

Soil carbon content and character in an old-growth forest in northwestern Pennsylvania: a case study introducing pyrolysis molecular beam mass spectrometry (py-MBMS)

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“Capsule”: *Soil carbon stocks in a chronosequence of forest stands arising from natural disturbance were found to increase with increasing time since disturbance.*

Abstract

This study was conducted to: (1) test the utility of a new and rapid analytical method, pyrolysis molecular beam mass spectrometry (py-MBMS), for the measurement and characterization of carbon in forest soils, and (2) examine the effects of natural disturbance on soil carbon dynamics. An additional objective was to test the ability of py-MBMS to distinguish recent from more stable humic substances, and to relate this information to the ecology and history of the sites. To test the utility of the py-MBMS technique, we investigated soil carbon stocks in a chronosequence of stands arising from natural disturbance in the Tionesta Scenic and Research Natural Areas. Soil carbon increased with increasing time since disturbance; although the exact shape of the carbon accumulation curve is not known, it appears that the rate of carbon accretion is initially rapid and then levels off, with a possible maximum of 86 metric tons/ha to a depth of 30 cm. This study also demonstrates that py-MBMS is a valid method for characterizing soil carbon and can be used with little sample preparation. In addition, multivariate analysis of the mass spectra from Tionesta soils can distinguish both sites and depths on the basis of their pyrolysis products; both long-lived and short-lived carbon forms were identified. Published by Elsevier Science Ltd.

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1. Introduction

Soil carbon concentration and content are important properties of forest soils. Carbon is a major component of soil organic matter, which is closely linked to site productivity by improving water holding capacity, reducing bulk density, releasing nutrients, and providing much of the cation exchange capacity of the soil. Soil carbon also provides the energy source used by the belowground community in order to carry out the biochemical processes that drive nutrient cycling. In addition, soils are a substantial carbon reservoir, a role that is becoming increasingly important in the climate change and forest sustainability policy arenas. While many studies report organic matter content, soil organic carbon has often been omitted from forest management studies, which have traditionally focused on nitrogen and phosphorus. While the measurement is not technically difficult, sample preparation is time consuming

and therefore expensive; the cost incurred for large numbers of samples can preclude extensive soil organic carbon measurements. While loss-on-ignition is an inexpensive method, it is not recommended as the sole measure of carbon concentrations (Sollins et al., 1999).

As scientists strive to understand and quantify the global carbon cycle and the possible effects of global change, the role of soils in carbon sequestration is acquiring new prominence. Until we know the dynamics of carbon accumulation and understand how management actions affect those dynamics, including the amounts of carbon stored in both short- and long-lived carbon pools, it will be difficult to effectively manage forest soils as a carbon sink (McColl and Gressel, 1995). One of the first steps to improving our understanding is to develop rapid methods of soil carbon analysis that require little or no sample preparation, which will enable more numerous and complete investigations of soil carbon pools and dynamics.

Molecular beam mass spectrometry (MBMS) has been used successfully to analyze biomass samples (Agblevor et al., 1994; Evans et al., 1996; Tuskan et al., 1999) and

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offers rapid measurement of carbon concentration of whole soil samples with no sample preparation required. By performing pyrolysis on the sample before introduction into the spectrometer (py-MBMS), information on the chemical nature of the carbon can also be obtained. While Schulten and Leinweber (1999) used pyrolysis field ionization mass spectrometry (py-FIMS) to characterize soil carbon, the molecular beam method has not been previously applied to forest soils. Py-MBMS offers several advantages for rapid soil characterization: sample introduction and analysis are rapid, with results obtained within minutes, the instrument is robust and stable, allowing several hundred samples to be run in 1 day, and the method of sample introduction preserves high molecular weight products which may be lost when other methods are used (Evans and Milne, 1987).

Since accumulation of carbon in soils is believed to be a fairly slow process, direct observation generally cannot be used and many investigators rely on the chronosequence approach (Black and Harden, 1995; Compton et al., 1998). While valuable, there are difficulties inherent in the substitution of space for time (Pickett, 1989) and the lack of suitable sites often leads to a lack of replication. One of the greatest impediments to drawing sound conclusions from chronosequence studies is the lack of detailed land use history. Since the time to full soil recovery from a previous land use is not known, prior management or land use activities are likely to confound present day measurements. Without pre-treatment carbon data, reference values are based on areas of similar vegetation that are believed to be free of recent disturbance (Hix and Barnes, 1984; Pennock and van Kessel, 1997).

The Tionesta Scenic and Research Natural Areas, located on the Allegheny National Forest in northwestern Pennsylvania, offer a rare opportunity to investigate the effects of natural disturbance on soil carbon stocks. The land use history is well documented and the Areas have been essentially unmanaged for centuries—no timber management has occurred, though scattered trees were removed to create sites for oil and gas wells and pipeline rights-of-way. This lack of prior land use effects provides an excellent area for the study of soil carbon dynamics. Several large wind events have removed the overstory in portions of the Tionesta tract, leading to the establishment of forest stands of distinct age classes. Since the soils are fairly similar over the Areas and prior work documents that there is no effect of soil type on forest vegetation (Hough, 1942), the Areas provide a natural laboratory in which to investigate the effect of natural disturbance on forest soil carbon stocks. In addition, the absence of forest management activities facilitates the interpretation of carbon characterization data produced by py-MBMS.

This study had three main objectives: (1) to test the utility of the py-MBMS method for carbon analysis of forest soil samples, (2) to explore the effects of natural

disturbance on forest soil carbon dynamics in the Tionesta Scenic and Research Natural Areas (TSRNA), and (3) to use py-MBMS to distinguish recent from more stable humic substances and relate this information to the ecology and management of the area.

2. Materials and methods

2.1. Site description

The TSRNA comprise an important remaining remnant of the virgin beech–hemlock climax forest that once covered six million acres of the Allegheny Plateau. Remote and difficult to access, this 1672 ha tract escaped the turn of the century logging of the entire region and was purchased by the Federal Government in 1936. At that time, only the surface rights were purchased and some oil and gas activity has occurred in the tract. In the 1980s the subsurface rights were acquired, and while the oil and gas companies retain the right to work existing wells, new development is prohibited. The effects of these activities are manifested as small openings in the forest for wells and pipelines, and a network of unimproved roads that are closed to public access. The location of the TSRNA is shown in Fig. 1.

The climate is cool and humid, with average annual precipitation of 1067 mm and an average annual temperature of 8°C. The TSRNA are located on the northern unglaciated portion of the Allegheny Plateau, with stream-cut V-shaped valleys and flat uplands (Bjorkbom and Larson, 1977; Aguilar and Arnold, 1985). The soils are derived from shales and sandstones and are fine loamy, mixed, mesic Aquic Fragiudults that are acidic (pH 3.4–4.5). Forest vegetation is primarily beech–hemlock climax forest, with some hemlock trees $\approx$ 600 years old. Major wind events have led to the establishment of hardwood-dominated stands within the Areas. In this study, three such sites were used: a northern hardwood stand (maple–birch–beech), that arose after a blowdown in 1808, an Allegheny hardwood stand (cherry–maple) and

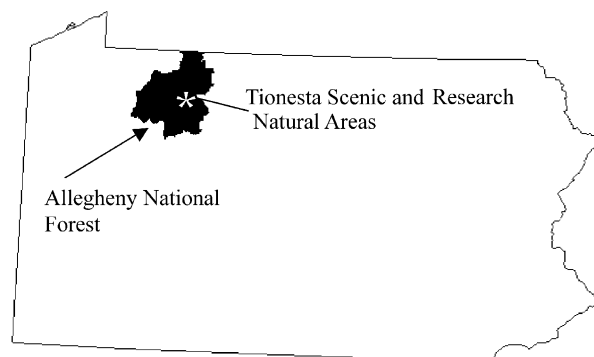


Fig. 1. Map showing general location of the Tionesta Scenic and Research Natural Areas.

that resulted from a wind event in 1872, and another northern hardwood (birch–beech) stand arising from a tornado in 1985. In addition, an undisturbed beech–hemlock stand was sampled. Stand ages were estimated using increment cores (Chris Nowak, unpublished data). All plots exhibit pit-and-mound topography, but within-plot slope is minor.

2.2. Sampling protocol

In each of the 1872, 1808, and 1985 blowdown areas and in the intact beech–hemlock stand, a 0.2-ha plot was established. In April 2000, 16 mineral soil cores were collected on a systematic grid in each study plot. Cores were 5 cm in diameter and were divided into 0–5, 5–15, and 15–30 cm depth increments. The 16 cores were composited systematically in the field by depth increment to form four samples per depth increment per plot for analysis. Since this was a pilot study, the sample stands were not replicated. Three intact cores were collected from each study plot for bulk density measurements. These cores were not composited, although they were separated into the depth increments described above. All forest floor material was discarded; while the forest floor is an important component of the forest carbon cycle, this study was established to test the utility of a new method for soil analysis. When the method has been further tested and refined, it will be applied to forest floor materials. Samples were transported to the laboratory and refrigerated 1–3 days until processing was complete. The soils were oven dried at 65°C for 48 h then passed through a 2-mm mesh sieve. Bulk density samples were oven-dried at 105°C for 48 h then passed through a 2-mm mesh sieve; all carbon content numbers refer to the rock-free fine soil fraction. Although analysis using the py-MBMS method does not require sample preparation, standard preparation was used to facilitate cross-comparison between standard dry combustion and MBMS results, and to reduce variability during this method-development phase. After preparation, soil samples were sent to the National Renewable Energy Laboratory for analysis by MBMS, and to an analytical laboratory (Penn State Agricultural Analytical Services Laboratory) for analysis by standard dry combustion methods (modified Dumas method, Nelson and Sommers, 1996).

2.3. Laboratory analyses

Samples analyzed by standard combustion analysis were ground to 40 mesh and then run on a Fisons NA 1500 Elemental Analyzer (PSU Agricultural Analytical Services Laboratory). Samples analyzed by MBMS were coarsely ground to reduce variability due to the 100-mg sample size, placed into quartz boats, and introduced into a quartz tubular reactor where the samples were

either combusted in O₂/He at 700°C or pyrolyzed in He at 500°C. The product gases then expand across an orifice into the first stage vacuum chamber of the MBMS. As this method is refined, larger sample sizes will be used, eliminating the need for grinding and fine sieving. In either pyrolysis or combustion mode, sample amounts from 1 to 1000 mg can be analyzed by this technique; larger samples can help overcome the natural heterogeneity of soils. Principal components analysis was used to identify patterns and structure in the sample set and relate these observations to the underlying chemistry of the soil organic matter.

3. Results

3.1. Method performance-MBMS vs. standard combustion

Soil carbon concentrations as measured by MBMS in combustion mode were similar to those from standard combustion analysis. All MBMS samples were run in triplicate and for this stage of development, reproducibility was good (S.E.M. for 0–5 cm increment triplicates ranged from 0.04 to 0.44). Initial variability was high but was considerably reduced after samples were coarsely ground prior to analysis. Fig. 2 shows the results of MBMS versus conventional combustion analysis for carbon concentration; the diamonds represent the average of the triplicate samples, while the open circles are a Carlos Erba soil reference material. At carbon concentrations of 3.5% and below, the relationship is clearly linear with excellent agreement between the two methods for this stage of method development (Fig. 3). When carbon concentrations are above 3.5%, the MBMS analysis consistently returns lower % C values than conventional combustion analysis.

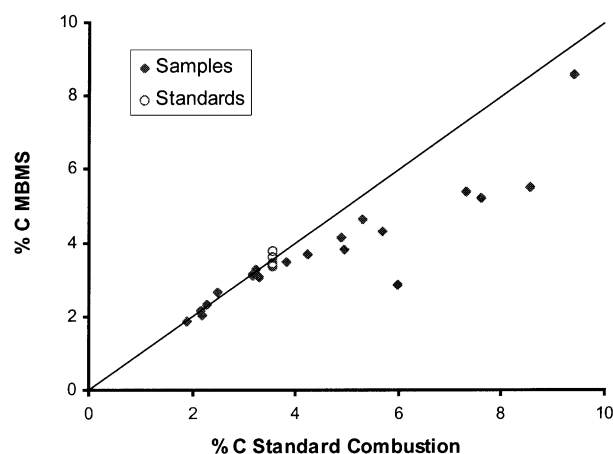


Fig. 2. Comparison of carbon concentration data by MBMS and conventional combustion analyses. Open circles are a 3.5% C soil reference material provided by Carlos Erba; solid line is 1:1 line for reference.

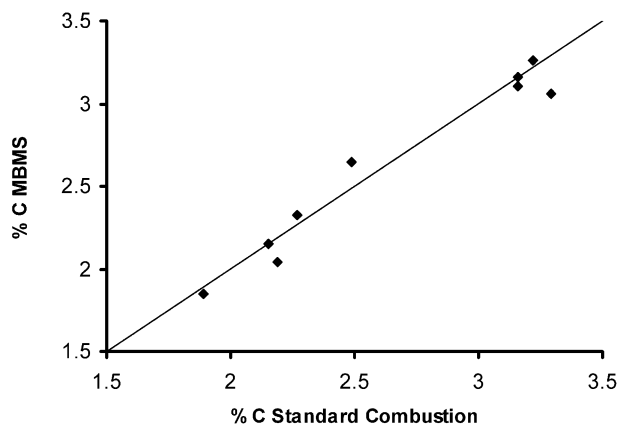


Fig. 3. Comparison of carbon concentration data by MBMS and conventional combustion analyses, 0–3.5% range only. Solid line is 1:1 reference line.

3.2. Carbon concentration and content

Carbon concentrations from conventional combustion analysis are given in Table 1 for each site and depth increment. Concentrations generally increased with time since disturbance, and ranged from a low of 2.46% in the 0–5 cm layer of the 1985 blowdown site to a high of 6.26% in the 0–5 cm layer of the 1808 stand. Carbon concentrations in both the 5–15 cm and 15–30 cm depth increments increased with time since disturbance, with the lowest values in the 1985 blowdown stand and the highest in the virgin beech–hemlock (VBH) site. The standard error of the mean for each site and depth are also given in Table 1; the higher values for the 0–5 cm layer at every site reflect the higher variability inherent in surface soils. Carbon content (the product of carbon concentration and bulk density) increased with time since disturbance, ranging from 45 metric tons/hectare (mt/ha) to a depth of 30 cm in the 1985 site to 86 mt/ha to a depth of 30 cm in the VBH site. The results for all depths and sites are given in Fig. 4.

3.3. Pyrolysis and factor analysis

Pyrolysis of the Tionesta soil samples produced multiple complex spectra; for such complicated data sets, simple data analysis by the comparison of two mass spectra is of limited use. Multivariate analysis (principal components analysis) has been shown to be a useful tool for pattern recognition in pyrolysis mass spectrometry to show the similarities and differences between samples, and the correlated behavior among the mass variables. We use this approach to first show the structure of the data set and how the patterns associated with site and depth are observable, and then to calculate the subspectra responsible for the differences that can be interpreted chemically. Factor analysis was performed for the average mass spectra for each sample; Fig. 5 shows the factor score plot for the 107 samples in the

Table 1
Carbon concentrations and standard error of the mean for each site

Depth	1985		1872		1808		VBH	
	%C	S.E.	%C	S.E.	%C	S.E.	%C	S.E.
0–5 cm	2.46	(0.20)	3.89	(0.91)	6.26	(0.76)	5.70	(1.06)
5–15 cm	1.40	(0.11)	1.69	(0.18)	2.59	(0.25)	2.92	(0.19)
15–30 cm	1.06	(0.18)	1.41	(0.11)	2.10	(0.32)	2.32	(0.18)

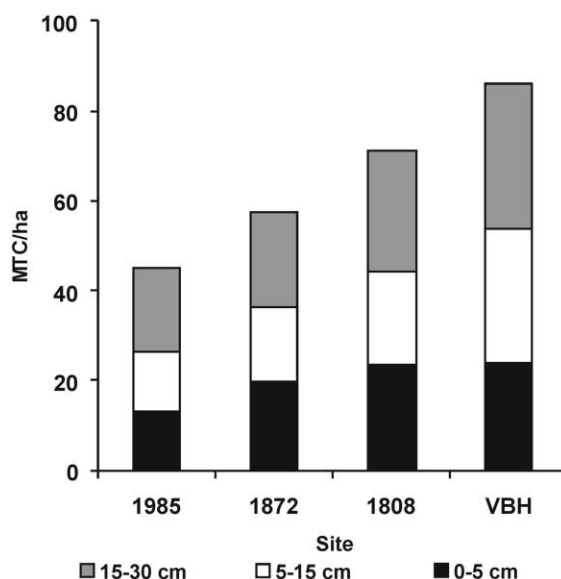


Fig. 4. Carbon content, in metric tons per hectare, for each site and depth increment.

database. The factor scores are weighted combinations of the original masses that were highly correlated and so reflect the underlying trends in the data set. Each mass variable is weighted by its correlation coefficient with the factors, and the factors are calculated to explain the maximum amount of variance in the data set and are orthogonal to each other. In this data set, the first four factors explain 35, 11, 6.5 and 3.7 % of the variance, respectively. The score plots are an efficient way to show the variance in the data set and the differentiation of classes of samples based on the py-MBMS results. The three ellipses show the groupings for soil depths and the symbols represent the different sites; Fig. 5 shows that both site and depth are clearly differentiated by MBMS analysis.

4. Discussion

4.1. MBMS method performance

MBMS and conventional combustion analysis results agreed well in the 0–3.5% C range (Fig. 3). At concentrations above 3.5%, it appears that the instrument is responding in a nonlinear manner and variability

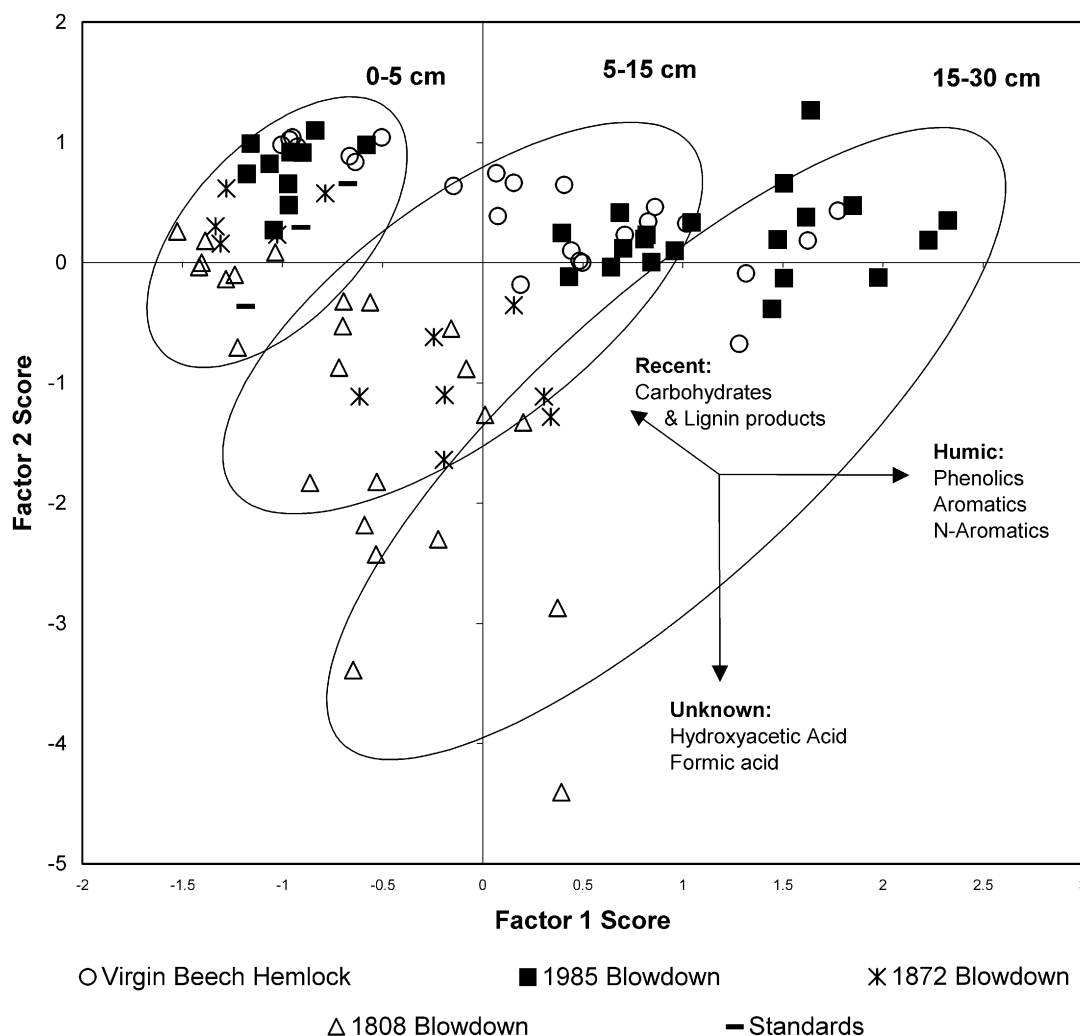


Fig. 5. Factor plot of data from pyrolysis spectra.

increased (Fig. 2). There are several possible reasons for this response: (1) presence of carbonate not detected by MBMS soil combustion, (2) incomplete MBMS combustion, or (3) instrument non-linearity. The first two explanations would explain the consistently lower MBMS results, but at the pH values encountered in this area, the presence of carbonate is unlikely. In the northeastern soils sampled by the USDA Forest Service Forest Health Monitoring (FHM) program, carbonate was rarely detected, and only at pH values of 7 or above (FHM database, 1999). Incomplete combustion is a viable explanation; conventional analysis included a burst of O_2 as the sample was dropped (D. Cousino, PSU Agricultural Analytical Services Laboratory, personal communication), while MBMS samples were run in a 4:1 He/O_2 mix.

This work reports the initial test of this method, which is under continuing development. Further work will include running a 5-point calibration on EDTA-based standards with MBMS and conventional analyses, increasing the O_2 to ensure complete combustion,

and running carbonate with MBMS to see if CO_2 evolves. If a nonlinear instrument response or consistent deviation from results by elemental analysis is confirmed, then sufficient data will be gathered to construct a calibration. Overall, the MBMS demonstrated good agreement between replicate samples as indicated by the calibration points in Fig. 3.

The MBMS in combustion mode can be used to measure total nitrogen and sulfur concentrations as well as carbon, although development to this point has focused on carbon measurements. One of the advantages of the molecular beam method is that, compared to other mass spectrometry methods, larger sample sizes can be used. Future work will utilize 0.5-g samples, which will eliminate the need for any preparation other than air drying and passing through a coarse mesh sieve to remove large fragments and roots. With continued methodological development, it will be possible to analyze unprocessed, field-moist samples with the same precision and accuracy as prepared soils. Our results suggest that MBMS is a promising method for soil

carbon analysis, and further improvements will make this method a rapid and reliable means of soil carbon, nitrogen, and sulfur analyses. The time savings from the elimination of sample preparation and the additional information gained from pyrolysis make this technology a valuable tool for soils research.

4.2. Effects of natural disturbance on soil carbon stocks: carbon content and character

Although this is an unreplicated pilot study, soil carbon content and concentration clearly differ between stands of different age classes, with carbon storage increasing with increasing time since disturbance (Fig. 6). In the wake of stand-replacing wind events, the forest type changed from beech–hemlock to northern hardwood (1808 and 1985 sites) and Allegheny hardwood (1872 site). The patterns of carbon storage seen in these sites may be a result of the physical disturbance from the blowdown events, the resulting change in vegetation, or a combination of the two. Since this study is an initial case study, additional sampling and analysis are required to draw firm conclusions about the causes of these observed patterns. However, these initial results do demonstrate that py-MBMS can provide an array of soil carbon information. Although Fig. 5 illustrates that all four sites can be distinguished, the VBH and 1985 sites occupy a similar space, indicating comparable carbon signatures and suggesting that the 15 years since disturbance were insufficient for a detectable change in carbon chemistry to have occurred. The 1808 and 1872 sites, however, contain carbon much higher in acids and lower in humics; the difference from the other sites is large and easily detectable. The change in signatures could be the result of the oxidation of debris and forest floor materials after disturbance, the shift in vegetation type and resulting change in litter input, or a combination of the two. Changes in the microbial

communities as a result of changes in vegetation may also be reflected.

Ongoing investigation includes searching for species-specific soil carbon signatures, identifying the carbon signatures of the litter and wood of the major species, and characterization of the microbial communities at each site. As the various signatures are established and the chemical identity of each component in the mass spectra is verified, our understanding of carbon dynamics following disturbance at TSRNA will improve, and the usefulness of py-MBMS as an analytical tool will increase.

One of the difficulties with modeling changes in soil carbon at TSRNA is that the shape of the carbon accumulation curve is not known, nor is the rate or maximum achievable level of carbon storage. Although the TSRNA provide only four well-documented age classes, we can begin to address these questions. Since the VBH site has been undisturbed for at least 600 years, the value of 86 mt/ha may be taken as a reasonable estimate of the maximum potential carbon accumulation for this geographic area. Although there are only four points in Fig. 6, these initial data suggest a logistic accumulation curve with rapid accretion during the first 200 years trending to slow accumulation over the next 400 years. Additional examples of each age class are slated for sampling, which should provide a clearer picture of carbon accumulation dynamics in the Tionesta tract. An interesting point to note is that if we assume that the 1985 site had a pre-disturbance carbon content similar to that of the VBH site, changes in carbon content were easily detectable 15 years after disturbance (Fig. 4), while changes in the carbon signature were not (Fig. 5), suggesting that different temporal scales are at work.

The ability to differentiate sites indicates that the method is sensitive to small differences in carbon signatures that should offer the ability to detect changes in carbon composition as a result of management activities. The long-term goal of this ongoing project is to quantify the amount of carbon in the short-lived and long-lived pools, and to detect changes in the relative amounts of carbon in those pools on short-term time scales. As this work progresses, we will continue to identify organic carbon constituents and learn more about how management affects the quantity and chemical nature of that carbon.

5. Conclusion

The data show that soil carbon increases with increasing time since disturbance in the Tionesta Scenic and Research Natural Areas. Although the exact shape of the carbon accumulation curve is not known, it appears that the rate of accretion is initially rapid and

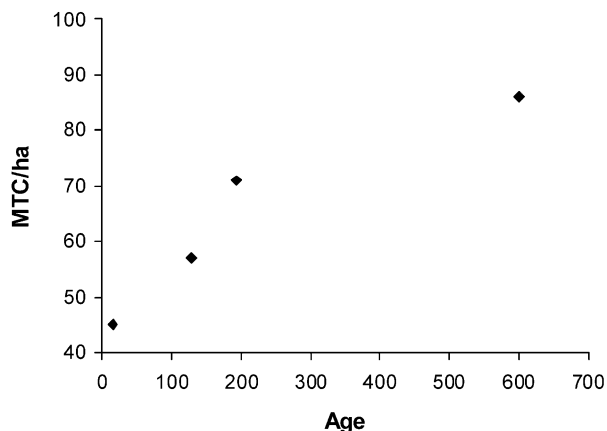


Fig. 6. Carbon content (metric tons/hectare) to 30 cm versus stand age.

then begins to level off, with a possible maximum of 86 mt/ha to a depth of 30 cm. This study also suggests that pyrolysis molecular beam mass spectrometry is a useful method for measuring soil carbon and can be applied with little sample preparation. In addition, multivariate analysis of the mass spectra from Tionesta soils can distinguish both sites and depths on the basis of their pyrolysis products; both long-lived and short-lived carbon forms were identified. With continued development, this analytical technique offers the opportunity to vastly expand our understanding of the role of soils in carbon sequestration and carbon cycling, and to assess how management practices can enhance that role.

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