increasing residual inputs from the introduced *Spartina alterniflora* (a C_4 plant) onto Yangtze River wetlands in China after 8 years, had shown a clear shift from the original *Scirpus mariqueter* (a C_3 plant) δ^{13} C values to that typical of a C_4, $\delta^{13}C$ isotope signatures. These examples of how $\delta^{13}C$ is affected by vegetative litter sources can alter the soil organic matter $\delta^{13}C$ values over relatively short time frames, give evidence to support the reasoning that residual inputs from the C_4 grass litters could have decomposed enough in 7 years for the soil organic C to be enriched by the C_4 $\delta^{13}C$. This is also supported by significant positive correlations of total weed biomass to soil $\delta^{13}C$, total N, HWEOC, HWETN, PMN, and soil moisture content, suggesting that as weed biomass (plant residues) increased so did the magnitude of these parameters. Although results showed only small shifts in $\delta^{13}C$ isotope signatures (approximately −0.05‰ to 0.1‰) in the soil under routine weed control treatments, the differences were statistically significant. Unfortunately, the determination of $\delta^{13}C$ values for each plant type was outside the scope of this research, the predominant species (*Pinus* spp. and *Imperata* spp.) photosynthetic groups have been reported in other literature (Chmura and Aharon 1995). There was also a change in $\delta^{13}C$ with soil depth. Changing $\delta^{13}C$ with depth is explained by Cheng et al. (2008) and Jobbagy and Jackson (2000) as an effect of increased root biomass and residues from their decomposition in the upper soil layers. Jobbagy and Jackson (2000) also suggest that SOM accumulation with depth was not only a function of the above-ground vegetation contributing to the residues but also the interaction between soil texture, type of C present, and precipitation.

Soil $\delta^{13}C$ in the planting row was also influenced by the main effect of fertilizer at the 0–5- and 5–10-cm depths. At this position and these depths, the $\delta^{13}C$ was more enriched as a result of luxury fertilization (approximately +0.35). The effect of luxury fertilization on $\delta^{13}C$ was limited to the planting row where it was applied. Schlesinger (1977) and Alvarez (2005) suggest that nitrate fertilization can increase soil C but only when the residues of the increased plant biomass are returned to the soil. Girvan et al. (2004) found that the use of fertilizers had the potential to increase microbial biomass and facilitate shifts in the microbial

communities. If this were so, we would expect a similar response to fertilizer treatments from HWEOC in the planting row, and this was not the case.

4.3 Treatment and sampling effects on soil N pools, $\delta^{15}N$, and other related soil parameters

Results indicated that weed control and fertilization treatments also influenced soil N pools (Hypothesis 2) and $\delta^{15}N$ (Hypothesis 3). Routine weed control was also associated with the significant differences found in soil moisture content, PMN, and HWETN at the 0–5-cm depth in the inter-planting row and total N in the planting row. PMN is influenced by soil moisture and temperature and therefore their increase may lead to greater N mineralization. Routine weed control provided a soil mulching effect which could have decreased evaporation and reduced temperature variation from the soil surface prior to the time of sampling. Routine weed control resulted in more favorable conditions for soil N accumulation and as a result greater nutrient cycling was facilitated. PMN, NH_4^+-N, and HWETN showed similar trends at the 0–5-cm depth (although NH_4^+-N was not significant) with higher concentrations in the inter-planting row in the routine weed control treatments. PMN was also significant at the 5–10-cm depth at both sampling positions between weed control treatments. A number of studies have linked the reductions of some labile soil organic matter fractions to a decline in microbial N supplies (Cookson and Murphy 2004; Cookson et al. 2005). The question remains if this could also be reason for the decrease in HWETN as a result of luxury weed control treatments. When weed biomass (tonnes per hectare) was correlated to the soil parameters at the pooled positions at the 0–5-cm depth, results indicated that the weed biomass showed significant relationships with soil total N, PMN, and HWETN and soil moisture content. This suggests that total and labile N parameters varied as a result of weed biomass. Smethurst and Nambiar (1989) and Woods et al. (1992) recognized the pros and cons of N immobilization by weeds in plantations. Principle components analysis at both sampling positions in the 0–5-cm soil depth also indicated a significant contribution of labile N (NO_3^--N and NH_4^+-N), $\delta^{15}N$, and HWETN to the variation in soil nutrient patterns.

Results indicated a trend that was consistent with higher concentrations of $\delta^{15}N$ and NO_3^--N as a result of luxury weed control treatments. Both these parameters showed significant negative relationships to HWEOC, HWETN and PMN at pooled depths and positions. NO_3^--N showed a similar trend to soil $\delta^{15}N$ in the luxury weed control treatments, but unlike soil $\delta^{15}N$, NO_3^--N was not significantly different between the weed control treatments. This could have been the result of soil spatial variability encountered during sampling and because N transformations are influenced by many factors (Hogberg 1997; Hogberg and Johannisson 1993).

As the luxury weed control treatments produced very low weed biomass, there were very few weeds in these treatments to assimilate N. Large pools of NO_3^--N in soils have the potential to be lost out of the soil profile. This is because NO_3^--N is a mobile compound and if it is not taken up by microbes, plants or roots it can be lost by denitrification, volatilization or leached from the soil (Nadelhoffer and Fry 1994). Higher $\delta^{15}N$ in the luxury weed control treatments coincided with higher nitrate accumulations and demonstrated the potential for their loss. Soils can become enriched in $\delta^{15}N$ as a result of N losses through ammonium volatilization, nitrification, and denitrification. Huygens et al. (2008) found that fractionation could not alone explain large $\delta^{15}N$ variation patterns but concluded that $\delta^{15}N$ enriched microbial compounds were related to high $\delta^{15}N$ in the soils. The increase of $\delta^{15}N$ with depth results from the accumulation of organic materials enriched in $\delta^{15}N$, compared to above-ground inputs which are generally low in $\delta^{15}N$ (rainfall and plant litter). This, along with the variations in above-ground weed biomass, could explain why the variation of $\delta^{15}N$ was limited to the top 10 cm of the inter-planting row. These results highlight the influence of early vegetation management on the N cycling processes in coastal sandy soils after 7 years of plantation establishment.

5 Conclusions

Luxury weed control treatments significantly reduced weed biomass leading to a reduction in soil organic matter accumulation. The reduction of soil organic matter in the top 0–10-cm of soil influenced the availability of various nutrients, soil labile C and N pools, and soil moisture. In the absence of weed biomass, there was a decrease in labile C pools and soil $\delta^{13}C$, with negative correlations among soil $\delta^{15}N$, HWEOC, and HWETN. Routine weed control practices led to a larger pool of weed residues and the subsequent active cycling of C and N pools as indicated by the increased HWEOC, HWETN, PMN and $\delta^{13}C$. This study has implicated the consequences of early-age plantation management techniques to C and N cycling in soils and their on-going effects to long-term soil fertility, in an exotic pine plantation of subtropical Australia. The uses of $\delta^{13}C$ and $\delta^{15}N$ in the association with other labile nutrient indices (HWEOC, HWETN, PMN) have proven to be useful indicators of litter recycling and potentially soil microbial processes; N transformations; and N losses and nutrient cycling pathways as a result of the effects of weed control treatments after 7 years of plantation development.

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