Forest Ecology and Management 355 (2015) 91–100

Contents lists available at ScienceDirect

Forest Ecology and Management

journal homepage: www.elsevier.com/locate/foreco

The effect of fertilization levels and genetic deployment on the isotopic signature, constituents, and chemistry of soil organic carbon in managed loblolly pine (*Pinus taeda* L.) forests ☆

Jason G. Vogel [a,*], Dongmei He [a,b], Eric J. Jokela [c,1], William Hockaday [d], Edward A.G. Schuur [e,2]

[a] Texas A&M University, Department of Ecosystem Science Management, 2138 TAMU, College Station, TX 77843, United States
[b] Nanjing Forestry University, 159 Longpan Road, Xuanwu, Nanjing, Jiangsu, China
[c] University of Florida, School of Forest Resources and Conservation, 353 Newins-Ziegler Hall, Gainesville, FL 32608, United States
[d] Baylor University, Department of Geology, One Bear Place #97354, Waco, TX 76798, United States
[e] Center for Ecosystem Science and Society, Department of Biological Sciences, Northern Arizona University, Box 5640, Flagstaff, AZ 86011, United States

ARTICLE INFO

Article history:
Received 31 January 2015
Received in revised form 7 May 2015
Accepted 15 May 2015
Available online 29 May 2015

Keywords:
Loblolly pine
Soil organic carbon
Radiocarbon
Density fractionation
Nuclear magnetic resonance

ABSTRACT

Soil organic carbon (SOC) mass and its constituents, chemistry, and isotopic signatures ($\Delta^{14}C$, $\delta^{13}C$) were examined for two different loblolly pine (*Pinus taeda* L.) research installations located in north-central Florida. Both studies were designed as split-plots with the whole plots as different levels of fertilization and herbicide application (cultural intensity), and full-sib families of loblolly pine were the splits. The cultural intensities and the families of loblolly pine were different at each site and so each site was analyzed separately. The plantations were aged 9 or 10 years at the time of soil sampling. At both sites, the overall mass of SOC to a depth of 0–30 cm was unresponsive to the level of family growth or cultural intensity and did not show a trend with aboveground biomass. The SOC pool was further separated into live roots and wood; and density fractionation was used to separate the SOC sample into a light fraction (LF) (<1.7 g cm^{-3}) and heavy fraction (HF) with the LF dissected further for charcoal and dead roots. Higher fertilization levels generally depressed fine root (<1 mm) biomass, but whether the effect was significant varied with family and soil horizon. The HF was a relatively small component (<5%) of SOC in these sandy textured soils, but at one of the two sites, the HF was significantly increased with more intensive silviculture and for the faster growing family. The $\Delta^{14}C$ value of the LF-SOC for one slow growing family under low culture (136 ± 11‰) differed from the faster growing low culture plot, and its relationship to the atmospheric $\Delta^{14}C$ record suggested that the LF-SOC likely originated prior to stand establishment. The LF chemistry was determined with solid-state ^{13}C nuclear magnetic resonance (NMR) and cultural intensity did not significantly affect SOC chemistry. However, the family effect was significant for carbohydrates at one site, and for lignin and lipids at the other site. Overall, these results suggest that tree genetics in managed forests can influence SOC chemistry and that the relatively small fractions of SOC can change with management intensity; however, the effect of cultural intensity is minimal for the largest components of SOC and there is no clear relationship between SOC dynamics and aboveground production under the management regimes, and stand ages, examined with these two research installations.

1. Introduction

The forests of the southeastern United States are important to the region's C cycling, offsetting nearly 13% of annual anthropogenic CO_2 emissions through C sequestration (Han et al., 2007). The region's forests are changing; however, with a growing proportion of the land base that was once unmanaged woodland now being intensively managed pine forests (Wear and Greis, 2002). Intensive pine forest management involves silvicultural practices such as fertilization, the control of competing vegetation,

☆ This article is part of a special issue entitled "Carbon, water and nutrient cycling in managed forests".

* Corresponding author. Tel.: +1 (979) 845 5580.
E-mail addresses: jason_vogel@neo.tamu.edu (J.G. Vogel), ybfqxy@126.com (D. He), ejokela@ufl.edu (E.J. Jokela), William_Hockaday@baylor.edu (W. Hockaday), ed.schuur@nau.edu (E.A.G. Schuur).
[1] Tel.: +1 (352) 846 0890.
[2] Tel.: +1 928 523 3559.

http://dx.doi.org/10.1016/j.foreco.2015.05.020
0378-1127/

and the planting of genetically-improved seedlings. These practices are used because they increase net primary production, which could affect regional C budgets through sequestration in long-lived wood products and by shortening the time before peak biomass is reached (Gan et al., 2012). In addition, soil organic carbon (SOC) response to these treatments could be beneficial to C sequestration as some studies have observed increased SOC with fertilization (Shan et al., 2001; Vogel et al., 2011). Alternatively, a negative trend in SOC has been observed with intensive weed control (Shan et al., 2001; Echeverría et al., 2004). Tree genetic makeup has also been shown to shift soil structure and C distribution in SOC fractions in as few as 6 years after planting (Sarkhot et al., 2008). Together these studies suggest that management approaches may change the SOC dynamics of managed forests, potentially altering the degree that these forests offset anthropogenic emissions of CO_2.

Forest management approaches are designed to increase forest productivity, as a result, the practices will also mostly increase the rate of C input to SOC. Silvicultural treatments increase aboveground productivity, and with this change the rate of canopy development and self-thinning rates, resulting in increased needle and wood litter inputs to the surface SOC (Jokela and Martin, 2000). Leaching of dissolved organic carbon to surface mineral soils may also increase with organic layer thickness, and belowground inputs to SOC as coarse root fractions would increase with tree size (Jenkins et al., 2003). In contrast to aboveground components of the C cycle, fine root production with fertilization in managed loblolly pine forests can decrease (Maier and Kress, 2000) or increase (King et al., 2002). The elimination of competing understory plants has been shown to decrease root biomass in managed pine stands (Shan et al., 2001). In comparison to silvicultural practices, the effects on belowground processes by the deployment of specific genotypes is less studied for southern pine forests, but Tyree et al. (2014) found that variation in aboveground characteristics for different genotypes of loblolly pine corresponded to variation in both soil CO_2 efflux and heterotrophic respiration.

Losses of C from SOC will be directly affected by how silvicultural practices influence microbial activity as it responds to changes in nutrient availability, the chemistry and abundance of different substrates, and soil structure. For nutrient availability, microbial response to N additions has been extensively studied, but the results have been mixed. Many studies have observed a suppression of the microbial enzymes that are focused on recalcitrant materials (e.g. lignin) (Neff et al., 2002; Keeler et al., 2009), while the decomposition of more easily decomposed materials (e.g. cellulose) has either increased or not changed (Sinsabaugh et al., 2005; Grandy et al., 2008). In grain agricultural systems, SOC has sometimes increased with fertilization (Luo et al., 2010), but the effect is often dependent on the soil depth examined (Khan et al., 2007). Similar to cereal cropping systems, intensively managed southern pine forests are often fertilized with both N and P together (Fox et al., 2007), with K and other macronutrients (Carlson et al., 2014), or in a mixture that includes micronutrients (Vogel and Jokela, 2011); these varied nutrient additions could cause significant deviation from what has been commonly observed for N alone. Nutrient additions may also indirectly affect the chemistry of what substrate is deposited because of changes in relative allocation between roots and aboveground litter (King et al., 2002), which might shift among different genotypes of loblolly pine (Tyree et al., 2014). The chemistry of litter tissues is also directly affected by competition control, which generally removes deciduous species that often have a tissue chemical makeup that results in faster decomposition. In contrast, native SOC from deciduous forests can have lower rates of microbial respiration rates than SOC from coniferous forests (Fissore et al., 2008). Genotypes of loblolly pine will naturally have differences in tissue chemistry, with a commonly planted genotype in managed forests having a variant in the lignin concentration of wood (Ralph et al., 1997). Moreover, resin production is under significant genetic control and increases with the rate of tree growth (Westbrook et al., 2013).

Forest management in the southeastern United States generally involves the varied use of multiple practices (fertilization, competition control, genetic deployment) to increase tree productivity. Because these silvicultural practices are applied in range of ways that make it difficult to relate binary treatment designs (e.g. fertilization vs. no-fertilization) to actual forestry practices, the overall objective of this research was to determine if the level of tree productivity brought about by silvicultural intensity (fertilization levels, competition control) and the genetic deployment of specific families of loblolly pine affected the accumulation of SOC, and the chemical makeup of SOC. Two sites that had contrasts in silvicultural experiments and that incorporated two replicate family blocks × silvicultural intensity treatments were studied in north-central Florida. Previous research in one of the proposed research sites indicated that both Fertilization and Family influenced soil aggregate formation and stabilization in as few as 4 years (Sarkhot et al., 2007a,b, 2008). We hypothesized that increased aboveground productivity would correspond to an increase in soil C amounts and that this change would be reflected in changes in chemistry and a shift in the radiocarbon signatures of SOC towards modern atmospheric values.

2. Methods

The two research sites are part of a series of experiments established by the University of Florida's Forest Biology Research Cooperative. These experiments were designed to test the effects of relative levels of management intensity and genetic deployment on forest growth. One study site was located northeast of Gainesville, FL, in the Austin Carey Experimental Forest (ACF) (29°44′58″N, 82°13′03″W). The other study site was located near Sanderson, FL (30°14′25″N, 82°19′54″W). The areas have a similar average temperature of 20 °C, with Lake City, FL approximately 14 km west of Sanderson having an average precipitation of 1360 mm and Gainesville, FL approximately 8 km from ACF having an average precipitation of 1228 mm (National Climate Data Center, 1971–2000). Both studies were established on poorly drained Spodosols (Pomona and Leon series for the ACF and Sanderson sites, respectively) (USDA Soil Survey Staff). The Pomona series is classified as a sandy, siliceous, hyperthermic Ultic Alaquod, while the Leon series is classified as a sandy, siliceous, thermic Aeric Alaquod. The Pomona series has a Bh (spodic) horizon within 74 cm of the surface and a Btg (argillic) horizon at a depth of 130 cm. The Leon series has a Bh horizon within 38 cm of the surface, but does not contain an argillic horizon.

Both of the studied forests were previously slash pine (*Pinus elliottii* Engelm *var. elliottii*) forests, and after the initial harvest a raised bed was created with a harrow plow. The beds at the high and low intensity Sanderson plots, and the high intensity ACF plots received two passes of the plow, and the low intensity ACF plot received one pass of the plow. The loblolly pine was hand planted, with the Sanderson site planted in January 2000 and the ACF site planted in December 2000, resulting in a 1-year difference in stand age. The high and low intensity treatments were both treated before planting with the herbicides Chopper® (imazapyr) and Garlon® (triclopyr) at manufacturer-prescribed rate listed on the packaging. After the initial planting, divergent levels of fertilization and herbicide control were implemented in order to create variation in management intensity that could be used to assess tree growth response. At both sites, the high intensity management

Table 1
Cumulative element nutrient application rates for high and low culture treatments through 2008 in two managed loblolly pine sites in Florida.

Site	Culture	N	P	K	Mg	Ca	S	B	Zn	Mn	Fe	Cu
				kg ha^{-1}								
ACF	H	450	100	150	38	11	67	1	3	3	8	1
	L	50	60	0	0	0	0	0	0	0	0	0
Sanderson	H	760	180	120	45	45	65	1	3	2	32	4
	L	220	80	0	0	0	0	0	0	0	0	0

plots had undergone repeated vegetation control soon after tree planting and received fertilization in the first year. In addition, spot treatments kept ground cover below a 30% threshold through age 3 on the intensive plots. By age 5, the tree crowns had closed the canopy and the herbaceous and woody understory coverage was relatively sparse at both Sanderson (Roth et al., 2007) and ACF (Vogel, personal observation). The total amounts of elemental fertilizer differed between the high- and low-intensity treatments (Table 1), with application rates varying at each site.

Multiple families of controlled-pollinated loblolly pine (full-sib) were planted at both sites but only a relatively 'fast' and 'slow' growing family was contrasted. We note, however, that all four families were likely superior growers relative to genetic material from natural forests because the families were crosses of 2nd generation genetic selections. The fast growing families at both sites had a common parent "Loblolly pine 756". Originally from South Carolina, this family has been one of the most widely planted loblolly pine families in the southeastern United States. The relatively slow growing families at both sites did not have a common parent. The slow growing family at ACF had its provenance from east Texas, USA while the provenance of the slow growing family at Sanderson was unknown and likely from a mixture of regions. The loblolly pine families were planted as containerized 1-year seedlings, at the Sanderson site at a spacing of 1.22 m × 2.75 for 2990 trees ha^{-1} while at ACF the seedlings were planted at 1.83 m × 3.05 m spacing or 1790 trees ha^{-1}. Plots for each treatment and family were replicated within each of four blocks, resulting in 16 research plots at each site. The trees in each research plot were measured for diameter at 1.37 m and tree height (Drum, 2014), and these measurements were used with generalized allometric relationships for loblolly pine to estimate aboveground biomass (Gonzalez-Benecke et al., 2014). All tissues were assumed to be 50% C.

Three soil cores were collected from the sides of beds within a plot. The sampling location was at the intermediate point between two live trees, with the first of the two trees randomly selected. The next tree in the row needed to be alive for the location to be used, if it was not, then another live tree was randomly selected. The reason for this restricted sampling was that the effects of the genotypes would have been concentrated on the beds for the first few years after planting, and because fertilizers and spot herbicide treatments were directed towards the bed. Although this sampling precludes a full assessment of stand level effects of silviculture and genetics on SOC processes, a fully random sampling of the beds and the inter-bed areas would have likely included a significant amount of variation that obscured treatment effects.

Sampling occurred in January of 2010 at Sanderson, and April of 2010 at ACF. The soil was sampled with a 7.5 cm diameter steel corer that was 1.5 m long. The A horizon was divided into an upper 10 cm to create a consistent sampling unit, and then the remaining A horizon collected. This remainder stopped at the transition from the A and AE to the E. The E horizon was included as a distinct horizon in all cores; however, its C concentration was too low for analysis. The Bh horizon was encountered at varying depths across plots and often the full horizon could not be retrieved with the soil corer because of a shallow water table. At Sanderson, at least every plot had some of the Bh horizon collected and these partial samples were analyzed for proportional contributions of different fractions but not compared statistically. Each horizon was composited at the plot level.

The collected soils were composited by horizon for a plot, passed through a 2 mm sieve, and the material collected on the sieve separated into live root fractions (<1 mm, 1–2 mm, and 2–5 mm) or wood debris that was distinguishable from dead fine roots. Dead fine roots and char generally passed through the sieve and were collected during the density fractionation. The passed through mineral soil was analyzed for texture (hydrometer method) for four plots of each cultural intensity, and pH (1:2 soil to distilled H_2O) was measured for all plots.

Sieved mineral soils were further differentiated using density fractionation (Golchin et al., 1997). Density fractionation has become a common way to separate particulate organic carbon (light fraction) from mineral and organo-mineral particulates (heavy fraction) in soil studies (Young and Spycher, 1979). Various methods have been developed to separate soil into different fractions based on both size and density, using physical and chemical fractionation methods (Trumbore and Zheng, 1996), however, we applied a 2-pool fractionation technique using sodium polytungstate (SPT; H2Na6O40W12; Geoliquids, IL) as the separating heavy liquid solution. SPT is an inert, low-toxicity material commonly used for density separation. Batches of SPT were prepared in 1 L quantities, where 741 g of SPT was added to 856 mL of de-ionized water to provide a density of 1.7 g cm^{-3}. Then 125 mL of this SPT mixture was added to ~35.0 g of field moist soil in 225-mL polycarbonate conical-bottomed centrifuge tubes. The amount of solution was set to 3× the mass of estimated dry soil in order to ensure proper separation between soil fractions (Strickland and Sollins, 1987; Crow et al., 2007). Tubes were shaken standing upright for 1 h in order to suspend the LF (Sollins et al., 2006) and subsequently, vials were left to sit for 1 h to separate gravimetrically. Vials were then centrifuged for 30 min at 2400 rpm to accelerate separation (Crow et al., 2007).

The supernatant from the centrifuged samples was aspirated into a 125 mL Erlenmeyer flask, and the centrifuge tubes rinsed with 1.7 g cm^{-3} SPT in order to include all particles clinging to the sides and lid in the aspirated liquid. Particles that clogged up the aspirator were carefully removed using tweezers and added to the liquid. The aspiration hose was rinsed at the end with the SPT solution in order to ensure inclusion of all particles in the hose. This aspirated liquid (the LF or H) was then decanted into a 3-piece Whatman filter with a pre-weighed and pre-dried Whatman GF/F filter (0.7 μm pore size). The liquid was run through this vacuum-filtration system, and the sides of the filter reservoir rinsed with SPT solution in order to rinse particles down to the level of the filter paper. The filtered SPT solution was then collected and labeled for later recovery using a recovery column (Six et al., 1999). De-ionized water was then used to fill the filter reservoir in order to rinse the sample and was run through the system through the same filter. Once all the filtering was completed, the LF or HF captured on the filter was placed in a petri dish and weighed wet before being placed in the drying oven to dry for at least 48 h.

Charcoal floats at low liquid densities and therefore remains in the light fraction after separation (Golchin et al., 1997). To remove recalcitrant charcoal and the dead root fragment fraction from the LF, the dried samples on the filters were dissected for ~2 h by hand using fine-tipped tweezers and a dissecting microscope set to 10× magnification. Charcoal was likely present in these ecosystems due to frequent burning during pre-colonial up through the mid-twentieth century (Carter and Foster, 2004), though the forests had not burned since the recent planting. For elemental and isotopic analysis, the picked charcoal and roots were aggregated at the site and cultural treatment level. Charcoal and dead root fractions were also dried for at least 48 h at 65 °C.

The sediment that remained in the centrifuge vials subsequent to aspiration was rinsed by adding 150 mL de-ionized water, shaken for 15 min, and centrifuged (at 2400 rpm) for 15 min (Crow et al., 2007). The supernatant was aspirated and placed in a 500 mL beaker. This was performed a total of four times, resulting in a total of 500 mL of supernatant for each vial. This collected supernatant was run through the vacuum-filtration system, using a pre-weighed, pre-dried Whatman filter. This sample was labeled and its final dry weight added to that of the light fraction.

The sediments left over from the final rinse were determined to be the mineral-associated humic substances or HF. In order to include the sediment remaining on the bottom of the vial after scooping, 150 mL of de-ionized water was added to the vial and it was shaken by hand, run through the filtration system, and collected onto pre-dried filter paper. This sample was dried 48 h and its dry weight added to that of the HF. Every 8th sample run was a control and so for the LF separation, pure SPT was used and for the rinsing steps, pure de-ionized water was used. Deionized water was used to rinse the final HF sample.

The LF and HF for the A horizon and the A-Ae horizon, the charcoal fragments, and the dead roots were then ground with a mortar and pestle in preparation for C and N analysis and dry-ashing. The C and N analysis was performed with a NC analyzer (Flash EA 1112, ThermoElectron, Milan, Italy) on the LF, dead roots, and charcoal fragments. The per sample mass of dead roots and charcoal fragments were very low and were composited for adequate sample preparation but the C concentrations were applied to all mass estimates. The C concentration of the HF proved to be too low for the NC analyzer and, therefore, a 10 g sample was dry-ashed at 450 °C for 4 h to estimate organic carbon content. To estimate the amount of C in the HF so that this fraction could be compared to the other constituents, the % organic carbon estimate obtained with the dry-ashing method was assumed to be 50% C (Broadbent, 1953).

Isotopic analyses ($^{13}C/^{12}C$ and $^{14}/^{12}C$ ratios) were performed on the LF samples for the 0–10 cm horizon, and the composite charcoal and dead roots. The samples were first combusted inside previously heat-treated and evacuated quartz tubes that were sealed with the sample and cupric oxide (Vogel, 1992). The combusted sample was then released into a vacuum line and purified to only CO_2 using liquid N_2 and further reduced into graphite via Fe reduction in He (Vogel, 1992). Graphite samples were sent to the UC Irvine W. M. Keck Carbon Cycle Accelerator Mass Spectrometry Laboratory for $\delta^{13}C$ and $\Delta^{14}C$ analysis. The delta notation was used for the stable isotopic signature:

$$\delta^{13}C(‰) = \left(\left[\left(^{13}C/^{12}C\right)_{sample}/\left(^{13}C/^{12}C\right)_{PDB}\right] - 1\right) * 1000$$

where the isotopic ratio of the sample is divided by the isotopic ratio of a common standard (Pee Dee Belemnite (PDB)). Radiocarbon estimates were normalized for varying $^{13}C/^{12}C$ ratios with a standardization of the $\delta^{13}C$ value to 25‰, and were normalized for radioactive decay to a standard, oxalic acid (OX1, $\delta^{13}C = -19‰$) that is radiocarbon age-corrected to the year 1950 (Stuiver and Polach, 1977). In addition, the OX1 standard's measured activity is reduced by multiplying by 0.95, giving the fraction modern ($F^{14}C$) of the sample:

$$F^{14}C = \left(\left[^{14}C/\left(^{12}C + {}^{13}C\right)_{sample}\right]/\left[0.95 * \left(^{14}C/\left(^{12}C + {}^{13}C\right)_{(ox1)}\right)\right]\right)$$

The $F^{14}C$ values were converted to the more commonly used $\Delta^{14}C$:

$$\Delta^{14}C(‰) = \left[F^{14}C * \exp(-(y - 1950)/8267) - 1\right] \times 1000$$

where *y* equals the year of measurement, 1950 is the reference year for the OX1 standard, and 8267 is the mean life, or average number of years before radiocarbon undergoes radioactive decay (Torn et al., 2009). Estimates of analytical error were on average ±1.8‰ for $\Delta^{14}C$.

Nuclear magnetic resonance (NMR) was used to characterize the chemical constituents of the LF components of the 0–10 cm depth soil. The NMR spectra were obtained on a 300 MHz magnet with a Bruker AVANCE III spectrometer located at Baylor University's Paul Marchand laboratory. Between 40 and 120 mg were packed into a 4 mm (outside diameter) NMR rotor with a Kel-F cap, and spun at an MAS frequency of 12 kHz. The soil C functional group distribution was measured by a ramped-amplitude cross polarization pulse sequence with a contact time of 2 ms and a recycle delay time of 2.5 s and approximately 20,000–35,000 acquisitions (scans) were collected for each sample. The data collected were treated with an exponential appodization function and Fourier-transformed with 50–75 Hz line broadening to obtain a spectrum to which we applied manual phase and baseline corrections.

All NMR spectra were integrated according to predominant C functional groups located in the chemical-shift regions specified by Baldock et al. (2004). The peak areas were corrected for spinning sidebands of aromatic, phenolic and amide/carboxyl C using a computational procedure, and the corrected peak areas then fitted using the molecular mixing model of Baldock et al. (2004). The model also uses the elemental *C*/*N* ratios, measured by combustion elemental analysis, as a constraint in calculations of the *N*-containing compounds (protein and charcoal). The model returned estimates of the mass percentage of carbohydrate, protein, lignin, lipid, carbonyl, and charcoal; in addition to the sum of squares of deviation of spectra areas (measured vs. modeled) as means of assessing "goodness of fit". The accepted the model results if the sum of squares for these samples was <4% of the spectral area.

3. Statistics

The study sites were analyzed separately because they were different aged forests with different prior treatment histories (Table 1) and tree densities. All treatment comparisons were conducted using a split plot design with fertilization as the whole plot, and the loblolly pine families as the split. The analyses were performed using SAS statistical software (v 9.3) with the PROC MIXED command. Each soil component's mass and total *C* amounts were examined for normality using the Shapiro–Wilks test. Only the heavy fraction was log-transformed. The NMR estimates for chemical fractions, and percentage *C* values for soil fractions were transformed with a logistic function, as these values were expressed as percentages (Warton and Hui, 2011). Where a significant interaction was observed, treatment means were contrasted and *p*-values adjusted using the Tukey's HSD adjustment. Where averages are shown, the associated standard error from the full model is shown in the table entry. A significance level of $p < 0.1$ was used because of the heterogeneous nature of soil characteristics and the importance of estimating whether a change in a SOC constituent did occur.

4. Results

4.1. Soil characteristics

The transition from the A horizon to E horizon, or an AE horizon, occurred between 29 ± 6 cm (ACF) and 31 ± 4 cm (Sanderson) depth and its thickness was unaffected by treatment. A distinctive E horizon was observed around 30–50 cm depth at both sites. The Bh horizon was encountered between 42 cm and 109 cm at the Sanderson site (average = 63 cm) and this depth was unaffected by treatment. The Bh layer tended to be deeper at the ACF site (68–110 cm) and was often not present or could not be entirely recovered with the soil corer at both sites, and for this reason, it was not included in the statistical comparisons. Textural analysis indicated that the A horizon varied little and was similar within and between both sites, ranging from 91–93% sand, 5–7% silt, and 1–2% clay. Soil pH ranged from 3.8 to 4.1 across both sites for the surface horizons (0–30 cm) with no significant differences among treatments within a site.

4.2. SOC and constituents

The amount of SOC in the surface A horizon (0–10 cm) ranged between 22.0 and 26.5 Mg C ha^{-1}, and in the A to AE horizon (10–29 cm or 10–31 cm), the SOC mass ranged between 19.2 and 31.2 Mg C ha^{-1} across sites and treatments (Table 3). Together these two horizons contained as much or more C (average = 48 Mg C ha^{-1}) as the aboveground biomass of many treatments (Table 2). Despite the relatively large range in C concentration and SOC amounts, there were no significant differences in the C concentration or SOC for either soil layer.

The SOC constituents (root fractions and detrital wood) were a relatively small percentage of total SOC (<10%) but significant contrasts were found for Culture, Family, and Family × Culture for these fractions. The most common significant main effect was for the fine roots (<1 mm), where at ACF the high cultural treatment had a significantly ($p < 0.1$) lower root biomass than the low cultural treatments at both depth intervals (Table 4). For the <1 mm fine root biomass at Sanderson, the culture main effect was significant for the 0–10 cm horizon ($p < 0.05$) and the interaction Family × Culture significant for the 10–31 cm horizon (Table 4). There was also a significant Family × Culture interaction for the 1–2 mm fraction at Sanderson. The interactions suggest that under low culture, the fastest growing family allocated a greater proportion of biomass carbon to fine root production than the other family and treatment combinations. The other significant result for Sanderson was for the wood fragments found in the 0–10 cm layer, where both the Family and Culture main effects were significant ($p < 0.05$) reflecting a relative increase for the fast-growing family and high cultural treatment.

Table 2
Aboveground biomass accumulation for two managed pine sites in Florida having two levels of cultural intensity and a relatively fast and slow growing family of loblolly pine.

Site	Culture	Age	Fast grower Mg C ha^{-1}	Slow Grower
ACF[a]	High	10[b]	37.3[b]	31.9
	Low		24.0	12.9
Sanderson[a]	High	11	58.2	50.4
	Low		36.7	31.7

[a] Drum (2014).
[b] Soil sampling occurred ~1 year prior to the biomass estimate.

Table 3
Soil C concentrations and estimated mass for the A to AE horizons and bedded areas of two managed pine forests (ACF, Sanderson) in Florida with a fast and slow growing family and a high and low culture. There were no significant differences among treatments and the mass values include dead roots and char fragments.

Soil	Carbon (%)	Fast grower		Slow grower	
Depth (cm)	or Mass	High	Low	High	Low
		ACF			
0–10	%C	4.5 (1.1)	4.4	3.2	5.5
	Mg C ha^{-1}	22.0 (4.0)	25.4	23.2	26.5
10–29[a]	%C	1.8 (0.56)	1.8	1.8	2.3
	Mg C ha^{-1}	21.0 (6.4)	19.2	24.8	26.9
		Sanderson			
0–10 cm	%C	2.6 (0.48)	2.5	2.5	3.1
	Mg C ha^{-1}	24.0 (1.9)	23.8	24.6	26.1
10–31 cm	%C	2.9 (0.38)	2.6	2.4	2.9
	Mg C ha^{-1}	29.5 (5.6)	27.7	26.4	31.1

[a] Equivalent to the A horizon to AE transition.

4.3. Density fraction characteristics

The LF contributed the greatest proportion (83–85%) to SOC carbon content relative to the HF and the constituents (dead roots, charred material) within the LF (Table 5). There were no significant Family or Culture effects for the LF mass or its constituents. For the Sanderson site, both the Family and Culture effect was significant ($p < 0.05$) for HF in the A to AE horizon (10–31 cm) (Table 5), a result that followed similar but non-significant trends in the surface A horizon. At the Sanderson site, the charcoal picked from the 0–10 cm LF was the next largest proportional contributor to SOC carbon content at 3% of total SOC-C mass, while at ACF, the HF at 4% of SOC-C mass was the next largest fraction. Deeper within the A horizon, the HF became the next largest contributor to SOC at both sites after the LF (Table 5), with dead roots and charcoal contributing very similar amounts.

The average $\delta^{13}C$ signature of the LF of the A horizon (0–10 cm) ranged from −27.2‰ to −30.2‰ and from 81‰ to 136‰ for $\Delta^{14}C$ (Table 6). There was a general trend for $\delta^{13}C$ to be more negative under low culture at both sites, but the trend was not significant at either site (Table 6). For $\Delta^{14}C$ there were no consistent trends with culture, but at ACF a significant Family × Culture interaction reflected that the slow growing, low culture treatment combination had a more positive value than the other treatment combinations and differed significantly from the fast grower, low culture treatment combination (Table 6). The range in isotopic values for dead roots and charred material, composited at the cultural treatment level for each site, did not overlap for $\delta^{13}C$ but did for $\Delta^{14}C$ (Fig. 1).

At both sites, lignin ranged from 26% to 37% of SOC and was the largest chemical constituent of the A horizon's (0–10 cm) LF, while the second largest class of compounds was carbohydrates (Fig. 2). The main effect of Family was significant at Sanderson for lignin and lipids, and for carbohydrates at ACF (Table 7). The only instance of the Culture main effect being significant was for carbonyl ($p = 0.09$) at the ACF site, and there were no significant Family × Culture interactions.

5. Discussion

The Spodosols of Florida contain SOC amounts that generally exceed 100 Mg C ha^{-1} in the upper 1 m (Stone et al., 1993), and for the upper ~30–35 cm, stored *C* values have been reported that ranged from 39 to 53 Mg C ha^{-1} in mature 26 year-old loblolly pine forests (Vogel et al., 2011) to 29–43 Mg C ha^{-1} in 17 year-old slash pine forests (Shan et al., 2001). These values tended to be similar to those reported here (Table 3), despite our measurements being

Table 4
Mass of live roots by size class (<1 mm, 1–2 mm, 2–5 mm) and detrital wood for two managed loblolly pine forests (ACF, Sanderson) in Florida receiving different cultural (Clt) treatments and families (Fam) of planted pine. The first average values are followed in parentheses by the standard error of the full model.

Soil		Fast grower		Slow grower		*P*-values[a]		
Depth (cm)	Type	High	Low	High	Low	Clt	Fam	Clt × Fam
		Units = g C m^{-2}						
		ACF						
0–10	<1 mm	53 (8)	81	27	78	***0.064***	0.295	0.600
	1–2 mm	33 (7)	31	24	30	0.673	0.315	0.433
	2–5 mm	161 (39)	175	171	112	0.597	0.543	0.402
	Wood	374 (140)	283	135	463	0.550	0.803	0.112
10–29	<1 mm	37 (11)	47	15	108	***0.081***	0.481	0.145
	1–2 mm	36 (18)	50	56	113	0.141	0.934	0.584
	2–5 mm	234 (366)	324	680	173	0.341	0.498	0.185
	Wood	112 (65)	43	41	112	0.993	0.962	0.251
		Sanderson						
0–10	<1 mm	60 (19)	143	54	127	***0.002***	0.544	0.798
	1–2 mm	28 (11)b	43 a	43 ab	29 b	0.913	0.990	***0.023***
	2–5 mm	106 (39)	118	108	29	0.210	0.110	0.100
	Wood	570 (80)	157	297	147	***0.040***	***0.030***	0.261
10–31	<1 mm	12 (6)b	49 a	18 b	27 b	0.100	0.104	***0.013***
	1–2 mm	46 (10)	38	51	49	0.419	0.832	0.745
	2–5 mm	483 (110)	293	803	324	0.236	0.178	0.254
	Wood	112 (63)	95	50	158	0.998	0.658	0.239

Significant ($p < 0.1$) values are in bold and italics.
[a] Degrees of freedom = 1 for all analyses.

Table 5
The light fraction (LF), and constituents (dead roots and charred material), and the heavy fraction (HF) mass of two managed loblolly pine sites (ACF, Sanderson) in Florida receiving different cultural (High vs. Low) treatments and families (Fast vs. Slow grower) of planted pine. The first average values are followed in parentheses by the standard error of the full model.

Soil		Fast grower		Slow grower	
Depth (cm)	Type	High	Low	High	Low
		Units = g C m^{-2}			
		ACF			
0–10	LF	1460 (241)	1480	1140	1590
	Roots[a]	23 (15)	36	47	34
	Char	26 (20)	78	32	59
	HF	52 (12)	40	32	58
10–29	LF	2230 (770)	1180	2270	2190
	Roots	32 (15)	19	44	50
	Char	19 (30)	18	22	50
	HF	119 (78)	115	171	93
		Sanderson			
0–10 cm	LF	1360 (230)	1100	1080	1300
	Roots	69 (11)	25	55	32
	Char	55 (14)	14	26	21
	HF	30 (9)	28	34	15
10–31	LF	1580 (880)	1120	1670	1230
	Roots	13 (17)	14	23	21
	Char	13 (37)	46	40	39
	HF[b]	28 (6)	12	35	28

[a] Dead root fragments.
[b] A significant result ($p < 0.05$) was found for Clt and Fam.

concentrated on the planted beds. Interestingly, the *C* concentrations reported in this study tended to be greater than those observed for previous studies of the upper ~30 cm (Table 3), with most studies reporting values for *C* concentrations that were less than 2.0% (Stone et al., 1993; Ahn et al., 2009; Vogel et al., 2011). One reason for this difference might be explained by the stage in stand development, where these relatively young forests (9 and 10 years of age) were still losing the forest floor and wood fragments incorporated into the surface mineral soils during harvest and site preparation. Both study sites were previously mature slash pine forests, and the harvest of these forests could at stand establishment result in as much ~75 Mg C ha^{-1} being added to the

Table 6
The isotopic signatures ($\delta^{13}C$, $\Delta^{14}C$) of the light fraction of two managed loblolly pine sites (ACF, Sanderson) in Florida receiving high and low cultural (Clt) treatments and slow and fast growing families (Fam) of planted pine. The soil depth examined was 0–10 cm. The first average values are followed in parentheses by the standard error of the full model.

Isotope (‰)	Fast grower		Slow grower		*P*-values[a]		
	H	L	H	L	Clt	Fam	Clt × Fam
	ACF						
$\delta^{13}C$	−26.0 (1.6)	−27.2	−26.5	−27.3	0.44	0.88	0.79
$\Delta^{14}C$	96 (11) ab	81 b	97 ab	136 a	0.19	0.18	***<0.01***
	Sanderson						
$\delta^{13}C$	−27.3 (1.8)	−30.2	−26.9	−29.4	0.39	0.66	0.89
$\Delta^{14}C$	109 (10)	89	99	95	0.26	0.86	0.44

Significant ($p < 0.1$) values are in bold and italics.
[a] Degrees of freedom = 1 for all analyses.

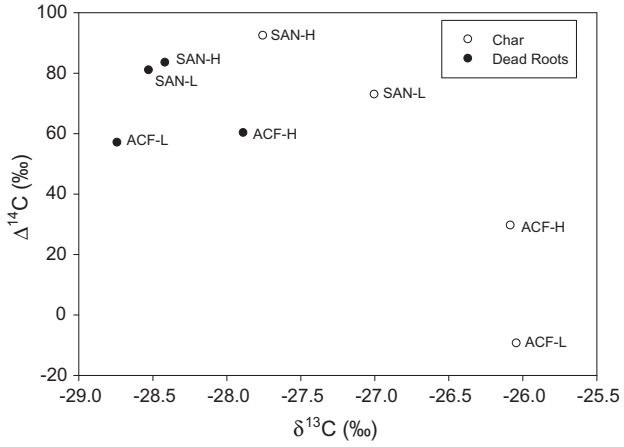

Fig. 1. Isotopic values of dead fine roots and charred material composited at either a high (H) or low (L) cultural treatment level for two different managed forests (ACF, Sanderson (San)) in north central Florida.

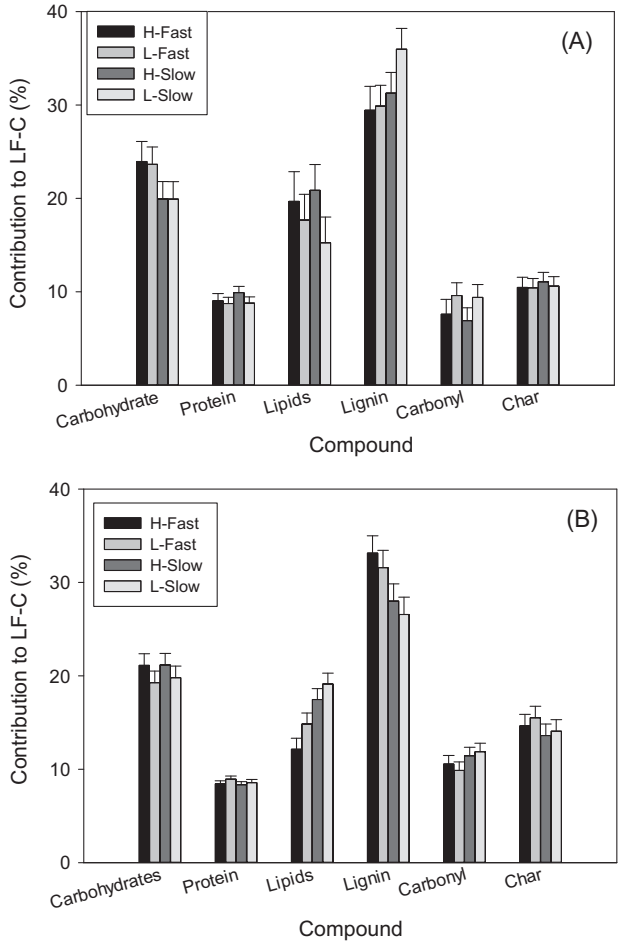

Fig. 2. Percent contribution to the light fraction soil carbon (LF-C) of different organic compound classes at two managed pine sites (A) ACF and (B) Sanderson and under a high (H) and low (L) cultural regime and for a Fast or Slow growing pine family. The 0–10 cm soil depth was examined.

surface soil as forest floor, branch and foliage slash, and coarse root material (Gholz and Fisher, 1982). Incorporated material could help explain the results of Ahn et al. (2009), who reported that young plantations of pine had SOC concentrations in the surface 30 cm that were nearly double those of mature upland unmanaged forests and *C* concentrations that were 30% larger than mature pine plantations. As these forests transition from a state of ongoing *C* loss to more steady-state soil *C* dynamics, it is likely that silvicultural and family effects on SOC will become more easily discerned because the variability caused by detritus incorporation should decrease with stand age.

The planted pine forests of the southeastern United States are managed for increased productivity using a number of different approaches (fertilization, selection of genetically improved planting stock, and competition control), applied at varying levels that reflect the tradeoffs between expense and profit (Fox et al., 2007). In general, forest managers understand that the greater the application of silvicultural practices or the level of genetic control used in seedling choice, the greater the expected gain in growth (Jokela et al., 2004). Thus, the hypothesis that most directly relates to forest management practices is that increased aboveground forest growth and biomass will result in an increase in SOC as a result of increased inputs of *C* to the soil (Vogel et al., 2011). In this study, aboveground biomass for the higher cultural intensities was on average 42% greater than the low cultural treatments across sites and families, while the fast growing families had on average 22% more biomass than the slow growing families across cultural treatments (Table 2). In addition, both litterfall and the forest floor mass were increased by cultural intensity to a greater degree than genetic selection (Drum, 2014). It seemed reasonable then to hypothesize that the largest effect on SOC constituents would be attributed to the cultural intensity. This was the case for fine roots (<1 mm), and here the main effect of increased culture was generally negative and its interaction with family was significant for most soil depths (Table 4). In contrast, a significant positive culture effect was observed for detrital wood at the Sanderson site. However, there was no indication that cultural intensity alone altered the largest pool of SOC—the LF. Rather, culture mainly affected relatively small pools of SOC like roots or the HF, or culture interacted with family at a given site to create a significant difference in the radiocarbon signature of the LF SOC.

Intraspecific variation in tissue chemistry is expected for all non-clonal plant species, and this variation should have consequences for ecosystem processes (Schweitzer et al., 2008a; Bailey et al., 2009). However, it has not been previously observed, as we report here, that the genotypic variation of families of pine alters the chemistry of the LF of SOC. This family effect occurred even though these controlled crosses of loblolly pine still have sibling levels of genetic variation. Notably, the fast grower was the same family between ACF and Sanderson, but its effect on SOC chemistry was not consistent relative to the comparison family (Table 6), which may have reflected the difference in comparison families at the respective sites. If forestry professionals in the future use more seedlings that are clones (Jokela et al., 2004), it is likely that the effect of tree genetics on SOC chemistry will become more easily discerned. For example, Stovall et al. (2013) observed significant clonal variation in root exudation in loblolly pine seedlings and Tyree et al. (2014) observed variation in heterotrophic respiration among different pine clones. In model systems of *Populus* spp., both the decomposition rates of aboveground litter (Madritch et al., 2006) and the surrounding soil microbial communities (Schweitzer et al., 2008a,b) have been shown to be sensitive to

Table 7
Statistical results (F statistic and p-values, df = 1) of NMR compound estimates for the 0–10 cm soil layer of two managed loblolly pine sites (ACF, Sanderson) in Florida receiving different cultural (Clt) treatments and families (Fam) of planted pine.

Compound	ACF						Sanderson					
	Clt		Fam		Clt × Fam		Clt		Fam		Clt × Fam	
	F	P	F	P	F	P	F	P	F	P	F	P
Carbohydrate	0.01	0.92	5.00	***0.05***	0.02	0.88	1.54	0.30	0.00	0.84	0.08	0.79
Protein	0.99	0.34	0.40	0.54	0.31	0.59	1.31	0.28	0.40	0.54	0.18	0.68
Lipid	2.16	0.17	0.18	0.68	0.47	0.51	3.71	0.15	*20.9*	***<0.01***	0.70	0.43
Lignin	1.51	0.27	3.75	0.10	0.91	0.38	0.63	0.49	*10.1*	***0.02***	0.00	0.96
Carbonyl	*3.42*	***0.09***	0.00	0.96	0.02	0.90	0.01	0.95	2.9	0.14	0.30	0.60
Char	0.52	0.52	0.29	0.61	0.01	0.93	0.33	0.58	1.4	0.26	0.03	0.86

Significant ($p < 0.1$) values are in bold and italics.

genotype. In this study, we cannot discern whether the family effects on SOC chemistry were related to differential rates of litter input or its chemistry, or some relationship between the decomposer community and the plant. However we note that in loblolly pine plantations, root net primary production across studies ranges from 0.55 Mg C ha^{-1} yr^{-1} to 5.0 Mg C ha^{-1} yr^{-1} (Lai et al., 2002; Palmroth et al., 2006) while heterotrophic respiration ranges from 4.07 to 10.6 Mg C ha^{-1} yr^{-1} (Lai et al., 2002; Schafer et al., 2003), suggesting that decomposition processes rather than root dynamics alone were the more likely source of variation in SOC chemistry. Given the growing appreciation of how 'priming', or the accelerated decomposition of native SOC by new litter inputs can affect SOC mineralization (Kuzyakov and Domanski, 2000), it is likely that the rhizosphere directly interacted with heterotrophic activity to affect SOC chemistry.

The one significant effect for the $\Delta^{14}C$ of the LF was from the slow growing family under the low cultural treatment at ACF. The $\Delta^{14}C$ of this fraction (136‰) corresponded to an atmospheric value of $\Delta^{14}C$ that was closest to the year 1992 (Hua et al., 2013), approximately 12 years prior to stand establishment. This result, in conjunction with the trend toward this treatment having a greater soil *C* concentration (Table 3) and LF mass (Table 5), but the slowest tree growth (Table 2) than the other treatments at ACF, suggests that the Family × Culture influence was on the decomposition of plant or detrital material that pre-dated site planting. This might have occurred because priming was lower as rates of new root litter inputs were also likely lower in this treatment (Kuzyakov and Domanski, 2000). Alternatively the slow tree growth may have directly affected an abiotic characteristic that affected decomposition, for example the relatively open canopy of the slower growing trees may have created a drier environment for decomposing organisms that inhibited their activity (Borken et al., 2006), or lower rates of transpiration of the slow growing trees may have allowed for longer periods where a perched water table inhibited decomposition. In these sandy well-drained soils that have a relatively shallow water-table (1–2 m), both saturated and excessively dry and warm conditions often occurred within the same year (Drum, 2014), suggesting both factors could have affected decomposition processes differently across treatments.

The LF was the largest contributor to SOC in these forests at all depths; including the recoverable spodic horizons (Fig. 3). For these sandy soils, aggregate structure may be more important to SOC stabilization than the formation of HF on clay-surfaces (Sarkhot et al., 2007a). In soil types with more clay, a HF > 1.7 g cm^{-3} is often a much larger part of the mineral fraction than the LF (Golchin et al., 1997; Sollins et al., 2006) and the HF fraction is older than the LF (Crow et al., 2007). In a relatively un-weathered silt loam from Alaska, Kane et al. (2005) also found that that LF was a greater percentage of SOC than the HF. Despite HF's small contribution to SOC, it is interesting to note that in this study the HF did respond to both family and culture at the Sanderson site. Why this might have occurred is unclear but it reflects an overall trend in this study for smaller components of SOC being more responsive to treatment than the larger LF. If this trend is maintained throughout the forest's development, then SOC constituents like detrital wood or coarse roots could be a significant source of a proportional change in the SOC storage of managed forests.

One potential long-term change in SOC that could occur in managed pine forests is related to the production of charcoal and black carbon pools if the use of fire in private forest management becomes less prevalent in the southeastern United States (Haines et al., 2001). Prior to colonization and widespread conversion of forests to agriculture, forest fires in the Coastal Plain region of the southern United States had return intervals that likely ranged between 1 and 30 years (Komarek, 1974). Although less frequent in the 20th century, prescribed burning continued in the region as a forest management tool that was used to control competing vegetation, for fuels reduction, and in site preparation. Urbanization and concerns about liability by private landowners have put downward pressure on the use of prescribed fires in managed forests (Haines et al., 2001). Although charcoal particles were a relatively small fraction of SOC (Table 5) the NMR results suggested a larger occurrence of char in the LF (Fig. 2), perhaps as much as ~100 g C m^{-2} or an amount greater than the HF or charcoal fragments. Most charcoal composites had a 'bomb' radiocarbon signature but one composite from ACF (−9.4‰) was at least several decades old (Fig. 1), suggesting char may be a source of relatively old C in these systems. It is also likely that char and black carbon detected with NMR are older than distinguishable fragments, and that disintegrated fragments may be carried to deeper soils in these coarse-textured sands, potentially forming the basis of deeper SOC pools (Foereid et al., 2011). We point these patterns out because there are relatively few estimates of charcoal or black carbon from this soil type and fire frequency is a feature of the ecosystem that will likely change in the future in response to changes in forest management practices.

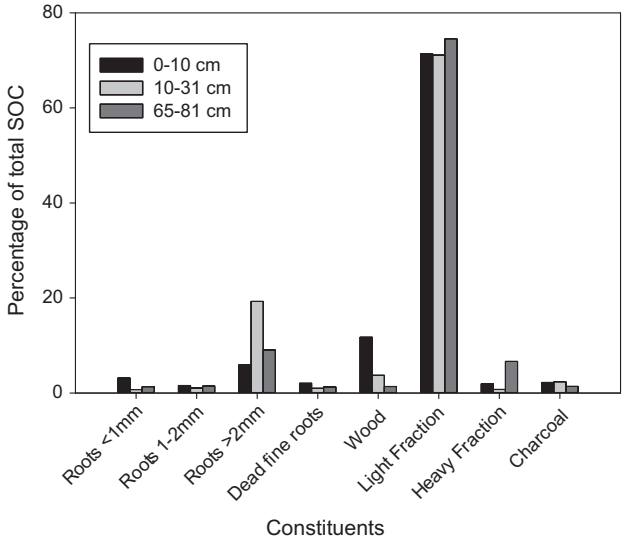

Fig. 3. Proportional contribution among recovered fractions of different components of soil organic carbon averaged across the two sites for the surface A horizon (0–10 cm), the remaining A horizon (10–31 cm), and the spodic horizon (65–81 cm, Sanderson only).

6. Conclusions

We found that even when aboveground biomass accrual is dramatically altered, the effect of silviculture on the total mass of SOC in the A-horizon appears to be modest in Florida Spodosols. This result may generally be due to the coarse texture and lack of reactive minerals in the A horizon, or in this study may reflect that at 9 and 10 years of age, these forests were in a state of transition between the effects of detrital incorporation during site preparation to the sustained effects of a fully developed pine overstory. We hypothesize that the changes that have occurred in SOC chemistry with family selection, and even in small fractions of SOC with cultural intensity, could result in substantial differences in SOC mass among cultural treatments and families at the end of a typical 25 year rotation. Testing this prediction will likely require a study design that differentiates between how decomposition processes are affected directly by tree genetics and culture, or indirectly as the result of a pine family's modification of the soil environment.

Acknowledgements

This research was funded under USDA Soil Processes grant (#2009-65107-05810) to Jokela, Vogel and Schuur. The Forest Biology Research Cooperative established these research installations and helped support the personnel involved with this research. Jokela and Vogel acknowledge the support of The Pine Integrated Network: Education, Mitigation, and Adaptation project (PINEMAP) a Coordinated Agricultural Project funded by the USDA National Institute of Food and Agriculture, Award #2011-68002-30185 during the preparation of this manuscript. We acknowledge the contributions of Lara Franco, Rosvel Bracho, and Ignacie Tumushime in sample collection and processing.

References

Ahn, M.Y., Zimmerman, A.R., Comerford, N.B., Sickman, J.O., Grunwald, S., 2009. Carbon mineralization and labile organic carbon pools in the sandy soils of a north Florida watershed. Ecosystems 12, 672–685.

Bailey, J.K., Schweitzer, J.A., Ubeda, F., Koricheva, J., LeRoy, C.J., Madritch, M.D., Rehill, B.J., Bangert, R.K., Fischer, D.G., Allan, G.J., Whitham, T.G., 2009. From genes to ecosystems: a synthesis of the effects of plant genetic factors across levels of organization. Philos. Trans. R. Soc. B – Biol. Sci. 364, 1607–1616.

Baldock, J.A., Masiello, C.A., Gelinas, Y., Hedges, J.I., 2004. Cycling and composition of organic matter in terrestrial and marine ecosystems. Mar. Chem. 92, 39–64.

Borken, W., Savage, K., Davidson, E.A., Trumbore, S.E., 2006. Effects of experimental drought on soil respiration and radiocarbon efflux from a temperate forest soil. Glob. Change Biol. 12, 177–193.

Broadbent, F.E., 1953. The soil organic fraction. Adv. Agron. 5, 153–183.

Carlson, C.A., Fox, T.R., Allen, H.L., Albaugh, T.J., Rubilar, R.A., Stape, J.L., 2014. Growth responses of loblolly pine in the southeast united states to midrotation applications of nitrogen, phosphorus, potassium, and micronutrients. Forest Sci. 60, 157–169.

Carter, M.C., Foster, C.D., 2004. Prescribed burning and productivity in southern pine forests: a review. For. Ecol. Manage. 191, 93–109.

Crow, S.E., Swanston, C.W., Lajtha, K., Brooks, J.R., Keirstead, H., 2007. Density fractionation of forest soils: methodological questions and interpretation of incubation results and turnover time in an ecosystem context. Biogeochemistry 85, 69–90.

Drum, C., 2014. Effects of silvicultural management intensity and genetics on soil respiration and belowground carbon allocation in loblolly pine plantations. University of Florida, MSc., p. 47.

Echeverría, M.E., Markewitz, D., Morris, L.A., Hendrick, R.L., 2004. Soil organic matter fractions under managed pine plantations of the southeastern USA. Soil. Sci. Soc. Am. J. 68, 950–958.

Fissore, C., Giardina, C.P., Kolka, R.K., Trettin, C.C., King, G.M., Jurgensen, M.F., Barton, C.D., Mcdowell, S.D., 2008. Temperature and vegetation effects on soil organic carbon quality along a forested mean annual temperature gradient in North America. Glob. Change Biol. 14, 193–205.

Foereid, B., Lehmann, J., Major, J., 2011. Modeling black carbon degradation and movement in soil. Plant Soil 345, 223–236.

Fox, T.R., Jokela, E.J., Allen, H.L., 2007. The development of pine plantation silviculture in the southern United States. J. Forest. 105, 337–347.

Gan, J.B., Smith, C.T., Langeveld, J.W.A., 2012. Effects of considering greenhouse gas consequences on fertilizer use in loblolly pine plantations. J. Environ. Manage. 113, 383–389.

Gholz, H.L., Fisher, R.F., 1982. Organic-matter production and distribution in slash pine (pinus-elliottii) plantations. Ecology 63, 1827–1839.

Golchin, A., Baldock, J.A., Clarke, P., Higashi, T., Oades, J.M., 1997. The effects of vegetation and burning on the chemical composition of soil organic matter of a volcanic ash soil as shown by C-13 NMR spectroscopy. 2. Density fractions. Geoderma 76, 175–192.

Gonzalez-Benecke, C.A., Gezan, S.A., Albaugh, T.J., Allen, H.L., Burkhart, H.E., Fox, T.R., Jokela, E.J., Maier, C.A., Martin, T.A., Rubilar, R.A., Samuelson, L.J., 2014. Local and general above-stump biomass functions for loblolly pine and slash pine trees. For. Ecol. Manage. 334, 254–276.

Grandy, A., Sinsabaugh, R., Neff, J., Stursova, M., Zak, D., 2008. Nitrogen deposition effects on soil organic matter chemistry are linked to variation in enzymes, ecosystems and size fractions. Biogeochemistry 91, 37–49.

Haines, T.K., Busby, R.L., Cleaves, D.A., 2001. Prescribed burning in the South: trends, purpose, and barriers. Southern J. Appl. Forest. 25, 149–153.

Han, F.X.X., Plodinec, M.J., Su, Y., Monts, D.L., Li, Z.P., 2007. Terrestrial carbon pools in southeast and south-central United States. Climatic Change 84, 191–202.

Hua, Q., Barbetti, M., Rakowski, A.Z., 2013. Atmospheric Radiocarbon for the Period 1950–2010. Radiocarbon 55, 2059–2072.

Jenkins, J.C., Chojnacky, D.C., Heath, L.S., Birdsey, R.A., 2003. National-scale biomass estimators for United States tree species. Forest Sci. 49, 12–35.

Jokela, E.J., Martin, T.A., 2000. Effects of ontogeny and soil nutrient supply on production, allocation, and leaf area efficiency in loblolly and slash pine stands. Can. J. Forest. Res. 30, 1511–1524.

Jokela, E.J., Dougherty, P.M., Martin, T.A., 2004. Production dynamics of intensively managed loblolly pine stands in the southern United States: a synthesis of seven long-term experiments. For. Ecol. Manage. 192, 117–130.

Kane, E.S., Valentine, D.W., Schuur, E.A.G., Dutta, K., 2005. Soil carbon stabilization along climate and stand productivity gradients in black spruce forests of interior Alaska. Can. J. For. Res. 35, 2118–2129.

Keeler, B.L., Hobbie, S.E., Kellogg, L.E., 2009. Effects of long-term nitrogen addition on microbial enzyme activity in eight forested and grassland sites: implications for litter and soil organic matter decomposition. Ecosystems 12, 1–15.

Khan, S.A., Mulvaney, R.L., Ellsworth, T.R., Boast, C.W., 2007. The myth of nitrogen fertilization for soil carbon sequestration. J. Environ. Qual. 36, 1821–1832.

King, J.S., Albaugh, T.J., Allen, H.L., Buford, M., Strain, B.R., Dougherty, P., 2002. Below-ground carbon input to soil is controlled by nutrient availability and fine root dynamics in loblolly pine. New Phytol. 154, 389–398.

Komarek, E.V., 1974. Effects of fire on temperate forests and related ecosystems: southeastern United States. In: Kozlowski, T.T. (Ed.), Fire and Ecosystems. Academic Press, London, UK, pp. 251–272.

Kuzyakov, Y., Domanski, G., 2000. Carbon input by plants into the soil. Review. J. Plant Nutr. Soil Sci. – Z. Fur Pflanzenernahrung Bodenkunde 163, 421–431.

Lai, C.T., Katul, G., Butnor, J., Siqueira, M., Ellsworth, D., Maier, C., Johnsen, K., Mckeand, S., Oren, R., 2002. Modelling the limits on the response of net carbon exchange to fertilization in a south-eastern pine forest. Plant, Cell Environ. 25, 1095–1119.

Luo, Z.K., Wang, E.L., Sun, O.J., 2010. Soil carbon change and its responses to agricultural practices in Australian agro-ecosystems: a review and synthesis. Geoderma 155, 211–223.

Madritch, M., Donaldson, J.R., Lindroth, R.L., 2006. Genetic identity of Populus tremuloides litter influences decomposition and nutrient release in a mixed forest stand. Ecosystems 9, 528–537.

Maier, C.A., Kress, L.W., 2000. Soil CO_2 evolution and root respiration in 11 year-old loblolly pine (Pinus taeda) plantations as affected by moisture and nutrient availability. Can. J. Forest. Res. 30, 347–359.

Neff, J.C., Townsend, A.R., Gleixner, G., Lehman, S.J., Turnbull, J., Bowman, W.D., 2002. Variable effects of nitrogen additions on the stability and turnover of soil carbon. Nature 419, 915–917.

Palmroth, S., Oren, R., McCarthy, H.R., Johnsen, K.H., Finzi, A.C., Butnor, J.R., Ryan, M.G., Schlesinger, W.H., 2006. Aboveground sink strength in forests controls the allocation of carbon below ground and its [CO_2] – induced enhancement. Proc. Natl. Acad. Sci. 103, 19362–19367.

Ralph, J., MacKay, J.J., Hatfield, R.D., OMalley, D.M., Whetten, R.W., Sederoff, R.R., 1997. Abnormal lignin in a loblolly pine mutant. Science 277, 235–239.

Roth, B.E., Jokela, E.J., Martin, T.A., Huber, D.A., White, T.L., 2007. Genotype x environment interactions in selected loblolly and slash pine plantations in the Southeastern United States. Forest. Ecol. Manag. 238, 175–188.

Sarkhot, D.V., Comerford, N.B., Jokela, E.J., Reeves, J.B., 2007a. Effects of forest management intensity on carbon and nitrogen content in different soil size fractions of a North Florida Spodosol. Plant Soil 294, 291–303.

Sarkhot, D.V., Comerford, N.B., Jokela, E.J., Reeves, J.B., Harris, W.G., 2007b. Aggregation and aggregate carbon in a forested southeastern coastal plain spodosol. Soil Sci. Soc. Am. J. 71, 1779–1787.

Sarkhot, D.V., Jokela, E.J., Comerford, N.B., 2008. Surface soil carbon size-density fractions altered by loblolly pine families and forest management intensity for a Spodosol in the southeastern US. Plant Soil 307, 99–111.

Schafer, K.V.R., Oren, R., Ellsworth, D.S., Lai, C.T., Herrick, J.D., Finzi, A.C., Richter, D.D., Katul, G.G., 2003. Exposure to an enriched CO_2 atmosphere alters carbon assimilation and allocation in a pine forest ecosystem. Glob. Change Biol. 9, 1378–1400.

Schweitzer, J.A., Bailey, J.K., Fischer, D.G., Leroy, C.J., Lonsdorf, E.V., Whitham, T.G., Hart, S.C., 2008a. Plant-soil-microorganism interactions: heritable relationship between plant genotype and associated soil microorganisms. Ecology 89, 773–781.

Schweitzer, J.A., Madritch, M.D., Bailey, J.K., LeRoy, C.J., Fischer, D.G., Rehill, B.J., Lindroth, R.L., Hagerman, A.E., Wooley, S.C., Hart, S.C., Whitham, T.G., 2008b. From genes to ecosystems: the genetic basis of condensed tannins and their role in nutrient regulation in a Populus model system. Ecosystems 11, 1005–1020.

Shan, J.P., Morris, L.A., Hendrick, R.L., 2001. The effects of management on soil and plant carbon sequestration in slash pine plantations. J. Appl. Ecol. 38, 932–941.

Sinsabaugh, R.L., Gallo, M.E., Lauber, C., Waldrop, M.P., Zak, D.R., 2005. Extracellular enzyme activities and soil organic matter dynamics for northern hardwood forests receiving simulated nitrogen deposition. Biogeochemistry 75, 201–215.

Six, J., Elliott, E.T., Paustian, K., 1999. Aggregate and soil organic matter dynamics under conventional and no-tillage systems. Soil Sci. Soc. Am. J. 63, 1350–1358.

Sollins, P., Swanston, C., Kleber, M., Filley, T., Kramer, M., Crow, S., Caldwell, B.A., Lajtha, K., Bowden, R., 2006. Organic C and N stabilization in a forest soil: evidence from sequential density fractionation. Soil Biol. Biochem. 38, 3313–3324.

Staff, S.S. Web Soil Survey. Natural Resources Conservation Service.

Stone, E.L., Harris, W.G., Brown, R.B., Kuehl, R.J., 1993. Carbon storage in Florida Spodosols. Soil Sci. Soc. Am. J. 57, 179–182.

Stovall, J.P., Seiler, J.R., Fox, T.R., 2013. Respiratory C fluxes and root exudation differ in two full-sib clones of *Pinus taeda* (L.) under contrasting fertilizer regimes in a greenhouse. Plant Soil 363, 257–271.

Strickland, T.C., Sollins, P., 1987. Improved method for separating light-fraction and heavy-fraction organic material from soil. Soil Sci. Soc. Am. J. 51, 1390–1393.

Stuiver, M., Polach, H.A., 1977. Reporting of C-14 data – discussion. Radiocarbon 19, 355–363.

Torn, M.S., Swanston, C.L., Castanha, C., Trumbore, S., 2009. Storage and turnover of organic matter in soil. In: Senesi, N., Xing, B., Huang, P.M., (Eds.), Biophysico-Chemical Processes Involving Natural Nonliving Organic Matter in Environmental Systems. pp. 216–232.

Trumbore, S.E., Zheng, S.H., 1996. Comparison of fractionation methods for soil organic matter C-14 analysis. Radiocarbon 38, 219–229.

Tyree, M.C., Seiler, J.R., Maier, C.A., 2014. Contrasting genotypes, soil amendments, and their interactive effects on short-term total soil CO_2 efflux in a 3-year-old *Pinus taeda* L. plantation. Soil Biol. Biochem. 69, 93–100.

Vogel, J.S., 1992. A rapid method for preparation of biomedical targets for AMS. Radiocarbon 34, 344–350.

Vogel, J.G., Jokela, E.J., 2011. Micronutrient limitations in two managed southern pine stands planted on Florida spodosols. Soil Sci. Soc. Am. J. 75, 1117–1124.

Vogel, J.G., Suau, L.J., Martin, T.A., Jokela, E.J., 2011. Long-term effects of weed control and fertilization on the carbon and nitrogen pools of a slash and loblolly pine forest in north-central Florida. Can. J. Forest Res.-Rev. Can. Recherche Forest. 41, 552–567.

Warton, D.I., Hui, F.K.C., 2011. The arcsine is asinine: the analysis of proportions in ecology. Ecology 92, 3–10.

Wear, D.N., Greis, J.G., 2002. Southern Forest Resource Assessment - Summary of findings. J. Forest. 100, 6–14.

Westbrook, J.W., Resende, M.F.R., Munoz, P., Walker, A.R., Wegrzyn, J.L., Nelson, C.D., Neale, D.B., Kirst, M., Huber, D.A., Gezan, S.A., Peter, G.F., Davis, J.M., 2013. Association genetics of oleoresin flow in loblolly pine: discovering genes and predicting phenotype for improved resistance to bark beetles and bioenergy potential. New Phytol. 199, 89–100.

Young, J.L., Spycher, G., 1979. Water-dispersible soil organic-mineral particles. 1. Carbon and nitrogen distribution. Soil Sci. Soc. Am. J. 43, 324–328.