Harvesting and Climate Effects on Organic Matter Characteristics in British Columbia Coastal Forests

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ABSTRACT

As part of investigations into the effects of harvesting old-growth forest, we characterized carbon in five organic matter pools in eight forest chronosequences of coastal British Columbia. Each chronosequence comprised stands in four seral stages from regeneration (3–8 yr) to old-growth (>250 yr), with second-growth stands mostly of harvest origin. Stands were located in two biogeoclimatic subzones with contrasting climate (wetter, slightly cooler conditions on the west coast of Vancouver Island than on the east). Carbon concentrations in fine woody debris (FWD), forest floor (LFH), fine roots from LFH, and two water-floatable fractions from 10 to 30 cm mineral soil (MIN-ROOT, 2–8 mm and MIN-FLOAT, <2 mm) showed no significant effects due to climate, seral stage, or site. There were some significant differences in N concentrations, but none related to seral stage. Carbon-13 cross-polarization with magic-angle spinning (CPMAS) nuclear magnetic resonance (NMR) spectra with principal component analysis of relative areas also showed little harvesting effect, but greater variation related to input of coarse woody debris (CWD) vs. roots high in tannin. Overall, there tended to be more spectral features associated with wood and lignin in the west; whereas some MIN-ROOT samples from the drier east side had aromatic intensity attributed to charcoal. The minimal effects of one harvest on organic matter are most likely due to the large legacy effect; however, more intensive management will probably result in less CWD retention, less charcoal input, and less microsite variability in these pools of poorly decomposed organic matter.

T HE Coastal Forest Chronosequence (CFC) project of the Canadian Forest Service was undertaken in response to public concern in British Columbia over the effects of clearcutting and the conversion of coastal old-growth to managed forests (Trofymow and MacKinnon, 1998). It comprises a broad range of studies on ecosystem function and sustainability in chronosequences (i.e., stands of different ages) from regeneration to old-growth in coastal forests on southern Vancouver Island (Trofymow et al., 1997). While most of coastal British Columbia (including northern and western Vancouver Island) has a mild, wet climate, conditions are drier and slightly warmer on the southeastern part of Vancouver Island. Establishment of the CFC sites on both east and west sides of southern Vancouver Island has enabled studies of successional change after harvesting in contrasting biogeoclimatic subzones (Pojar et al., 1991).

The British Columbia coastal forests are notable for high accumulation of organic matter, especially in the forest floor (Fons and Klinka, 1998; Keenan et al., 1993; Wells and Trofymow, 1997). The accumulation of coarse woody debris (CWD) is especially noticeable, and can have a major influence on the composition of organic horizons (de Montigny et al., 1993; Fox et al., 1994; Fyles et al., 1991; Preston, 1999). Increases in management intensity and harvesting frequency are expected to change both the quantity and nature of organic matter pools, with possible consequences for ecosystem carbon balance, sustainability, and biodiversity. A previous ^{13}C CPMAS NMR study of CWD in the CFC sites (Preston et al., 1998) showed no effect of a single harvesting disturbance on its organic composition. This study extends to five additional pools of poorly decomposed organic matter.

MATERIALS AND METHODS

Sites and Sampling

For the CFC study, eight chronosequence sites were established in 1991–1992 in coastal forests on southern Vancouver Island, four on the east and four on the west side (differentiation by subzone designated East, West). Each site is a chronosequence, comprising stands of four age ranges (seral stage) in close proximity. The age ranges for each seral stage are (reference year 1990): regeneration, 3 to 8 yr; immature, 25 to 45 yr; mature, 65 to 85 yr; and old-growth, >200 yr. Other criteria were that each chronosequence be within an area of 5×5 km^2 or smaller, with different age stands on similar slope, elevation (within 200 m), and aspect. All of the chronosequences are located within the Coastal Western Hemlock biogeoclimatic subzone (Pojar et al., 1991).

The East chronosequences are in the dry leeward coastal western hemlock biogeoclimatic subzone (CWHxm) and are dominated by Douglas-fir [*Pseudotsuga menziesii* (Mirb.) Franco] with a small component of western hemlock [*Tsuga heterophylla* (Raf.) Sarg.] and western red cedar (*Thuja plicata* Donn.). Sites on the west coast of Vancouver Island are in the wetter submontane coastal western hemlock subzone (CWHvm) and dominated by western hemlock with some amabilis fir (*Abies amabilis* Dougl.), western red cedar, and Douglas-fir. Site locations are: Victoria Watershed South, Victoria Watershed North, Koksilah, and Nanaimo Lakes in the East, and Renfrew, Red Granite Creek, Nitinat, and Klanawa in the West. Second-growth stands were of harvest origin except for mature stands at Koksilah and Klanawa, which originated from stand-destroying wildfire.

In each of the 32 stands, we established one plot (60×60 m^2), within which were located four subplots for sampling.

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Fine woody debris (FWD, 2 to 10 mm in diameter) was collected from a 1.0-m² area of each subplot, and the forest floor (LFH) was sampled as two slabs of 20×20 cm² per subplot. Mineral soil was sampled from one pit per subplot for bulk density and chemistry at three depths (0–10, 10–30, and 30–50 cm). Maps, and further details of plot establishment and sampling, are reported elsewhere (Trofymow et al., 1997).

Samples were stored at 2°C until they could be processed, usually within 2 wk. The FWD was dried in paper bags at 70°C for 1 wk, weighed, combined to one sample per plot, and ground to <2 mm in a Wiley mill. The LFH slabs were screened through an 8-mm sieve and the live roots >4 mm in diameter (LFH-ROOT) were separated based on their appearance, lack of decay, flexibility, and springiness relative to older decayed roots. After drying for 7 to 10 d at 70°C, LFH and LFH-ROOT samples were weighed, combined to one sample each per plot, and ground to <2 mm.

After air-drying for 2 to 3 wk, mineral soil samples from all depths were sieved into size fractions of >25 mm, 8 to 25 mm, 2 to 8 mm, and <2 mm. The two largest size fractions were dried and weighed, but not analyzed further. For the 2- to 8-mm fraction, water floatation was used to separate the root–organic component (designated MIN-ROOT) from the fine gravel. After drying and weighing, the MIN-ROOT samples from the four subplots were combined and ground to <2 mm prior to chemical analysis. The heavy 2- to 8-mm residue was dried and weighed but not further analyzed. A subsample of the <2-mm fraction from each subplot was finely ground in a Siebtechnik mill for analysis.

For this study we also separated a light fraction floatable in water (designated MIN-FLOAT) from the <2-mm mineral soil. To keep the number of samples to a manageable level, this fractionation was only done on samples from the 10- to 30-cm depth, and from three East and three West sites. Composite samples of the <2-mm fraction (total air-dry weight approximately 500 g) were prepared from the four 10- to 30-cm subplot samples using proportional weights corresponding to those obtained in the field. For the separation, 400 g of the composite sample was stirred with 1 L of deionized water, and the floatables removed with a strainer and suctioned through a Pasteur pipette. The water was decanted and the procedure repeated. The collected MIN-FLOAT fractions were dried at 70°C and ground to <2 mm.

Chemical Analysis and Nuclear Magnetic Resonance Spectroscopy

Total C concentrations were determined by dry combustion using a LECO (St. Joseph, MI) CR-12 analyzer. Total N was determined with a LECO FP-228 analyzer, except that the Kjeldahl method was used for the <2-mm mineral soil samples. Whereas C and N analyses were done on samples from all sites and depths, it was necessary to restrict NMR sample numbers, as previously done for a study of CWD in the chronosequences (Preston et al., 1998). Therefore, MIN-ROOT and MIN-FLOAT fractions were prepared and analyzed for only one depth (10–30 cm). Further, one East and one West site (Nanaimo Lakes and Renfrew) were excluded for all sample types, with the exception that the MIN-FLOAT fractions were inadvertently prepared from the four Renfrew plots, rather than those from Klanawa.

For NMR analysis small subsamples were further ground to <200 μm. Carbon-13 CPMAS solid-state NMR spectra were obtained using a Bruker (Karlsruhe, Germany) CXP-100 spectrometer at 25.20 MHz for ¹³C. The sample was spun at >4.0 kHz in a 7-mm rotor. The ¹H 90° pulse length was 3.6 μs, the recycling time was 1.5 s, and the contact time was 0.5 ms. Some spectra were obtained with dipolar dephasing (DD), for which a delay of around 50 μs is inserted between the cross-polarization and acquisition steps. The DD spectra retain intensity for carbons with no attached hydrogens or those with some molecular-level mobility, even in the solid state, whereas rigid carbons with attached hydrogens are lost from the spectrum.

In retrospect, a contact time of 1 ms would have been a more suitable choice, as for most organic matter samples, it gives maximum intensity overall and better detection for groups with slower cross-polarization such as nonprotonated aromatic, phenolic, and carboxyl C (Preston 1996, 2001; Smernik and Oades, 2000). Certainly, the relative areas were only used for comparison among the samples in this study, and the wide range of aromaticities detected were sufficient to reveal important differences in OM inputs and decomposition pathways, including distinct characteristics of tannins, lignin, and char.

Two samples, MIN-ROOT and MIN-FLOAT from Plot 11 (Victoria Watershed North, regeneration), were reexamined in more detail at 75.47 MHz on a Bruker MSL 300 at a 4700-Hz spinning rate and contact time of 1 ms. In addition to normal CP spectra, the total suppression of sidebands (TOSS) sequence was used to obtain CP and DD spectra without spinning sidebands (SSB). Quantitative spectra were obtained using single-pulse excitation (SPE, also called Bloch decay) with a ¹³C 90° pulse of 3.9 μs and a 90-s relaxation delay. The SPE spectra were corrected for the background signal from the probe and Kel-F rotor cap (Bruker) by subtracting the free induction decay (FID) obtained from an empty rotor. The number of scans was matched by scaling the background FID.

Chemical shifts are reported with respect to tetramethylsilane (TMS) using adamantane as the secondary reference. Spectra were divided into chemical shift regions as follows (with allowance for slight variation for different sample types): 0 to 47 ppm, aliphatic; 47 to 60 ppm, methoxyl and N-alkyl C from protein; 60 to 95 ppm, O-alkyl; 95 to 110 ppm, di-O-alkyl; 110 to 140 ppm, aromatic (no oxygen attached); 140 to 165 ppm, phenolic; 165 to 210 ppm, carboxyl and carbonyl (including acids, amides, esters, aldehydes, and ketones). Olefinic C may also contribute to the 110- to 140-ppm region. The relative area of each region was obtained from the integral curves generated by the Bruker software.

For the 75 MHz spectra, areas for the methoxyl and O-alkyl regions were combined, the carboxyl–carbonyl region was extended to 220 ppm, and the carboxyl and O-alkyl regions were corrected for SSB intensity from the aromatic region. The SSB correction was done under the usual assumption of equal intensity for the upfield and downfield sidebands (Preston, 2001). However, prior to this, to avoid overcorrecting for the SSB, we used the relative areas of the carboxyl and carbonyl regions in the TOSS spectra to sketch in their counterparts in the CP and SPE spectra and then determined SSB vs. genuine peak intensities by cutting and weighing. While this procedure doubtless also carries some uncertainly, the alternative would assign too much of the intensity in the 165- to 220-ppm region to SSB, whereas the TOSS and DD-TOSS spectra reveal a broad, genuine carbonyl peak at 200 ppm.

Data Analysis

Analysis of variance (AOV) of the organic C fractions was performed using the General Linear Models procedure as implemented in the SAS statistical software (SAS Institute, 1985). For each variable, an initial two-way AOV was run to test for the effects of subzone, seral stage, and the two-way interaction. Because of the blocked design of the study (four

Table 1. Mean total C, total N, and C to N ratio in five organic matter fractions in different subzones and seral stages. Standard errors are given in parentheses.†

	East (CWHxm)						West (CWHvm)						Combined					
	REG	IMM	MAT	OG	Zone		REG	IMM	MAT	OG	Zone		REG	IMM	MAT	OG	All	
C, mg g⁻¹																		
FWD	487	485	490	475	484	(4.4)	488	474	494	493	487	(6.6)	487	480	492	484	486	(3.8)
LFH	396	423	420	430	417	(7.3)	423	449	439	442	438	(6.9)	410	436	430	436	428	(5.3)
LFH-ROOT	487	497	511	492	496	(4.9)	482	490	503	495	493	(3.3)	484	493	507	493	494	(2.8)
MIN-ROOT	473	464	477	452	467	(7.7)	433	457	456	456	450	(5.5)	453	460	467	453	459	(4.9)
MIN-FLOAT	374	379	354	334	360	(8.8)	365	359	350	349	356	(6.1)	370	369	352	340	358	(5.3)
N, mg g⁻¹																		
FWD	4.8	5.2	5.9	4.1	5.0	(0.4)	3.9	5.0	4.4	3.0	4.1	(0.3)	4.4	5.1	5.1	3.6	4.6	(0.2)
LFH	7.8	8.9	7.9	9.6	8.6	(0.4)	9.2	10.4	10.4	8.2	9.5	(0.6)	8.5	9.7	9.1	8.9	9.1	(0.4)
LFH-ROOT	4.8	4.2	3.6	3.6	4.1	(0.3)	4.4	5.4	4.7	3.6	4.5	(0.3)	4.6	4.8	4.2	3.6	4.3	(0.2)
MIN-ROOT	2.9	3.1	2.8	3.2	3.0	(0.2)	4.5	6.5	4.4	3.2	4.8	(0.5)	3.7	4.8	3.6	3.2	3.8	(0.3)
MIN-FLOAT	5.7	6.2	5.2	5.1	5.5	(0.3)	7.6	9.1	8.4	6.6	8.1	(0.6)	6.7	7.7	6.8	5.7	6.7	(0.4)
C to N ratio																		
FWD	1040	1013	856	1410	1080	(122)	1248	1019	1149	1653	1269	(78)	1129	1016	1003	1531	1171	(74)
LFH	519	486	562	449	504	(27)	507	460	439	554	490	(34)	513	473	501	501	497	(21)
LFH-ROOT	1038	911	1134	1566	1162	(159)	1204	955	1141	1471	1193	(94)	1121	933	1137	1518	1177	(91)
MIN-ROOT	1782	1619	1891	1555	1712	(123)	1006	872	1047	1433	1090	(174)	1394	1246	1469	1494	1401	(119)
MIN-FLOAT	695	621	698	658	668	(34)	482	429	417	535	459	(26)	588	525	558	609	568	(31)

† CWHxm, dry leeward coastal western hemlock biogeoclimatic subzone; CWHvm, wetter submontane coastal western hemlock subzone; REG, regeneration, 3 to 8 yr; IMM, immature, 25 to 45 yr; MAT, mature, 65 to 85 yr; OG, old-growth, >200 yr; FWD, fine woody debris; LFH, forest floor; LFH-ROOT, forest floor roots, MIN-ROOT, mineral soil fraction, 2 to 8 mm; MIN-FLOAT, mineral soil fraction, <2 mm.

sites within each subzone) a separate one-way AOV was run for the East and West side subzones to test for the effects of site and seral stage. The same set of AOVs were performed on the relative proportion values for each of the NMR spectral regions but gave only small or inconsistent effects of subzone or seral stage.

The NMR data were reanalyzed with principal component analysis (PCA) using PRINCOMP (SAS Institute, 1985) with the NMR proportional areas as variables. Examples of PCA application to NMR data are discussed in a recent review (Preston, 2001); briefly, the new variables, or principal components (PCs) are linear combinations of the original variables, located in directions that explain most of the variation in the data set. The contribution of each variable to a PC is proportional to the square of its loading on the PC, and each sample is described by its position (score) along the PC axes. To distinguish difference between sites, values of the first and second PCs were plotted and examined to see if sites clustered by subzone or seral stage. Highly significant effects were taken to be at $P < 0.005$, significant effects were at $P < 0.05$, and trends at $0.05 < P < 0.01$. The original NMR and chemical data and full AOV results are available from the authors.

RESULTS AND DISCUSSION
Carbon and Nitrogen

Table 1 summarizes data for C, N, and C to N ratios for the five organic matter pools (means of samples from replicate plots). The highest total C concentrations were found for FWD (474–494 mg g⁻¹) and LFH-ROOT (482–511 mg g⁻¹) followed by MIN-ROOT (433–477 mg g⁻¹), LFH (396–449 mg g⁻¹), and MIN-FLOAT (334–379 mg g⁻¹). For total C, AOV analyses (not shown) did not reveal any significant differences ($P < 0.05$) due to subzone, seral stage, or site; however, there was a greater variation in N concentrations. The AOV showed that N in FWD was significantly higher in East than West sites (5.0 and 4.1 mg g⁻¹, respectively, $P = 0.043$), while MIN-FLOAT N was higher in West sites (8.1 mg g⁻¹ West, 5.5 mg g⁻¹ East, $P = 0.002$). Similar

effects were found for the corresponding C to N ratios; in addition, C to N ratio was higher for MIN-ROOT in the West ($P = 0.011$).

The AOV of site and seral stage effects within East and West subzones showed only a site effect for N and C to N ratio in MIN-ROOT ($P = 0.013$ and 0.024, respectively) in the East. For the West, there was a site effect for C to N ratio of LFH ($P = 0.046$), and a highly significant seral stage effect for FWD C to N ratio ($P = 0.002$), which is especially noticeable as a difference in old-growth vs. regeneration, immature, and mature. To summarize, the analytical data did not show any large effects of seral stage, subzone, or site on C concentrations of these organic matter pools. For N, there was greater zonal and site variation, but only one significant difference related to seral stage (C to N ratio in FWD in the West).

Table 2 gives (along with plot numbers) recoveries of C and N for the separation of the MIN-FLOAT fraction from the <2-mm 10- to 30-cm mineral soil. The MIN-FLOAT fraction accounted for 16 to 30% of C in the East, and generally lower proportions in the West (5–24%). The proportion of N recovered in this fraction was lower than that of soil C in all cases. Compared with both LFH-ROOT and MIN-ROOT, the MIN-FLOAT fraction had higher N, and lower C and C to N ratio (Table 1), indicating a greater degree of decomposition. Comparable results were obtained for a similar floatable fraction from a 40-yr Douglas-fir stand located near the Victoria Watershed North plots (Preston et al., 1994). In that study, floatables from the B horizon accounted for somewhat higher proportions of C and N (30.7 and 17.5%, respectively), but had similar C and N concentrations (370 mg C g⁻¹ and 7.2 mg N g⁻¹) as MIN-FLOAT from immature East sites (379 mg C g⁻¹, 6.2 mg N g⁻¹, Table 1).

The lower proportional recovery of C and N in MIN-FLOAT for the West samples mainly reflects the higher

Table 2. Percentage recovery of C and N in water-floatable fraction (MIN-FLOAT) separated from <2 mm mineral soil from 10 to 30 cm.†

				Percent of mineral soil C and N in MIN-FLOAT				
	East (CWHxm)				West (CWHvm)			
	Plot		C	N	Plot		C	N
			— % —				— % —	
VWS	REG	1	16	5	REN	REG	51	19	11
	IMM	2	16	8		IMM	52	9	5
	MAT	5	18	8		MAT	53	11	6
	OG	6	30	15		OG	54	17	11
VWN	REG	11	17	9	RGC	REG	61	5	2
	IMM	12	27	11		IMM	62	11	8
	MAT	13	28	12		MAT	63	7	5
	OG	15	30	16		OG	64	7	3
KOK	REG	21	25	16	NIT	REG	71	17	12
	IMM	22	25	10		IMM	72	24	17
	MAT	23	28	13		MAT	73	10	5
	OG	24	25	15					

† CWHxm, dry leeward coastal western hemlock biogeoclimatic subzone; CWHvm, wetter submontane coastal western hemlock subzone; REG, regeneration, 3 to 8 yr; IMM, immature, 25 to 45 yr; MAT, mature, 65 to 85 yr; OG, old-growth, >200 yr; VWS, Victorial Watershed South; VWN, Victoria Watershed North; KOK, Koksilah, REN, Renfrew; RGC, Red Granite Creek; NIT, Nitinat.

total stocks of C and N in mineral soil in the West sites, as yields on an areal basis (kg m^{-2}, not shown) were similar. The focus of this study is organic matter quality, and evaluations of C, N, and other nutrients on an areal basis in all compartments of the CFC plots are available elsewhere (Blackwell and Trofymow, 1998; Trofymow and Blackwell, 1998; Wells and Trofymow, 1997). Although the organic matter pools investigated so far in the CFC project have similar C and N concentrations, East sites may be more sensitive to harvesting disturbance because of lower C and nutrient stocks in living, detrital, and mineral soil pools.

Carbon-13 Cross-Polarization with Magic-Angle Spinning Nuclear Magnetic Resonance Spectroscopy

The PCA results for the five organic matter pools are presented in Table 3. For most materials only the first two PCs were important (Eigenvalues >1.0) and score plots in Fig. 1a–e are for only the first two PCs. Selected NMR spectra are shown in Fig. 2 (FWD), Fig. 3 (LFH), Fig. 4 (LFH-ROOT), Fig. 5 (MIN-ROOT) and Fig. 6 (MIN-FLOAT), in positions reflecting their disposition on the corresponding score plots. Spectra are not described in detail for each pool, as their general features are similar to published spectra.

Nuclear Magnetic Resonance—Fine Woody Debris

There was little variation in this pool by seral stage or subzone, with 20 of the 23 samples having very similar NMR spectra (represented by Fig. 2a,b) despite an apparently large spread in the plot of PC1 vs. PC2 (Fig. 1a). The typical peaks of cellulose and hemicellulose are seen at 65 ppm (C6), 72 to 75 ppm (C2, C3, C5), 83 and 89 ppm (C4), and 105 ppm (C1). For softwoods with a predominance of guaiacyl lignin, methoxyl is found at 56 ppm, as in these samples, while the phenolic region has a peak at 147 to 148 ppm with a shoulder at 152 to 153 ppm. Softwoods also have weak, fairly sharp signals for acetate at 21 and 173 ppm (Hatcher, 1987; Preston et al., 1990, 1998). However, these samples also show the influence of bark components (McColl and Powers, 1998); suberin produces additional broad signals in the carboxyl and alkyl regions (maximum at 33 ppm), while in the phenolic region, the mixture of condensed tannins and lignin results in a broad phenolic signal with a maximum at 153 to 154 ppm and shoulder

Table 3. Results of principal component analysis (PCA) of the ^{13}C cross-polarization with magic-angle spinning nuclear magnetic resonance (CPMAS NMR) area data for the five organic matter fractions. For each of the top three principal components (PCs), the eigenvalue and the proportional and cumulative variance it explains are shown with the eigenvector of loadings for each shift region.†

				Eigenvector						
				Shift region (ppm)						
	Eigenvalue	Proportional variance	Cumulative variance	0–50	50–60	60–95	95–110	110–142	142–165	165–210
FWD										
PC1	3.54	0.51	0.51	0.260	0.138	−0.496	−0.476	0.492	0.444	−0.039
PC2	1.82	0.26	0.77	−0.106	−0.638	0.028	−0.047	−0.006	0.309	0.696
PC3	1.09	0.16	0.92	−0.821	0.304	0.269	−0.109	0.281	0.262	0.022
LFH										
PC1	3.98	0.57	0.57	−0.443	0.382	−0.369	−0.332	0.447	0.446	−0.114
PC2	1.26	0.18	0.75	0.136	−0.231	−0.477	−0.118	−0.002	0.065	0.826
PC3	0.84	0.12	0.87	−0.337	−0.452	0.014	0.708	0.336	0.261	0.019
LFH-ROOT										
PC1	4.08	0.58	0.58	0.348	−0.357	−0.471	−0.292	0.478	0.465	0.012
PC2	1.39	0.20	0.78	0.333	0.497	−0.102	−0.504	−0.102	−0.198	0.573
PC3	0.89	0.13	0.91	−0.528	−0.354	0.135	0.024	0.076	0.177	0.734
MIN-ROOT										
PC1	3.26	0.47	0.47	−0.349	0.080	0.542	0.457	−0.501	−0.282	0.195
PC2	1.61	0.23	0.70	−0.427	−0.603	−0.058	0.115	0.128	0.496	0.419
PC3	1.00	0.14	0.84	−0.012	0.548	−0.022	−0.416	−0.191	0.246	0.655
MIN-FLOAT										
PC1	2.21	0.32	0.32	−0.367	0.385	−0.251	−0.457	0.624	0.221	0.082
PC2	1.91	0.27	0.59	−0.530	0.375	0.623	0.254	−0.046	−0.042	−0.350
PC3	1.30	0.19	0.77	−0.233	−0.420	−0.063	0.393	0.116	0.772	−0.030

† FWD, fine woody debris; LFH, forest floor; LFH-ROOT, forest floor roots; MIN-ROOT, mineral soil fraction, 2 to 8 mm; MIN-FLOAT, mineral soil fraction, <2 mm.
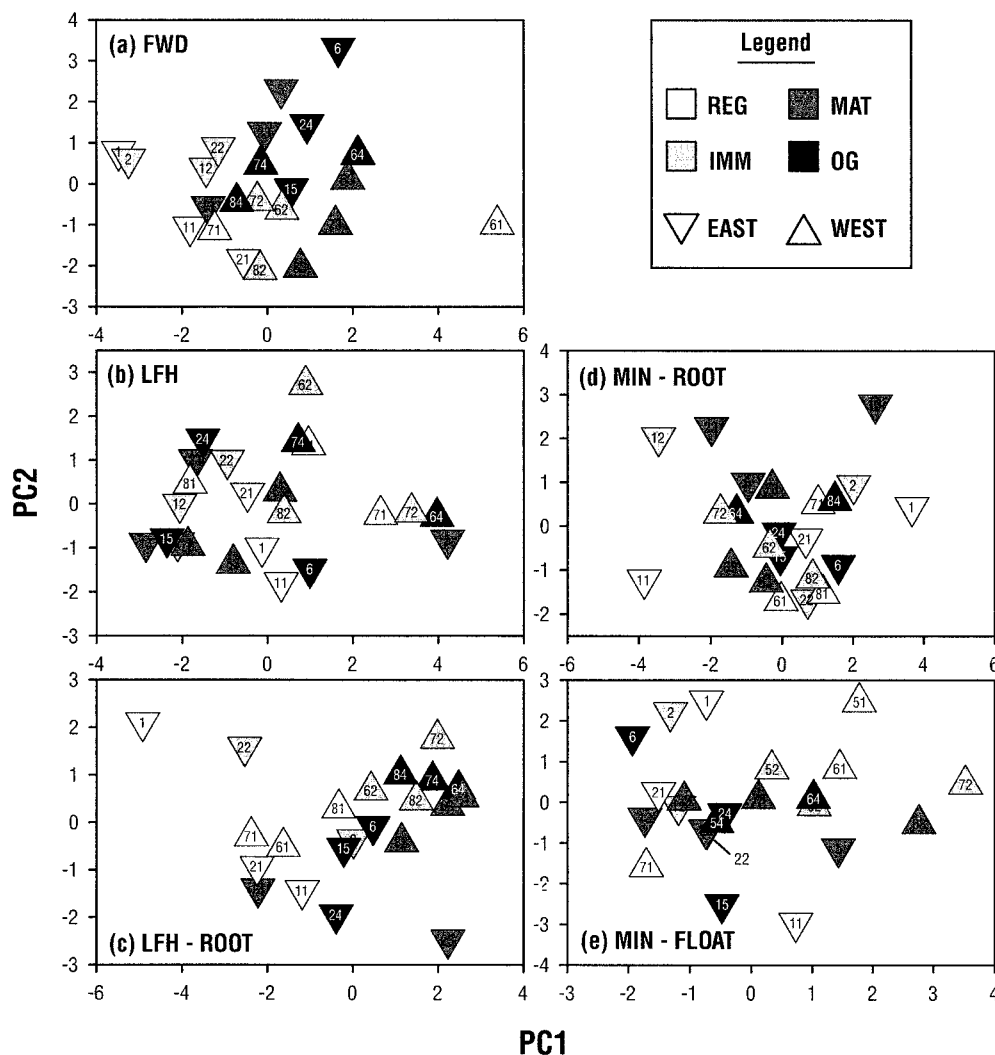
Fig. 1. Principal component analysis (PCA) score plots for the five organic matter pools from the Coastal Forest Chronosequence (CFC) plots: (*a*) fine woody debris (FWD), (*b*) forest floor (LFH), (*c*) forest floor roots (LFH-ROOT), (*d*) mineral soil fraction, 2 to 8 mm (MIN-ROOT), (*e*) mineral soil fraction, <2 mm (MIN-FLOAT).

at 146 to 147 ppm (Lorenz et al., 2000; Preston, 1999). Two samples (Plots 1 and 2) with the lowest scores on PC 1 had higher O- and di-O-alkyl C, and lower alkyl C content, presumably due to a higher ratio of wood to bark (Fig. 2a). These spectra all indicate a composition of fresh or slightly decomposed wood plus bark.

The spectrum and PC scores differed for the sample from Plot 61 (West, Red Granite Creek, regeneration, Fig. 2c), which was similar to coarse woody debris (CWD, >12 cm in diameter) after extensive decomposition by brown-rot fungi (Preston et al., 1990, 1998). Relative loss of polysaccharide and accumulation of lignin result in more prominent signals at 56 ppm (methoxyl), and in the aromatic region, with the phenolic signal typical of guaiacyl lignin (148 ppm, shoulder at 152 ppm) without influence of bark. Our field experience is that woody debris less than about 5 cm in diameter tends to remain white or light in color while losing structural strength (white-rot persists), while for CWD, brown-rot fungi predominate in the later stages of decay, so that Sample 61 may be an anomaly.

For this pool, PC1 accounted for 0.51 of the variance, with largest negative loading on NMR regions 60 to 95 and 95 to 110 ppm (O-alkyl and di-O-alkyl C), and largest positive loading on regions 110 to 142 ppm and 142 to 165 ppm (aromatic and phenolic). Thus, increasing PC1 score is consistent with decreasing carbohydrate and increasing lignin content as indicated in the spectra. Principal Component 2 (0.26 of variance) had negative loading for methoxyl C and positive for phenolic and carboxyl C, but it was difficult to see a PC2 gradient in the spectra. There was some tendency for West samples to score higher on PC1 and lower on PC2, and also for the mature and old-growth sites to have higher scores on PC1. The small variations in the spectra probably arise from a combination of species (tree and understory), age of material upon reaching the forest floor, and residence time. The overall result was that the chemical nature of FWD showed little influence of either subzone or stand age after one harvest. However, our interpretations were limited by the use of composite, plot-level samples. While it is not possible to run large

FWD

(a) East
VWS 1
REG

(b) West
NIT 74
OG

(C) West
RGC 61
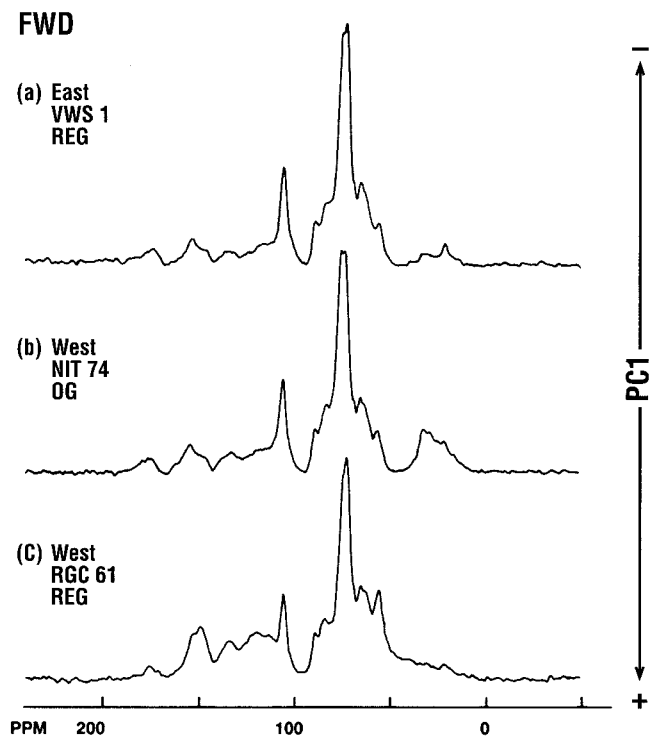**REG**

PC1

PPM   200   100   0

Fig. 2. Carbon-13 cross-polarization with magic-angle spinning nuclear magnetic resonance (CPMAS NMR) spectra for selected fine woody debris (FWD) samples, illustrating variation along Principal Component (PC) 1. REG, regeneration, 3 to 8 yr; OG, old-growth, >200 yr. VWS, Victoria Watershed South; NIT, Nitinat; RGC, Red Granite Creek.

numbers of samples, future work could target samples characterized as to color, mechanical strength, bark cover, species, and origin from live or dead shoots.

Nuclear Magnetic Resonance—Forest Floor

The main features of the LFH spectra (Fig. 3) are similar to those reported for forest floor (Kögel-Knabner et al., 1988, 1992; Zech et al., 1992), particularly in this geographical region (de Montigny et al., 1993; Fox et al., 1994; Prescott and Preston, 1994; Preston et al., 1994; Preston, 1999). The main peaks are found at 30, 56, 73, 105, 130, 148 to 152, and 173 to 178 ppm, and in general are broader than for FWD or LFH-ROOT, probably reflecting both a variety of organic matter inputs, and the effects of decomposition. Principal Component 1 (0.57 of variance) has positive loadings for methoxyl, aromatic, and phenolic C, and negative loadings for alkyl, O-alkyl, and di-O-alkyl C (Table 3). The pattern is similar to that for PC1 of FWD, except for the different sign for alkyl C. Even more than for FWD, spectra with increasing scores on PC1 have increasing similarity to spectra of highly decayed CWD (Preston et al., 1990, 1998). The spectrum of LFH from Plot 5 (East, Victoria Watershed South, mature) with the highest PC1 score is similar to CWD after selective loss of polysaccharide, and accumulation of guaiacyl lignin, but low alkyl and carboxyl intensity. Spectra of samples 6, 63, and 62, with PC1 scores of 0.5 to 1.0, also have a very woody "signature," but with more alkyl and carboxyl C.

By contrast, samples with the most negative PC1 scores (e.g., Plot 13, Fig. 3) are more typical of organic horizons derived from litter and roots. Aromatic intensity is lower, the phenolic signal is split, indicative of the presence of tannins found in foliage and roots, and there is a larger alkyl signal from the accumulation of plant waxes, cutin (foliage), and suberin (roots). While PC1 places the LFH samples along a gradient reflecting nonwoody to woody origin, for PC2 (0.18 of variance), the largest loading is positive for carboxyl C, and this gradient can be seen in the spectra. The increase in carboxyl C along PC2 may be associated with greater oxidation during decomposition. It is less likely to reflect increasing input of plant or microbial lipids, as there is only a small positive loading for alkyl C, consistent with the lack of change of the alkyl intensity along PC2.

There do not appear to be effects associated with seral stage, but differences by subzone can be seen. As found for FWD, more of the West samples fall on the right-hand side of the plot; that is, they have a stronger woody characteristic (Fig. 1b). This is consistent with the higher accumulations of C in CWD on the West side (Trofymow and Blackwell, 1998; Wells and Trofymow, 1997), for which windthrow rather than fire is the most important natural disturbance (Gagnon and Bradfield, 1987; Keenan et al., 1994). On the other hand, most of the East samples have low or negative PC1 scores, indicative of less input or persistence of wood. However, the sample with highest PC1 score is from East Plot 5, while two of the West plots, one regeneration (81) and one mature (73), have high negative PC1 scores. This illustrates the importance of individual site, and even of stand level history. In British Columbia coastal forests, especially in the wetter subzones, large fallen logs can persist for centuries, serving as nurse logs and then forming organic horizons (Daniels et al., 1997; Gagnon and Bradfield, 1987).

Similar LFH characteristics were found for cutover sites on northern Vancouver Island, in a similar biogeoclimatic subzone to the West sites (Preston, 1999). In that study, many spectra of organic horizons (sampled as 0–10 and 10–20 cm) showed a high input or persistence of lignin, while others sampled in close proximity were characteristic of litter and root input. In the CFC sites, the lack of seral stage effect in the LFH and other pools may reflect the limited history of logging in this region, so that disturbed sites still reflect the legacy carbon of many centuries of only natural disturbance. Highly woody signatures are less apparent in forest floor and organic horizons of more highly managed or drier forests (Kögel-Knabner et al., 1988, 1992; Prescott and Preston, 1994; Preston et al., 1994; Zech et al., 1992).

Nuclear Magnetic Resonance—Forest Floor Roots

Spectra of this pool show a striking variation with increasing PC1 and decreasing PC2 scores (Fig. 4). As for FWD and LFH, PC1 (0.58 of variance) has positive loadings on phenolic and aromatic C, and negative loadings on the O- and di-O-alkyl regions associated with
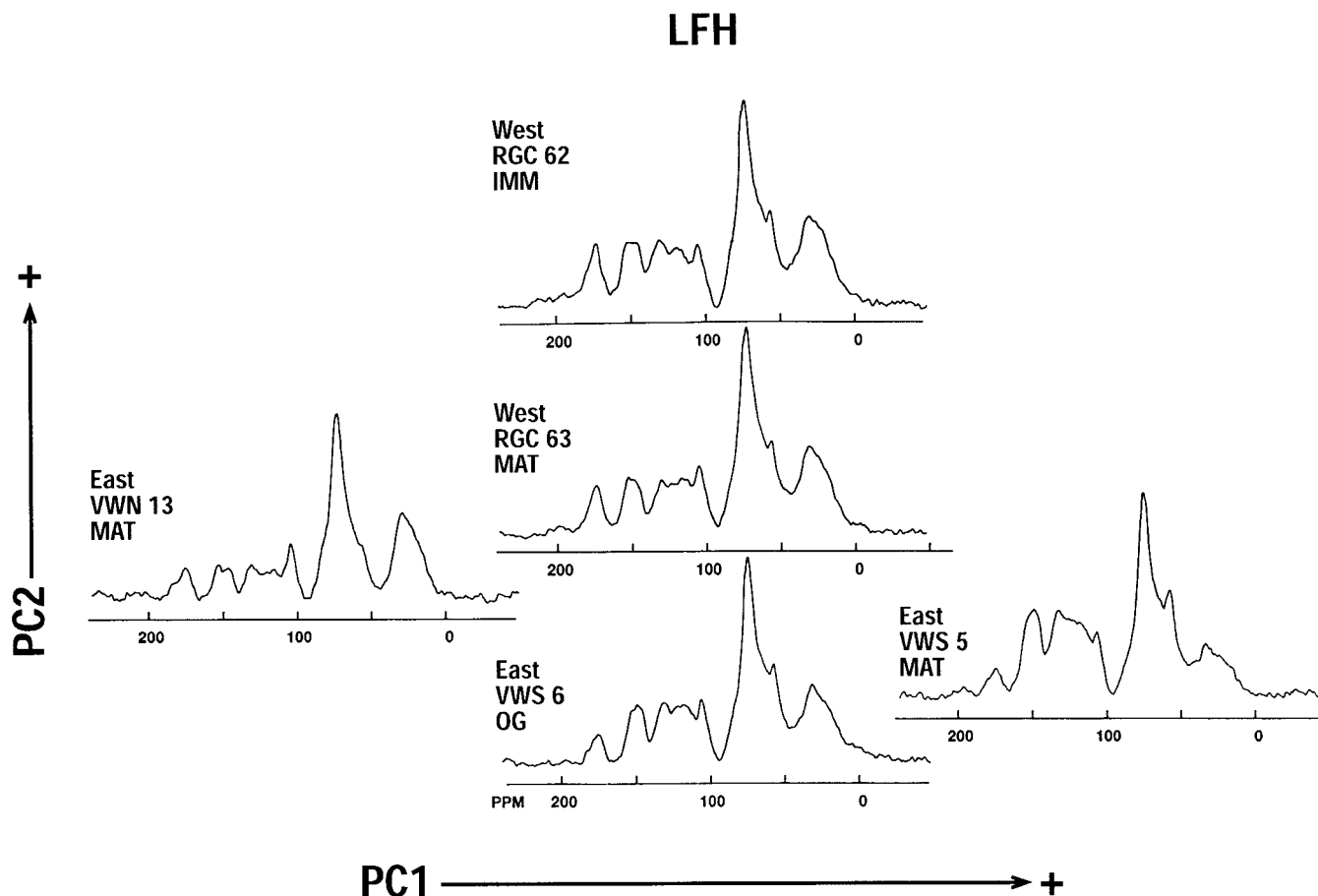
Fig. 3. Carbon-13 cross-polarization with magic-angle spinning nuclear magnetic resonance (CPMAS NMR) spectra for selected forest floor (LFH) samples, illustrating variation along Principal Component (PC) 1 and PC 2. IMM, immature, 25 to 45 yr; MAT, mature, 65 to 85 yr; OG, old-growth, >200 yr. VWS, Victoria Watershed South; VWN, Victoria Watershed North; RGC, Red Granite Creek.

carbohydrate. The loading is negative on methoxyl and positive on alkyl C. However, in contrast to FWD and LFH, the increase in aromatic and phenolic intensity along PC1 is clearly associated with increasing tannin content, as manifested by the typical split phenolic region (145 and 155 ppm), and the "sawtooth" pattern in the aromatic and di-O-alkyl region (131, 117, 105, and shoulder at 96–97 ppm) (Lorenz et al., 2000; Preston, 1999; Preston et al., 2000). The presence of tannin is also supported by the DD spectrum of Sample 11, which has the marker peak for condensed tannins at 105 ppm (Preston and Sayer, 1992; Wilson and Hatcher, 1988). The methoxyl loadings are negative on PC1 and positive on PC2, also consistent with an increase in tannin rather than lignin content. The increase in alkyl intensity along PC1 can be attributed mainly to suberin of roots. Samples from both West and older sites tend to have higher scores on PC1 (Fig. 1c). These subzone and seral stage effects probably arise from differences in species composition, and age of the roots.

Nuclear Magnetic Resonance—Mineral Soil Fraction, 2 to 8 mm

Principal component analysis was particularly useful in interpreting the more complex variations in this frac-

tion. Principal Component 1 (0.47) has positive loadings on O-alkyl and di-O-alkyl C, and negative loadings on alkyl, aromatic, and phenolic C. As shown in Fig. 5a, the two samples with the most negative PC1 scores (Plots 11 and 12) have high intensity for both aromatic and alkyl C (130 and 29 ppm, respectively), while those with the highest positive scores are similar to LFH-ROOT Samples 11 and 22 (Fig. 4), with low lignin and high tannin indicators. As discussed later, the strong aromatic signal in some samples may be derived from charcoal. The expansion in Fig. 5b shows the variety in samples with similar PCA scores. Samples 5, 73, 15, and 64 have signatures consistent with roots and/or charcoal, whereas Samples 72 and 63 have a woodier signature.

In contrast to PC1, there is little obvious variation along PC2 (0.23 of variance), which has negative loadings for alkyl and methoxyl C, and positive loadings for phenolic and carboxyl regions. This may arise from the multiple sources of variation—inputs of charcoal, wood, or roots of different ages and diameters, all further modified by decomposition. Samples with the highest and lowest scores on PC1, as well as the highest PC2 scores, were from the East, while West samples had a lower range on both PC axes (Fig. 1d). The samples with a charcoal fingerprint were all found in East sites, which are more likely to have been disturbed by fire (Trofy-
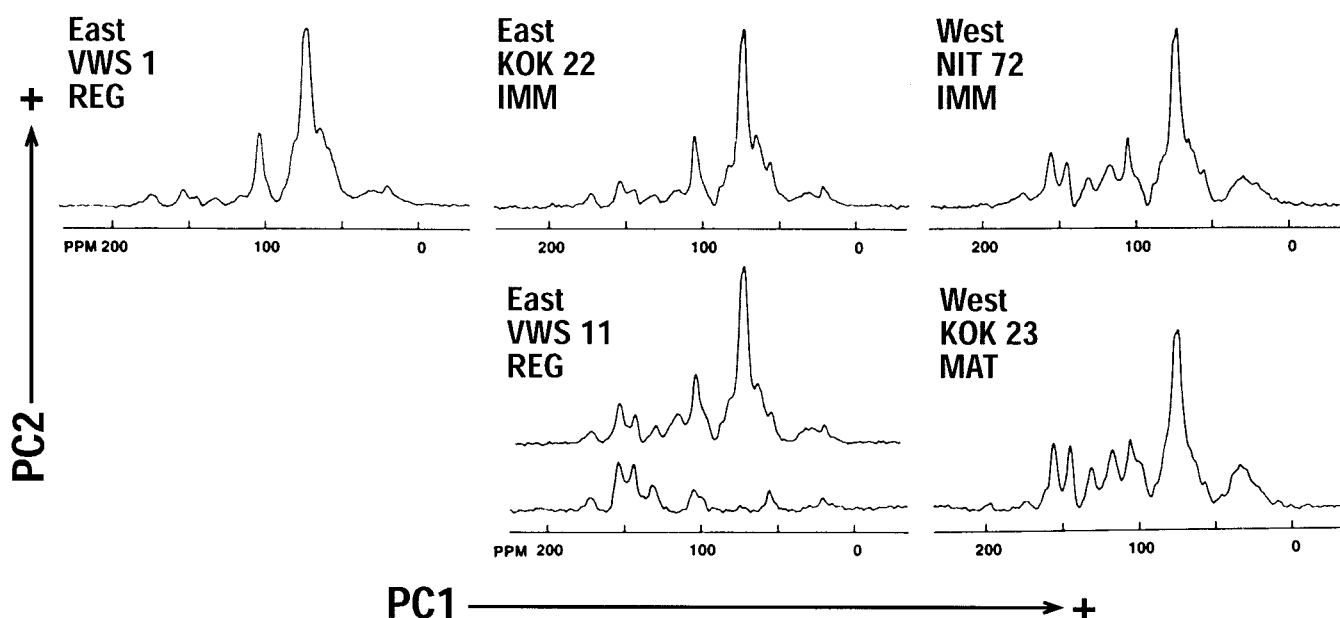
LFH-ROOTS

Fig. 4. Carbon-13 cross-polarization with magic-angle spinning nuclear magnetic resonance (CPMAS NMR) spectra for selected forest floor root (LFH-ROOT) samples, illustrating variation along Principal Component (PC) 1 and PC 2. The dipolar dephasing (DD) spectrum is also shown for Sample 11. REG, regeneration, 3 to 8 yr; IMM, immature, 25 to 45 yr; MAT, mature, 65 to 85 yr. VWS, Victoria Watershed South; KOK, Koksilah; NIT, Nitinat.

mow and Blackwell, 1998). The two samples with a distinctive woody signature were from West sites, consistent with its greater accumulation of CWD.

Nuclear Magnetic Resonance—Mineral Soil Fraction, <2 mm

The PC loadings for this fraction (Table 4) and the characteristics of the spectra (Fig. 6) indicate two trends. Increasing scores along PC1 (0.32 of variance) are associated with a change from samples with greater root–tannin character to more woody–lignin character, while samples with more negative PC2 scores are higher in carboxyl and alkyl C. There is considerable similarity to LFH, for which PC1 was also associated with increasing woody character. Spectra of both pools have broader features than FWD, LFH-ROOT, and MIN-ROOT, consistent with decomposition of biopolymer inputs, and although the signs are opposite, both show a gradient of alkyl and carboxyl C along PC2, which is much more obvious for MIN-FLOAT. An increasing ratio of alkyl C to O-alkyl C has been proposed as an indicator of decomposition (Baldock and Preston, 1995; Baldock et al., 1997; Preston, 1996).

There were some similarities between MIN-ROOT and MIN-FLOAT samples from the same plots. The strong wood–lignin fingerprint in MIN-ROOT 63 and 72 is carried over from the MIN-ROOT fraction, and also from LFH for Sample 72. High alkyl C intensity was found in both MIN-ROOT and MIN-FLOAT Plot

11, but only in MIN-FLOAT Plot 15. However, the high charcoal-type aromatic content of some MIN-ROOT samples was not found in the corresponding MIN-FLOAT samples.

Similar to FWD and LFH, there was some tendency for West samples to have higher positive scores on PC1, and thus a more woody character (Fig. 1e). However, there were no obvious seral stage or subzone effects

Table 4. Relative areas of ^{13}C nuclear magnetic resonance (NMR) spectra for two samples under different acquisition conditions.†

	Percent of total area for chemical shift region (ppm)					
	0–50	50–95	95–110	110–142	142–165	165–220
				%		
			25 MHz, CP			
MIN-ROOT Plot 11, 547 mg C g^{-1}	22	20	7	41	8	2
			75 MHz, CP			
	21	29	9	30	6	6
			75 MHz, SPE			
	12	14	8	46	12	9
			25 MHz, CP			
MIN-FLOAT Plot 11, 391 mg C g^{-1}	31	27	5	18	10	9
			75 MHz, CP			
	21	29	10	24	8	7
			75 MHz, SPE			
	19	28	12	24	10	9

† MIN-ROOT, mineral soil fraction, 2 to 8 mm; MIN-FLOAT, mineral soil fraction, <2 mm; CP, cross-polarization; SPE, single-pulse excitation.
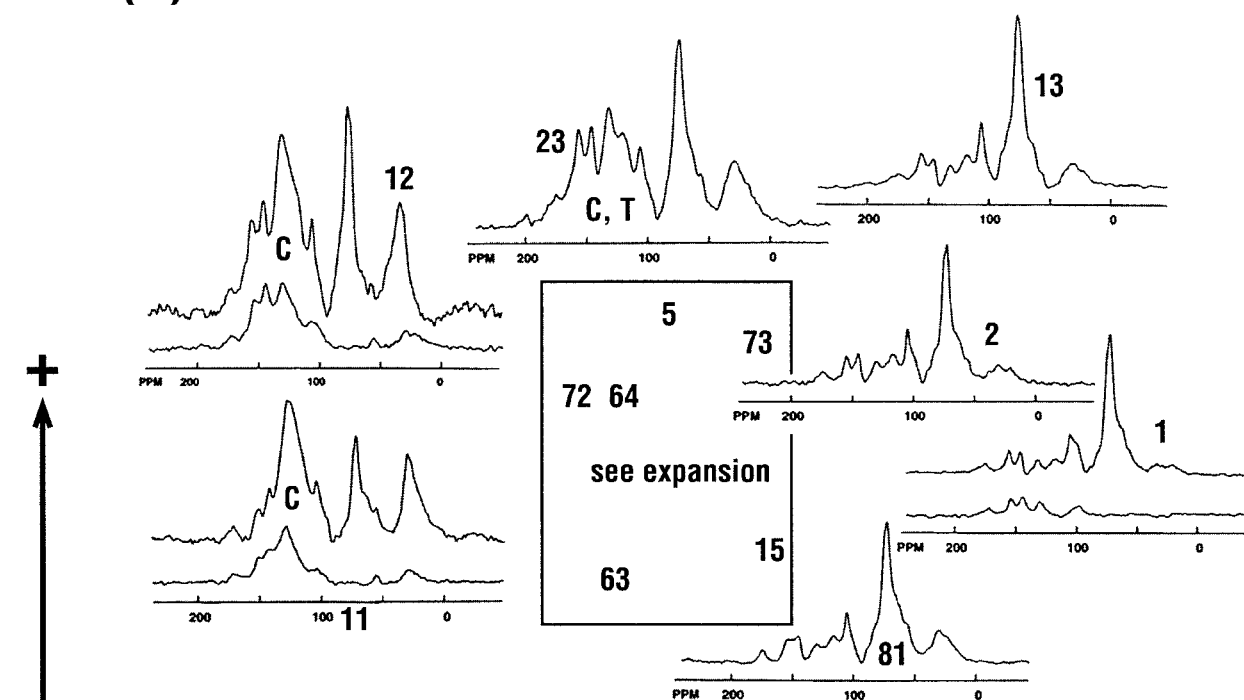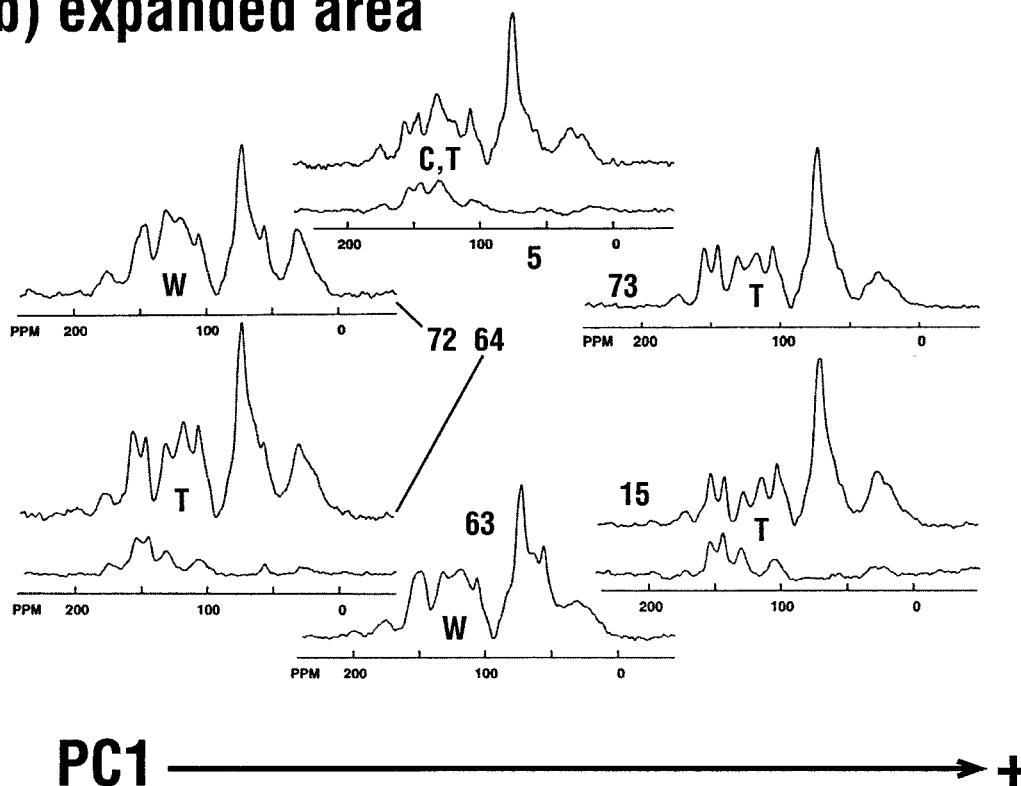
Fig. 5. Carbon-13 cross-polarization with magic-angle spinning nuclear magnetic resonance (CPMAS NMR) spectra for selected 2- to 8-mm mineral soil fraction (MIN-ROOT) samples, illustrating variation along Principal Component (PC) 1 and PC 2 (*a*). The central region with six samples in close proximity is expanded in (*b*). Inserts show dipolar dephasing (DD) spectra for selected samples. Strong features from wood, tannins, and char are indicated by W, T, and C, respectively.
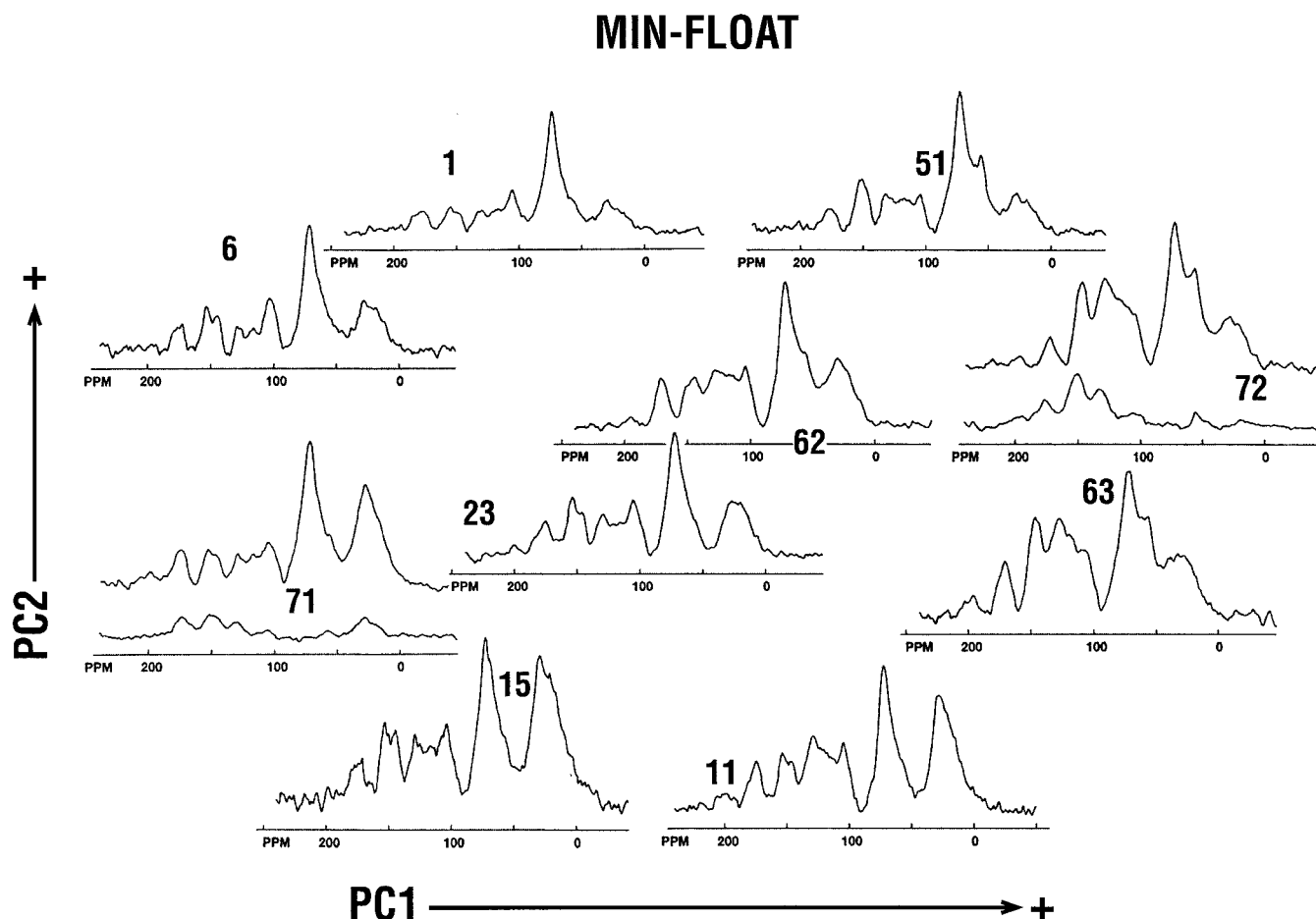
Fig. 6. Carbon-13 cross-polarization with magic-angle spinning nuclear magnetic resonance (CPMAS NMR) spectra for selected <2-mm mineral soil fraction (MIN-FLOAT) samples, illustrating variation along Principal Component (PC) 1 and PC 2. Inserts show dipolar dephasing (DD) spectra for Samples 71 and 72.

along the negative PC2 gradient associated with increased decomposition.

Quantitative Nuclear Magnetic Resonance Spectra

Pyrogenic C has recently been identified as a significant contributor to organic matter in some soils (Schmidt et al., 1999; Skjemstad et al., 1999; Smernik and Oades, 2000). It has low cross-polarization efficiency, because C nuclei in highly condensed aromatic structures are remote from H. Mobile long-chain alkyl C is another pool with low CP efficiency (Hu et al., 2000; Preston, 2001). This study was carried out at low magnetic field (25 MHz for ^{13}C), where spinning sidebands present little problem, but the sensitivity was insufficient for SPE spectra. To test for missing mobile or pyrogenic C, two samples were reexamined at 75 MHz, the MIN-FLOAT and MIN-ROOT fractions from Plot 11.

At 75 MHz, the normal CPMAS spectrum for MIN-ROOT 11 (Fig. 7a) shows serious distortion by SSB. These are suppressed in the TOSS spectrum (Fig. 7b) although some intensity may be lost. However, both TOSS and DD-TOSS spectra were useful for qualitative examination of lineshapes and positions (Fig. 7b,d), which were also used to refine the estimate of SSB area.

The DD-TOSS spectrum (Fig. 7d) reveals the broad tannin marker at 106 ppm, a relatively small methoxyl signal at 56 ppm, and some mobile long-chain C at 30 ppm. (Similar general properties can be seen in the corresponding DD spectrum at 25 MHz in Fig. 5a.) The SPE spectrum (Fig. 7c) has been corrected for spectrometer background, which in fact was very slight for this sample. With SPE, the proportion of aromatic C was 46%, compared with 30% for CP, while the sum of aromatic and phenolic C increased from 36 to 58%. There was also a slight shift in the peak maximum, from 127 ppm with CP to 129 ppm with SPE. This may be due to the increased prominence in the latter of more highly condensed carbon structures derived from charcoal rather than lignin and tannin.

Spectra with variation of contact time (0.5–10 ms, not shown) showed that detection of this aromatic carbon was not improved with longer CP time, similar to results of Freitas et al. (1999) for a laboratory-charred peat and our recent results (unpublished) for charred wood from a forest fire. Compared with the 25-MHz CP spectrum, that at 75 MHz mainly had higher relative intensity at 50 to 93 ppm and 165 to 220 ppm, and lower at 110 to 140 ppm. This may be largely due to inadequate correction for SSB intensity at 75 MHz.

By contrast, although the SPE spectrum for MIN-

MIN-ROOT 11

(a) CP

(b) TOSS

(c) SPE

(d) DD-TOSS
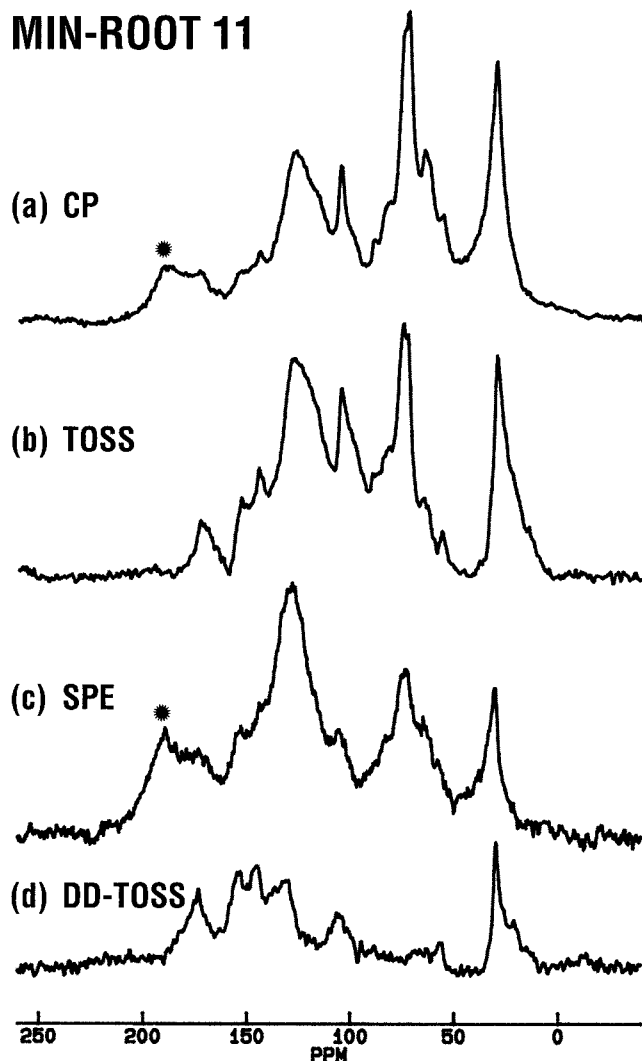
250   200   150   100   50   0
PPM

Fig. 7. Carbon-13 nuclear magnetic resonance (NMR) spectra at 75 MHz for the 2- to 8-mm mineral soil fraction (MIN-ROOT) Plot 11: (*a*) cross-polarization with magic-angle spinning (CPMAS); (*b*) CP with total suppression of sidebands (TOSS); (*c*) single-pulse excitation (SPE) after background correction; and (*d*) dipolar dephasing (DD) with total suppression of sidebands (TOSS). Main spinning sidebands (SSB) are indicated by *.

MIN-FLOAT 11

(a) CP

(b) TOSS

(c) SPE

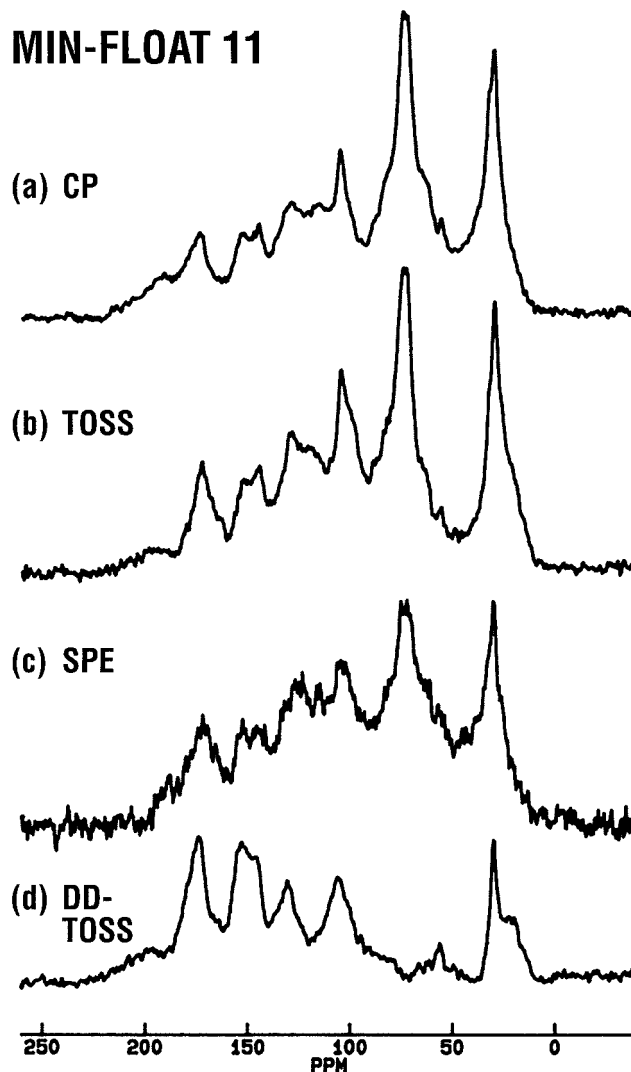(d) DD-TOSS

250   200   150   100   50   0
PPM

Fig. 8. Carbon-13 nuclear magnetic resonance (NMR) spectra at 75 MHz for the <2-mm mineral soil fraction (MIN-FLOAT) Plot 11: (*a*) cross-polarization with magic-angle spinning (CPMAS); (*b*) CP with total suppression of sidebands (TOSS); (*c*) single-pulse excitation (SPE) after background correction; and (*d*) dipolar dephasing (DD) with TOSS.

FLOAT Plot 11 (Fig. 8c) had broader features than the CP one (Fig. 8a), the relative areas were essentially the same, while the alkyl region showed the greatest discrepancy between 25 and 75 MHz. The DD-TOSS spectrum (Fig. 8d) was similar to that for MIN-ROOT 11, with mixture of lignin and tannin features, and some mobile C at 30 ppm. However, the bulk of the alkyl C appeared to be well detected by CP for both samples, and aromatic C derived from lignin was much better detected than that derived from char.

CONCLUSIONS

Carbon-13 CPMAS NMR can be used to characterize organic matter pools across forest landscapes. The spectra provide insight into inputs, decomposition processes, and nature of past disturbances for forest chronosequences in contrasting biogeoclimatic subzones. Subzonal influ-

ences predominated, with West sites generally showing a greater influence of high-lignin, woody residues in LFH and MIN-FLOAT. The influence of condensed tannins was seen in the two root fractions (LFH-ROOT and MIN-ROOT), also with increasing prominence in the West, while pyrogenic C was found in some MIN-ROOT samples from the drier East. In additional to subzonal differences, NMR spectra with PCA highlighted the importance of heterogeneity in the organic matter, resulting from differences in site and even stand history.

Both NMR and C and N analysis of the five pools, and a previous study of CWD (Preston et al., 1998), showed little effect of disturbance (seral stage) on organic matter quality after only one harvest, and that natural biogeoclimatic forces still dominate in these sites. Repeated cycles of human disturbance and intensive logging may be expected to perturb the nature of the organic matter, and hence the biodiversity and func-

tioning of these forests; specifically, the lower stocks of soil C in the East site may indicate a greater sensitivity to disturbance and intensive management.

ACKNOWLEDGMENTS

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