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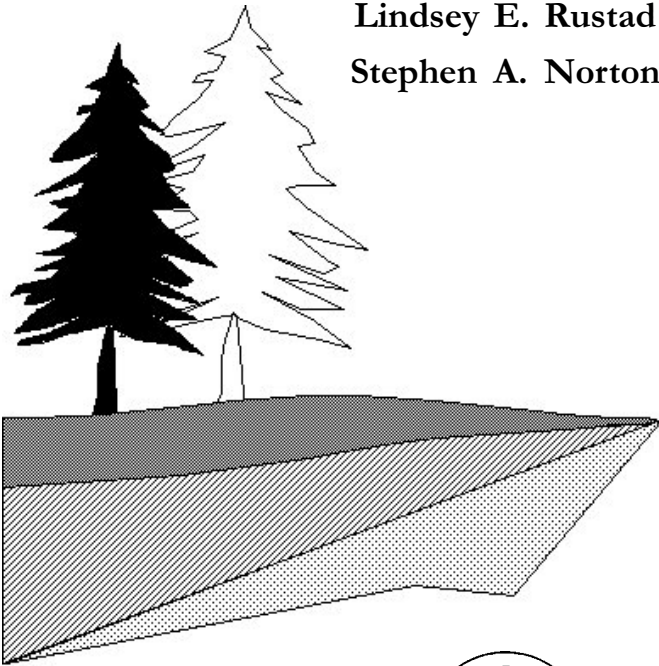
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INTRODUCTION

Since the beginning of the Industrial Revolution, humankind has drastically altered biogeochemical cycles on both ecosystem and global scales (Rastetter et al. 1991). Ultimately, global change may be driven by exponential population growth and concomitant resource consumption (Vitousek 1994). Currently, the concentration of atmospheric carbon dioxide (CO_2), a greenhouse gas, is a record 29% above its pre-industrial level, the highest level in the past 160,000 years (Flavin and Dunn 1998). Due to fossil fuel combustion, particularly coal, eastern North America was characterized by rapid increases in atmospheric sulfur (S) deposition from the mid-1800s to the mid-1900s, with a small decline since the 1970s (Husar 1986). Furthermore, both agricultural and industrial activity are responsible for increased inputs of atmospheric nitrogen (N) to many mid-latitude ecosystems (Peterson and Melillo,1985; NADP/NTN 1998). Elevated emissions of nitrogen oxides and ammonia are primarily due to the combustion of fossil fuels, the manufacture and use of fertilizers, livestock waste, and the burning of biomass (Galloway et al. 1995). Estimates of dry and wet N deposition to temperate ecosystems are more than 14 Tg (10^{12}g) per year (Melillo et al. 1989). Galloway et al. (1994) predicted that global N emissions may double by 2020, while emissions in the U.S. and Canada may increase by nearly 33%. The effects of global change are extensive, ranging from proposed climate change to acidic deposition, but the changes collectively affirm that the earth's nutrient balance has been drastically altered.

A major question of how terrestrial ecosystems will respond to these regional and global changes regarding carbon (C) remains unanswered (Rastetter et al. 1991). The existence of the "missing C sink," the imbalance of emissions, atmospheric accumulation, modeled ocean uptake, and emissions from changing land use, shows our lack of understanding of C storage and cycling. Recently, the Kyoto conference, challenging the industrialized world to reduce greenhouse emissions, provided an impetus to discern ecosystem C source/sink relationships (Kok et al. 1998).

Interactions among nutrient cycles may determine whether ecosystems function as nutrient sources or sinks (Rastetter et al. 1991). Specifically, links between C and N cycles may regulate the distributions, amounts, and turnover rates of C in N-limited forest systems. Biomass production and decomposition represent two such linkages. Carbon allocation and efficiency of nutrient use are key properties of overall plant function, controlling the quantity and quality of forest litter (Aber and Melillo 1991). Subsequent litter

decomposition partitions C flow into pools with various turnover rates (King et al. 1997). The balance between decay rates, dependent upon litter quality, climate, moisture/aeration, faunal activity, and microbial populations (Fogel and Cromack 1977), and C input will inevitably determine the persistence of soil organic matter.

A viable option for reducing atmospheric CO_2 may be C sequestration by agricultural and forested regions (Apps and Price 1995). The goal of terrestrial C sequestration is to develop management practices that favor the formation of stable forms of C. Forest ecosystems represent 60% of the terrestrial carbon budget (Lal et al. 1995), which suggests that forest management and forest disturbances may have a profound effect on C cycling. Quantifying and predicting the capacity of C sequestration in soils is impaired by the lack of accurate and universally accepted methods to measure the residence times associated with different forms or fractions of soil C. Nevertheless, as atmospheric CO_2 concentrations increase, the need for assessment techniques for soil C become increasingly important.

This publication was developed as part of an effort to evaluate the existing methodologies for determining C fractions in soils that might be applied to the question of forest soil C sequestration. A great deal of research has been done on this topic although often focused on agronomic soils. Forest land managers will be increasingly interested in identifying methods to monitor and to evaluate the effects of forest practices on soil C reserves. As well researchers are interested in this and the logical linkages to N cycling. Ultimately practical methods that can be widely utilized will be needed; these may come from current methods or be developed through research. This review offers a framework for this area of investigation.

INSIGHTS FROM THE LITERATURE

Soil organic matter (SOM) consists of a heterogenous mixture of interacting polymers (Sposito 1989). SOM originates from C fixed by plants delivered to the soil in the form of leaf and wood litter, roots, and root exudates. SOM is difficult to study due its heterogeneous nature, and SOM turnover rates range from days to millennia (Trumbore 1997; Stevenson and Cole 1999). Globally, soil organic matter contains approximately 1500-1600 x 10^{15} g C, and is more than either the atmosphere (750 x 10^{15}g) or terrestrial vegetation (500–800 x 10^{15}g) (Post et al. 1990). The rate of soil C turnover, a microbially mediated process, is dependent upon the quality of the original plant substrate (Meentemeyer 1978; Melillo et al. 1983; Stump and Binkley 1992) and

the soil physical environment, including temperature (Rustad and Fernandez 1998), particle-size and mineralogy (Baldock et al. 1992; Feller and Beare 1997), pH and oxygen availability (Bunnell et al. 1977), and aggregate formation and destruction (Tisdall and Oades 1982; Oades 1984).

Evaluating the size of C pools and C sequestration potential is presently one of the most serious and complex areas in environmental science (Trofimov et al. 1997). A universally accepted model for C turnover is nonexistent. Carbon turnover models generally include three components: litter inputs, one or more pools of SOM, and microbial biomass responsible for litter and SOM transformations (Christensen 1996; McGill 1996). Litter inputs are typically divided into decomposable and resistant plant material (Jenkinson and Rayner 1977). Other models divide litter inputs (and SOM) into three pools: a readily decomposable pool (water solubles, sugars, and proteins), structural cell wall materials (cellulose and hemicellulose), and a resistant pool of lignified materials (Hansen et al. 1991). The microbial biomass may be partitioned into labile and physically protected pools (van Veen et al. 1984). Regardless of the model used to categorize carbon pools, each pool is assigned a speculative turnover rate. These models are inadequate for predicting the complicated processes that control soil C turnover. No standard techniques exist for measuring the size of various pools of soil C; however, several techniques have been used to partition soil into biologically meaningful C fractions.

Chemical Fractionation

Dissolved organic matter/water soluble organic matter

Dissolved organic matter (DOM) is operationally defined as organic matter, in a solution, that can pass through a 0.45-Fm filter (Thurman 1985). Water soluble organic matter (WSOM) is defined as the SOM extracted with water or dilute salt solutions that can pass through a 0.45-Fm filter. Hot and cold water have been used to extract WSOM from both litter and SOM (Davidson et al. 1987; Sikora and McCoy 1990; Jandl and Sollins 1997). Samples may be analyzed colorimetrically with anthrone or with a total organic C analyzer (Davidson et al. 1987). The amount of WSOM extracted is dependent upon the chemistry of the extractant, which includes the ionic strength, pH, and the dominant anion (Herbert and Bertsch 1995).

Dissolved organic matter is a complex mixture of organic compounds, ranging from simple acids and sugars to complex humic substances. Dissolved organic C (DOC) is released from microbial

activity, root exudation, and leaching of organic matter (Schiff et al. 1997). DOC is plausibly the most labile and mobile form of C in SOM (Boyer and Groffman 1996). Water soluble C, determined to degrade rapidly (Jandl and Sollins 1997), may be an immediate energy source for microorganisms (Huang and Schoenau 1996). However, the nature of DOM is not fully understood because of its complexity. Therefore, DOM is typically partitioned into fractions based on solubility, molecular size, and sorption chromatography. The solubility of organic matter has traditionally been used as a basis for fractionation and is further discussed as the classical fractionation method. Thurman and Malcolm (1981) and Leenheer (1985) outline methods to determine the solute characteristics of solutions rather than soil. Three techniques are commonly employed to partition DOM and WSOM according to molecular size: gel permeation chromatography, ultrafiltration, and centrifugation (Herbert and Bertsch 1995). Fractionation of DOM by sorption chromatography separates organics in solution into hydrophobic and hydrophilic acids, bases, and neutrals (Leenheer and Huffman 1979). These techniques may provide insights on the nature and cycling of DOM and WSOM.

Hydrolysis

Carbohydrates represent approximately 5% to 20% of the total soil organic C and originate from plants, animals, and microorganisms (Stevenson 1994). Soil carbohydrates are composed of a mixture of complex polysaccharides, which are composed of monosaccharides. Five monosaccharides, including glucose, galactose, mannose, arabinose, and xylose, typically represent more than 90% of the total hydrolyzable carbohydrates (Chesire 1977). Soil carbohydrates contribute to the formation and stabilization of soil structure and have been primarily studied because of their relation to soil aggregation. Several studies have found positive correlations between soil carbohydrate concentrations and soil macroaggregate stability (e.g., Haynes and Swift 1990; Angers et al. 1993), although others have not (e.g., Carter et al. 1994). Hydrolysis procedures are commonly used to extract carbohydrates, sugars, amino acids, and amino sugars (Lowe 1993). Sulfuric acid is commonly used for hydrolysis, and the determination of saccharide concentrations can be done colorimetrically or using gas or liquid chromatography. In general, the amount of carbohydrates extracted increases with increasing acid concentration and temperature of hydrolysis (Gregorich et al. 1994).

Polysaccharides, because of their chemical structure, are likely an available source of energy for organisms (Chesire 1977), and have consequently been specifically extracted from the soil. Although

physical protection of polysaccharides may reduce their bioavailability (Gregorich et al. 1994), Lowe (1993) described a methodology to extract both total and labile polysaccharides. In both procedures, saccharide monomers are released by hydrolysis with sulfuric acid, then an estimation of the total sugar concentration in the hydrolysates is measured colorimetrically. This procedure may only be used with mineral soils with low polysaccharide concentrations or organic horizons with up to 40% organic matter. Lowe (1993) suggested that polysaccharide recovery may be incomplete with hydrolysis, due to the heterogeneity of soil polysaccharides. Stevenson (1994) also outlined a method to extract polysaccharides with NaOH, HCOOH, and hot water.

The ratio of [galactose plus mannose] to [arabinose plus xylose] in soils has been used to distinguish plant- and microbial-derived carbohydrates (Oades 1984; Hu et al. 1997). Pentoses, such as arabinose and xylose, are constituents of plant residues and are not readily synthesized by microbes. Galactose and mannose are sugars produced by microorganisms. Therefore, the determination of a ratio of these sugars allows for the identification of the source of carbohydrates. The procedure includes a digestion with $12\,M\,H_2SO_4$ and $1\,M\,H_2SO_4$. The samples are then analyzed for arabinose, xylose, galactose, and mannose on a gas chromatograph with a flame ionization detector (FID).

Lignin concentration and the lignin:N ratio are key factors that determine forest litter turnover rates (Meentemeyer 1978; Melillo et al. 1983; Stump and Binkley 1992); therefore, methods that determine lignin fractions are commonly used to estimate detrital C turnover. Forage fiber analysis (Kononova 1961; Fogel and Cromack 1977; Melillo et al. 1983) and forest-products analysis (Berg and Staaf 1980, 1981; McClaugherty and Berg 1987; Melillo et al. 1989) are commonly used fractionation schemes for litter tissue. However, Fernández et al. (1997) used a similar procedure outlined by Kononova (1961) to determine lignin concentrations in Humic Cambisols (0-15 cm) under a *Pinus sylvestris* forest in Spain. Both forage fiber and forest-products techniques involve acid hydrolysis with H_2SO_4. The forage fiber technique (Goering and Van Soest 1970) determines cellulose, lignin, and hemicellulose. The forest-products methodology partitions C into hydrolyzed carbohydrates, lignin, simple sugars, water soluble phenolics, and waxes, fats, and oils. Ryan et al. (1990) advocated a combined technique of extracting polar (simple sugars and polyphenols) and nonpolar (fats, oils, and waxes) fractions with the forest products method, and lignin and cellulose with the forage fiber technique.

Paul et al. (1995) suggested that a multi-step acid hydrolysis procedure may be an effective technique to extract labile constituents in the soil. The procedure includes two sequential acid hydrolysis steps using hydrochloric acid (0.5 N HCl and 6.0 HCl) followed by further fractionation with sodium hydroxide and water. Microorganisms labelled with ^{14}C were added to the soil prior to extraction as a means of detecting the lability of the extracts. The residue, representing the stable fraction, contained a small fraction (6%) of the microbial biomass.

Classical fractionation

The classical chemical fractionation based on the solubility of organic matter in alkaline and acid solutions yields humic acids (HA), fulvic acids (FA), and humin (Sposito 1989; Stevenson 1994). Fulvic acids have low molecular weights and are soluble in both strong acids and bases. Humic acids are soluble in bases, but precipitate in strong acids. The humin fraction is commonly defined as the portion of soil humus that remains behind after extraction with dilute alkali. FAs may have residence times of hundreds of years, whereas HAs and humin may have residence times of thousand of years (Paul and van Veen 1978). However, Kögel-Knabner (1992) suggested that the humin fraction is less humified than HA and FA fractions. HAs extracted with $0.1M\ Na_4P_2O_7$ are more aromatic than HAs extracted with 0.5M NaOH (Schnitzer and Schuppli 1989). Catroux and Schnitzer (1987) extracted residual humic acids by treating the residue of HA extraction with HCl-HF. The residual humic acids compared to traditional HAs were more homogenous, contained fewer proteinaceous materials and carbohydrates, were more aromatic, and had higher molecular weights.

Sequential extraction

Schnitzer and Schuppli (1989) described a sequential extraction of SOM that determines the major constituents and relative proportions extracted in each step. The sequence includes n-hexane, chloroform, $0.1M\ Na_4P_2O_7$ solution under N_2, 0.5M NaOH solution under N_2, and distilled water. The n-hexane extracted alkanes and fatty acids; chloroform extracted fatty acids, waxes, and long-chain alcohols; $Na_4P_2O_7$ removed SOM associated with metals and clays; NaOH and water partitioned free SOM. The NaOH solution removed more C and N from larger particle size fractions, while $Na_4P_2O_7$ removed more C and N from finer- rather than coarser–grained particle fractions. Because larger particles are thought to be associated with less decomposed materials, they concluded that the NaOH removed free SOM and $Na_4P_2O_7$ extracted complexed materials.

Oxidation

The Walkley-Black (1934) wet oxidation procedure also partitions SOM. Walkley and Black modified the titration method used by Schollenberger (1931) by omitting the external heat source in the procedure. The technique provides for the incomplete oxidation of organic C (Ulmer et al. 1992). SOM is oxidized with a dichromate solution and then titrated with ferrous ammonium sulfate. This procedure oxidizes approximately 76% of SOM and is commonly used to estimate total soil C concentrations (Ulmer et al. 1992).

Actual oxidation may not mirror the oxidative potential of a given soil. Site conditions such as temperature, moisture/aeration, and pH may alter rates of C turnover. The procedure assumes that the oxidation state of C is initially zero, although aromatic compounds have an oxidative state below zero (Tiessen and Moir 1993). In addition, the reduction of dichromate is affected by the presence of Fe^{2+}, Mn^{2+}, and Cl^-, which may result in an overestimation of the quantity of the easily oxidizable C fraction (Ulmer et al. 1992; Tiessen and Moir 1993). Chromic acid has been classified by the United States Environmental Protection Agency as a carcinogen, thereby making waste disposal a problem where municipal restrictions apply (Ulmer et al. 1992). Nevertheless, the Walkley-Black procedure is often used to determine the easily oxidizable fraction of SOM because of its low cost and quick procedure.

Similarly, Blair et al. (1995) outlined another procedure to fractionate soil C based on the degree of oxidation. The amount of C oxidized by $KMnO_4$ is compared to total soil C concentrations. The authors derived a lability index (LI) from the ratio of oxidized C to unoxidized C. A C pool index (CPI), used to detect changes in C pools, may be calculated by comparing the total C of a sample to a reference. In experiments the reference sample may be taken from a control site. The total pool size and labile pool are used to calculate a C management index (LI x CPI x 100). They concluded that the CMI offers a sensitive index of C change and can be used in experimental situations to determine the treatment effects on C pools.

Alkaline CuO oxidation is thought to release phenols from reactive sites of the lignin macromolecule. The sum of vanillyl, syringyl, and cinnamyl phenolic CuO oxidation products is used as a relative measure of total lignin concentrations. This procedure is used for plants (Hedges and Mann 1979) and for soils (Kögel 1986). The mass ratios of acids to aldehydes of the vanillyl and syringyl units are used to determine the degree of lignin oxidation. Zech et al. (1996) used this technique in forest soils to clarify lignin degradation.

Supercritical fluids

Supercritical fluids (SCFs) are compounds that have been raised past their critical temperature and pressure. A critical temperature is defined as the temperature above which a gas will not condense, regardless of the pressure applied. The critical pressure is defined as the pressure above which a liquid will not vaporize, regardless of the temperature applied. Thus, a SCF is able to diffuse and to move like a gas and to dissolve materials as a liquid does (Sanchez and Ruark 1995). They are commonly used for the extraction of specific compounds from complex mixtures (Schnitzer and Preston 1987). Supercritical fluids are considered a relatively mild and efficient extraction mechanism due to their high densities, high diffusion coefficients, and low viscosities (Schnitzer and Preston 1987). Organic matter extractants are often characterized by infrared and nuclear magnetic resonance spectroscopy, pyrolysis-field ionization mass spectrometry (Py-FIMS), and chemical methods.

Supercritical CO_2 was determined to be a mild and specific extractant for long-chain aliphatic materials (Shulten and Schnitzer 1991). Schnitzer et al. (1991) investigated the use of distilled water at constant pressure of 17.2 MPa at various temperatures. The results indicated that extracts at 200EC contained the highest concentrations of carbohydrates and N components. Investigation with ^{13}C-NMR indicated that in general the C in the extracts was primarily non-carbohydrate aliphatic C bonded to OH and O. PY-FMS demonstrated the presence of polysaccharides, n-fatty acids, n-alkanes, n-alcohols, sterols, N compounds, and mono- and dilignins. Schnitzer and Preston (1987) indicated that proportions of alkanes, alkanoic acids, and carbohydrates in extracts decrease as the polarity of the solvent increases.

Sanchez and Ruark (1995) have attempted to use the SCF extraction technique to separate organic matter into labile and recalcitrant pools using Freon-22 (chlorodifluoromethane). Freon-22 was selected as the solvent, because it produced results superior to CO_2 and NO_2. The authors avoided stronger solvents (e.g., methanol and acetone) because these solvents have high critical temperatures (240.0 and 235.5EC) that could lead to the thermal degradation of compounds. The technique assumes that the recalcitrant pool has a higher molecular weight and stronger Van der Waals and dipole-dipole interactions than the labile fraction. Therefore, the solvated fraction is thought to be the labile pool while the soil remaining is considered the recalcitrant portion.

The extractions were spectrally characterized (liquid state ^{1}H NMR), revealing the presence of phenolic, aromatic, and carboxyl

functional groups (Sanchez and Ruark 1995). Aliphatics, thought to be resistant to microbial decay, were not found in large quantities in the extractant. However, carbohydrate-type materials (O-alkyl), a portion of the labile pool, were not found in the extractant. After further investigation the authors concluded that O-alkyl C might not have been present in the original sample. They surmised that these labile substances might have already been microbially consumed before the soil sample was collected.

The C concentration after the extraction was statistically different from the original sample for two of three soil types sampled (Sanchez and Ruark 1995). The C concentration in the remaining soil was used to indicate a baseline or recalcitrant value for each soil. The data indicate that both the labile and recalcitrant pools changed seasonally. Sanchez and Ruark (1995) suggested that the value for the baseline measurement may vary from season to season if portions of the labile fraction are associated with humic macromolecules and have not been separated by microbial degradation. To determine if the technique is extracting a portion of the recalcitrant C pool, Sanchez and Ruark (1995) recommended that the method be evaluated further by ^{14}C dating.

<div align="center">Physical Fractionation</div>

The chemical structure of SOM is thought to be insufficient to characterize C turnover (Duxbury et al. 1989), thus physical fractionation techniques, considered less destructive than chemical extractions, are commonly employed. Physical fractionation provides information concerning the architecture of SOM, determining the extent to which residues have been biologically processed and the degree of physical occlusion or organo-mineral complexation (Ellert and Gregorich 1995). Physical protection is thought to hinder the access of enzymes and microbes, lowering turnover rates.

Density

Density fractionation is based on the observation that during the humification process, SOM becomes intimately associated with mineral portions of the soil (Barrios et al. 1996). Thus, more humified particles are thought to be associated with the mineral fraction. The light portion, having a density less than soil minerals, is assumed to be mineral-free. Heavy fractions may be separated by centrifugation and the light fraction decanted. Alternatively, the light fraction may be removed from the solution surface by suction after the heavy fraction is allowed to settle (Strickland and Sollins 1987).

Light (LF) and heavy fractions (HF) are typically obtained using liquids (organic or inorganic) with a density range from 1.6 to 2.2 g cm^{-3} (Christensen 1992; Gregorich and Ellert 1993). Densities below 2.0 g cm^{-3} may yield a light fraction with higher proportions of macroorganic matter (Christensen 1992), and relatively large fragments of non-humified plant residues (Ellert and Gregorich 1995). Solutions of sodium iodide (Sollins et al. 1984) and sodium-polytungstate (Cambardella and Elliot 1992; Cambardella and Elliot 1994) are widely used for density fractionation. Meijboom et al. (1995) suggested that stable silica suspensions (Ludox™) offer a plausible alternative to inorganic salts, which require long equilibration periods and are typically expensive and toxic. However, the high viscosity of Ludox™ may cause incomplete separation of small particles (Magid et al. 1996). Ludox™ stimulates mineralization, whereas sodium polytungstate may retard CO_2 evolution (Magid et al. 1996).

Christensen (1992) noted that LF yields are extremely sensitive to changes in the density of the fractionation liquids; therefore, he concluded that choice of density and temperature control during processing are critical factors, especially in the 1.9 to 2.4 g cm^{-3} range. Richter et al. (1975) recovered 50% more C when the density of a bromoform-ethanol mixture was raised from 1.9 to 2.0 g cm^{-3}. Light fractions may contain mineral particles, indicating that dispersion procedures are inadequate or critical densities commonly used are too high to isolate non-complexed SOM (Christensen 1992; Spycher et al. 1983).

The non-complexed or LF, composed of decomposing plant and animal tissues, is generally thought to have a rapid turnover, whereas the HF remains as a more recalcitrant fraction (Janzen et al. 1992; Ellert and Gregorich 1995). The LF is enriched in carbohydrates, although the origins of the carbohydrates are not distinct (Dalal and Henry 1988). Kanazawa and Filip (1986) concluded that more than 50% of microbial populations and enzyme activities occur in the LF. Using substrate induced respiration (SIR), Hassink (1995) suggested that the microbial biomass in the LF represents the active microbial biomass (not physically protected). Janzen et al. (1992) also demon-strated a correlation between the LF and the respiration and microbial N content, concluding that the LF functions as an indicator of labile SOM. Similarly, Dalal and Mayer (1987) reported that N (per unit fraction weight) declined more quickly in the LF than the HF for soils from a 20- to 70-year cropping sequence. Studies with isotopic tracers indicate that LF organic matter has a faster turnover rate than HF

(Gregorich et al. 1995), although the LF may be partitioned into pools with different turnover rates (Bonde et al. 1992).

Spycher et al. (1983) investigated the distribution of the LF within the soil profile of a 70-year-old Douglas-fir stand. The LF in the 0- to 3-cm depth increment of the mineral soil consisted of 53% of the total C and 45% of the total N. The proportions of LF N and C decreased dramatically in the 3- to 13-cm layer and then steadily decreased to 83 cm. The authors suggested that non-complexed SOM is less important as soil depth increases and that the downward percolation of dissolved organics is more important. Seasonal fluctuations in LFs were also demonstrated in this study, particularly in the upper mineral soil. The authors concluded that LF inevitably represents an important labile reservoir of C in forest ecosystems. Sollins et al. (1984) showed that net N mineralization (as a proportion of total N) was negatively correlated to LF C/N ratios and positively correlated to HF C/N ratios in Oregon forest soils. They found that HF (per unit weight) was a greater source of N (anaerobic incubation) in five of six forest soils. Furthermore, net N mineralization for the HF was greater than that for the whole soil, suggesting that the LF may immobilize released mineral N. Because LF materials are more chemically and physically similar to source materials than HF materials, LF decomposition tends to be N-limited. Heavy fraction materials are thought to be significantly humified and decomposed. Thus, HF decomposition tends to be C-limited, explaining why high HF C/N ratios encourage decomposition. Boone (1994) also demonstrated that the HF, although the primary long-term C sink, responded to short-term changes in litter input. He concluded that density fractionation does not clearly partition SOM into active and stable pools, suggesting future clarification on the transfer rates between HF and LF, distribution of microbial biomass, physical and chemical mechanism for SOM protection, and relationship between the active HF and LF.

Macroorganic matter

Large particles of organic matter, macroorganic matter, are separated from SOM by sieving or floatation and sieving. The size of macroorganic matter varies among investigations, although it is typically the sand-sized fraction (Ellert and Gregorich 1995). Fungal hyphae, seeds, spores, and faunal skeletons are all components of macroorganic matter (Gregorich and Janzen 1996). An investigation with ^{13}C NMR, suggested that the highest proportion of C in macroorganic matter and LF consists of carbohydrates (Baldock et al. 1992; Golchin et al. 1994a; 1994b).

Particulate organic matter

Cambardella and Elliot (1992) isolated a particulate organic matter (POM) C fraction in grassland soils. POM was derived by dispersing soil in 5 g L^{-1} hexametaphosphate and passing it through a 53-Fm sieve. The material remaining on the sieve was identified as POM. They suggested that POM represents the C fraction lost during the cultivation of grassland soils and can be equated to the LF as defined by Greenland and Ford (1964).

Particle-size

Particle-size fractionation is based on the observation that C in the sand fraction is generally more labile than C in clay and silt size fractions (Tiessen and Stewart 1983). The association of SOM with clay and silt particles is a dominant mechanism of physical protection (Theng 1979) and is important in determining the stability of SOM. Particle-size fractionation divides SOM into size classes by sieving (dry/wet) and sedimentation following dispersion. The objective is to achieve maximum dispersion of the soil with minimal alterations of the SOM (Feller and Beare 1997). Various dispersion techniques exist, although sonication and shaking are most commonly used. Sonication involves the transmission of sound waves that create microscopic bubbles, which disperse soil particles. Standard protocols for sonication are non-existent, and sonication has the potential to redistribute organic matter among size and density fractions (Collins et al. 1997). Shaking is assumed to be a less intense method. However, Christensen (1992) noted that high-speed shakers may cause abrasion of particles, and simple shaking in water may offer an incomplete dispersion. The author indicated that chemically assisted dispersion can introduce unintended changes in SOM structure and distribution, while suggesting that the resin shake procedure calls for further investigation.

Sieving, sedimentation, and centrifugation are collectively used to determine particle size fractions (e.g., Nicolardot et al. 1992; Shulten et al. 1993). Wet and dry sieving produce similar results if dry weight and C recovery are compared (Christensen 1985). When textural class is determined in addition to size fractionation, the hydrometer (Robarge and Fernandez 1986) or pipette methods (Gee and Bauder 1986) are used. Oliveira (1997) suggested that gamma ray attenuation provides a more time efficient means to determine particle size.

The majority of SOM is associated with silt- and clay-sized fractions (Hinds and Lowe 1980; Anderson et al. 1981; Schnitzer and Ivarson 1982; Christensen 1992). Percentages of C and N are commonly 1.2 to 4 times higher in silt- and clay-sized fractions than in

whole soils (Catroux and Schnitzer 1987). Soil organic matter in the sand-sized fraction, consisting primarily of plant debris (Gregorich and Ellert 1993; Shulten et al. 1993), has a rapid turnover rate compared to silt- and clay-size fractions (Tiessen and Stuart 1983; Christensen 1987; 1992; Nicolardot et al. 1992). Fine-textured soils may physically and chemically protect SOM from decomposition within and among microaggregates (Tisdall and Oades 1982; Hassink 1994).

Upon investigation with ^{13}C NMR, Baldock et al. (1992) demonstrated that the relative intensity of O-alkyl C (carbohydrate structures) decreases while alkyl C increases during decomposition. Alkyl C increases with decreasing particle size (Oades et al. 1987; Baldock et al. 1992). The persistence of alkyl C in various soil types and climatic conditions indicates its recalcitrance, which may be a function of structure and close association with clay particles (Theng et al. 1986). Additionally, Baldock et al. (1992) noticed a loss of O-alkyl C as particle size decreased, suggesting that the extent of decomposition was greater in fine particles. Amelung et al. (1999) also found that the concentration of lignin-derived phenols significantly decreased with decreasing particle-size fractions. The authors similarly concluded that decomposition increases with decreasing particle size.

Others have indicated that fine clay fractions have faster turnover rates than silt fractions (Christensen 1987; Nicolardot 1992), noting that clay fractions are associated with microbial and metabolite materials (Christensen 1992; Nicolardot et al. 1992; Franzluebbers et al. 1997) and higher C mineralization rates (Christensen 1992). However, dispersion techniques may redistribute labile microorganisms and other compounds that may be adsorbed by fine clays (Christensen 1992; Collins et al. 1997). For example, the SOM dissolved in the particle-size separation procedure may account for 1% to 11% of whole SOM, and unless soluble SOM is isolated separately it will be deposited in the finest fraction (Christensen 1992).

Density and particle size

Density and particle fractionation schemes have been combined to further define SOM fractions. Densimetric evaluations of particle-size fractions are typically conducted on sand-sized separates (Christensen 1992). Zhang et al. (1988) investigated the amount of floatable macroorganic matter in sand-sized separates and, based on C/N ratios, concluded that macroorganic matter was distinct from SOM and more firmly attached to the sand. Tiessen and Stewart (1983) found that SOM that floated off the top of sand fractions was extremely

labile and accounted for a large part of the cultivation-induced loss of whole SOM. However, Christensen (1992) concluded that the LF of sand-sized separates behaves similarly to LF by density separation in respect to turnover and may represent identical SOM pools.

Recently, silica suspensions (Meijboom et al. 1995) have been used to partition sand-sized particles (macroorganic matter, defined as >0.150 mm) into three groups (Hassink 1996; Magid et al. 1996; Hassink et al. 1997). Carbon to N ratios decreased in the order of light, intermediate, heavy, and non-macroorganic matter (Hassink 1995). Residue quality had the strongest affect on the LF, while not signifi-cantly affecting the C/N ratios of non-macroorganic matter (Hassink 1995). Recovery of light and intermediate fractions were sensitive to C input; consequently, these fractions were considered "active" (Hassink et al. 1997). Conversely, Magid et al. (1995) surmised that active fractions may be distributed among particles with various sizes and densities.

Aggregates

Tisdall and Oades (1982) and Oades (1984) suggested that soil aggregation influences C turnover. Microaggregates (less than 0.25 mm) consist of clay and humified matter joined by polyvalent cations. Microaggregates may be bound together to form macroaggregates (0.25 to 2.0 mm). The binding material in macroaggregates may be more transient, consisting of microbial and plant-derived polysaccharides, roots, and hyphae. Thus, microaggregates may be more stable and resistant to decomposition than macroaggregates. However, the ma-terial within macroaggregates may have intermediate turnover times and represent a large portion of SOM lost due to cultivation (Elliott 1986; Elliott and Coleman 1988). Aggregates may be partitioned from SOM by sieving as outlined by Degens and Sparling (1996). Aggregates have also been sonically disrupted in density (Golchin et al. 1994a, 1994b) and particle size fractions (Borchers and Perry 1991).

Mineralization

Mineralization is the conversion of an element from an organic form to an inorganic form (Paul and Clark 1989). C mineralization is measured by the CO_2 emanating from metabolizing organisms (Zibilske 1994). Thus, CO_2 measurements are often used to determine bioavailable C in soils. Carbon dioxide may be determined by alkali absorption and direct analysis (gas-solid chromatography or infrared gas analyzer).

Zibilske (1994) reviewed field and laboratory methods to determine C mineralization. Laboratory incubations provide controlled environ-

ments and uniform samples, thus making interpretation and extrapolations to natural conditions extremely difficult (Collins et al. 1997). The use of assorted incubation periods makes it difficult to compare studies. Extended incubations (100 to 250 days) may adequately reflect management effects (Paul et al. 1995); however, Davidson et al. (1987) used a seven-day incubation period to determine active C turnover. Results from field experiments are more reflective of *in situ* mineralization rates (Zibilske 1994). Field measurements reflect the respiration of the microbial biomass and roots (e.g., Edwards and Harris 1977). Zibilske (1994) indicated that mineralization may not accurately represent an index of biodegradation because measurements may not account for incomplete oxidation of C, the loss of CO_2 by pathways other than volatilization, or the uptake of metabolic intermediates. In addition, mineralization does not include the microbial biomass, which is often included as a component the labile C pool.

Release rates can be used in mathematical models that estimate C pool sizes and their turnover rates (Collins et al. 1997). Soil respiration may change seasonally, reflecting temperature and moisture regimes. Moisture, C input, and root respiration determine soil respiration on longer time scales (Kutsch and Kappen 1997). The temperature sensitivity of a process may be expressed as a Q_{10}-value, which quantifies the increase of the process rate with a temperature increase (Kirschbaum 1995).

Biological Fractionation

Microbial biomass

Although the microbial biomass is relatively small and has a turnover time of one to two years, microbes represent a vital component of C cycling (Hu et al. 1997). The microbial biomass transforms and recycles SOM and also serves as a source (during mineralization) or sink (during immobilization) of labile nutrients. One to five percent of total organic C and N may be stored in the tissue of microorganisms (Jenkinson 1987). Bauhus and Khanna (1999) estimated that average forest floor concentrations of microbial C and N were 7,770 mg kg^{-1} and 749 mg kg^{-1}, respectively. The microbial biomass quickly responds to changes in soil induced by soil perturbations (Carter 1986) and to soil moisture and aeration status (Skopp et al. 1990; Duxbury and Nkambule 1994). Thus, microbial biomass C to total organic C (C_{mic}/C_{org}) ratios are used to monitor SOM dynamics and are thought to be more sensitive indicators of dynamics than total organic C (Sparling 1992). Others have implied that the microbial biomass represents a

poor indicator of SOM dynamics, because populations may be easily and quickly influenced by changing moisture regimes (Blair et al. 1995). Hu et al. (1997) recommended that carbohydrate C to organic C ratios (C_{carb}/C_{org}) and microbial C to organic C (C_{mic}/C_{org}) ratios be used concurrently to gain insight on C cycling. Their model provides a framework to establish relationships between substrate quality and microbial biomass.

Three common chemical techniques exist to extract microbial biomass. Two chloroform methods are based on the procedure outlined by Jenkinson and Powlson (1976). In the chloroform extraction method soil samples are first fumigated with chloroform. Length of fumigation may range from one to five days, and soils with high clay contents may require a five-day fumigation period (Horwath and Paul 1994). Then both non-fumigated and fumigated soils are treated with 0.5 M potassium sulfate (K_2SO_4) (Brookes et al. 1985). The suspension is shaken and filtered. Extracted C may be measured by wet oxidation with potassium dichromate, digestion with persulfate, or with an automated soluble C analyzer. Evolved carbon dioxide is trapped with 1 M NaOH and titrated with HCl. The C content in the non-fumigated sample is subtracted from the C content in the fumigated soil and divided by the proportion of microbial-C extract to determine the total microbial biomass.

Chloroform-treated soil may also be incubated for 10 to 20 days (Vance et al. 1987). The CO_2 evolved from treated and non-treated soils is then measured. Biomass is determined by subtracting the quantity of CO_2 evolved in the non-fumigated sample from the quantity of CO_2 evolved in the fumigated sample. The resulting number is then divided by the proportion of microbial-C mineralized to CO_2.

Substrate-induced respiration (SIR) involves adding readily available substrates to samples and measuring the CO_2 evolution (Sparling et al. 1990). The surge of CO_2 is used as an index to represent the microbial biomass. West and Sparling (1986) suggest the use of a glucose solution as the substrate to be mineralized. Samples are incubated for 2 to 3 hours and the CO_2 respired is then measured by an infrared gas analyzer or a gas chromatograph. The respiration rate is then converted to microbial C (Sparling et al. 1990).

Root biomass

Root turnover is a key component of C cycling within soil systems; however, root dynamics are poorly understood (Gholz et al. 1985; Aerts et al. 1989; Vogt et al. 1996). Fine roots, whose turnover rates are inversely proportional to size, are extremely hard to measure given their size and intimate connection with soil minerals and organic

particles (Gholz et al. 1985). However, roots and mycorrhizae may contribute more C to soils than litterfall inputs (Raich and Nadelhoffer 1989; Henfrick and Pregitzer 1993). Root residue inputs in temperate forest soils are estimated to account for 20% to 50% of the total C input (Vogt et al. 1996).

Root biomass may be estimated by core sampling (Aerts et al. 1989; Ruess et al. 1996). Root production and turnover estimates are commonly based on repeated sampling procedures. Dead and live roots are sorted and separated. The roots are then dried and weighed. Root turnover may be drastically overestimated because of the extreme heterogeneity of root distribution (Singh et al. 1984) or underestimated due to the losses that occur through exudation, root herbivores, and loss of root hairs (Persson 1979).

Root distribution and turnover may also be measured by minirhizotron observation tubes (Taylor 1987; Aerts et al. 1989, 1992; Fitter et al. 1997). Tubes are generally placed in the soil at 45° to the soil surface. The tubes are then covered and sampled at regular intervals. Using fiber optics, images are recorded in fixed positions in the tube on each sampling date. Root birth and death rates may be calculated per frame, and thus root turnover may be determined. To estimate root production per unit area, the root length production information must be calibrated by root biomass data obtained in soil samples (Aerts et al. 1989). This procedure is advantageous because non-destructive measurements of root turnover may be obtained (Fitter et al. 1997), and the technique allows repeated measurement of the same roots (Aerts et al. 1989).

Carbon Dating

Carbon 14

Carbon isotopes are often used to determine turnover rates of SOM (Ryan et al. 1995). Three C isotopes occur naturally: ^{12}C, ^{13}C, and ^{14}C. Both ^{12}C and ^{13}C are stable, while ^{14}C is radioactive. The half-life of ^{14}C, an estimated 5568 years, is used to determine radiocarbon age. The residual activity (Becquerels per gram C) of a sample with an unknown age is compared to present and modern background samples. It is assumed that constant ^{14}C activity in the atmosphere leads to constant ^{14}C in living organisms (Hsieh 1992). Thus, natural levels of ^{14}C in soils may be measured, although soil depth and soil management affect ^{14}C age estimates (Paul et al. 1997).

The peak of thermonuclear bomb testing occurred in the 1950s and early 1960s. Neutrons were released into the atmosphere from

aboveground testing sites, reacting with atmospheric N_2 to produce excess ^{14}C. Subsequently, ^{14}C was oxidized to $^{14}CO_2$ and assimilated by plants. Plant residues were then transformed to SOM. Hsieh (1993) developed a technique to identify the active fractions of soil organic C based on excess ^{14}C from the bomb testing. In order to estimate the mean residence time (MRT) of soil organic C pools in surface soils, the yearly variation of ^{14}C in the atmosphere is required. Hsieh assumed that ^{14}C from the bomb testing affected only the active portion of the soil organic C, while not affecting recalcitrant pools. This method is thought to predict the MRT of active soil pools ranging in age from 1 to 80 years. Using this technique, Hsieh (1993) calculated the active MRT of soil organic C from Sanborn field to be 34 years, while Balesdent et al. (1988) independently calculated the MRT of soil organic C from Sanborn field to be 15 to 22 years using ^{13}C tracers. Hsieh (1993) concluded that this method can be used to determine the MRT of active SOM, whereas the pool size and mean age of recalcitrant SOM can be determined by the procedure outlined by Hsieh (1992).

Acid hydrolysis (6 M HCl) in conjunction with ^{14}C dating may be an effective way to determine the old C fractions in temperate forests (Trumbore 1993; Trumbore et al. 1996), although Paul et al. (1997) suggest that the nonhydrolizable fraction may overestimate old soil fractions. While removing proteins, nucleic acids, and polysaccharides, acid hydrolysis does not solubilize lignin, phenols, and some celluloses (Schnitzer and Khan 1978). Martel and Paul (1974) explored this conundrum by treating the nonhydrolizable portion with NaOH. The NaOH extract was determined to be slightly older than the residue. Paul et al. (1997) concluded that acid hydrolysis provides a methodology to estimate the pool size and flux rates of resistant organic C rather than old C fractions.

Stable isotopes

The difference in assimilation of ^{13}C between Calvin cycle (C_3) and Hatch-Slack cycle (C_4) plants provides another method for assessing the stability of SOM. C_3 plants incorporate less ^{13}C into their biomass than do C_4 plants (Cambardella and Elliot 1992). Stable isotope abundances are expressed as $\delta\,^{13}C$ values, where:

$$\delta\,^{13}C = [(^{13}C/^{12}C \text{ sample} - {}^{13}C/^{12}C \text{ standard}) / {}^{13}C/^{12}C \text{ standard}] \times 10^3$$

The standard was originally a limestone fossil (*Belemnitella americana*) from the Cretaceous Pee Dee Formation of South Carolina (Boutton 1991). This standard is no longer available, although other standards calibrated against the original can be obtained from the

National Institute of Standards and Technology or the International Atomic Energy Agency.

The $\delta^{13}C$ values of C_3 plants may range from -35 to -20‰, whereas the $\delta^{13}C$ values of C_4 plants range from -19 to -9‰ (Boutton 1991). Smith and Epstein (1971) showed that the ranges are typically narrower, with the $\delta^{13}C$ values of C_3 plants -28 to -25‰, and C_4 plants generally -12‰. Because the $\delta^{13}C$ ranges of C_3 and C_4 plants do not overlap, differences in isotope ratios can be used to quantify the contribution of each photosynthetic pathway to SOM in mixed plant communities (e.g., Balesdent et al. 1988; Follett et al. 1997; Bernoux et al. 1998). As plant communities change between C_3 and C_4 vegetation, there is a subsequent change in the $\delta^{13}C$ value of SOM (Ryan et al. 1995). Natural ^{13}C abundance is used to estimate C turnover when converting grasslands into agricultural systems (Balesdent et al. 1988; Follett et al. 1997), and tropical rainforests into sugar cane plantations (Vitorello et al. 1989; Neill et al. 1996). The accumulation or loss of each vegetative type can be detected and expressed with first order decay kinetics, mean residence times, or in years.

Oxidizable carbon ratio

The oxidizable C ratio (OCR), established by Frink (1994 1995), offers an alternative or supplement to ^{14}C dating. Soil samples were removed from archaeological sites in New England containing charcoal with a known age. Charcoal within a soil undergoes biochemical changes overtime (Frink 1994). These changes were directly related to the age of the sample within the environmental contexts of climate, biota, relief, and the soil chemical and textural matrix. Total C for soil and charcoal samples was estimated by the loss-on-ignition procedure, and the readily oxidizable C was estimated by the Walkley-Black technique. The OCR is the ratio of total C to readily oxidizable C. The OCR is dependent upon rainfall, temperature, soil depth, soil pH, and soil texture. A formula was designed to determine the age of a C sample, accounting for O_2 permeability (soil texture and depth), mean rainfall, mean temperature, C concentration, and soil pH. The unidentified factors affecting the oxidizability of charcoal were subsumed within a calculated constant. Relief and aspect may also be added to the equation.

The calculated OCR dates were highly correlated ($r^2 = 0.98$) with ages obtained from ^{14}C dating (Frink 1995). The dating capabilities of OCR do not have age constraints as do typical ^{14}C dating procedures. However, OCR has produced unreliable results in poorly drained, anaerobic soils, suggesting that accurate OCR dates may be dependant upon oxidizing conditions (Frink 1995).

Analytical Techniques

Several technological advances have allowed the characterization of complex heterogeneous substances such as SOM. These techniques have provided insights on C turnover by addressing transformations of SOM during decomposition and humification processes. Moreover, analytical techniques may be used to determine the ability of chemical and physical approaches to fractionate C into meaningful pools.

Near infrared reflectance spectroscopy

Near infrared reflectance spectroscopy (NIR) uses reflectance signals from bending and stretching vibrations in bonds between C, N, H, and O to measure chemical compounds in organic materials (Wessman et al. 1988; Bolster et al. 1996). Constituents (e.g., lignin, cellulose, proteins) of organics have unique absorptive properties in the near infrared region (700 to 2500 nm) of the spectrum (Wessman et al. 1988). To correlate the spectral results at each individual wavelength, calibration is required. Wessman et al. (1988) and McLellan et al. (1990) presented calibrations for the foliage of woody plants. Multi-linear regressions are commonly used to develop equations that predict the chemistry of foliar samples, as determined by wet chemical analysis (McLellan et al. 1990; Bolster et al. 1996). Wessman et al. (1988) and McLellan et al., (1990) concluded that NIR is an effective method for the determination of lignin, cellulose, and C in decaying foliage, litter, and green leaves. The authors suggested that this technique provides for a rapid (2 to 3 minutes), non-destructive procedure as compared to lengthy wet chemical analysis.

Nuclear magnetic resonance

Nuclear magnetic resonance (NMR) is a useful analytical tool in soil science. Several reviews provide explanations and techniques applicable to soils (Wilson 1987; Preston 1996; Randall et al. 1997; Veeman 1997). Nuclear magnetic resonance is based on the fact that many atomic nuclei act as magnetic dipoles. The rotation of the charged nucleus produces a magnetic field. The magnitude or strength of the dipole is proportional to the spin angular momentum. A soil sample or extract is placed between two poles of a magnet and subjected to a magnetic field, which promotes a nuclear transition from a lower spin state to an upper spin state. An equilibrium is established between the nuclei moving into the higher spin state and those that are relaxing, which produces a resonance condition that is a detectable signal when either the strength of the applied magnetic field or the frequency is held constant while changing the other. The position of the resonance signal is referred to as a chemical shift and

is compared to a standard peak to allow for the determination of the chemical environment. Because the frequency at which the nuclei resonate is determined by the intrinsic properties of the molecules in which they are contained, NMR is used to distinguish between C nuclei in different intramolecular environments (Wilson 1987). For example, CH_3 and COOH can be identified in acetic acid (CH_3COOH), because the two C groups have different chemical environments and thus resonate at different frequencies.

To study SOM, ^1H, ^2H, ^{13}C, ^{15}N, and ^{31}P nuclei are commonly used (Randall et al. 1997). Both solid and liquid samples may be analyzed, and a review of the advantages and disadvantages of each is provided by Randall et al. (1997). Magic-angle-spinning (MAS) may be applied to solid materials, thus organic matter does not need to be extracted from soil samples (Hemminga and Buurman 1997). High polar dipolar-decoupling, cross polarization (CP), and MAS result in high-resolution spectra that provide detailed information about the chemical structure of SOM. However, CP/MAS is limited by low C contents and the presence of paramagnetic species (particularly Fe^{3+}) (Anderson 1995; Hemminga and Buurman 1997) such that quantitative reliability may be a concern (Preston 1996). Paramagnetic species (weakly magnetized when brought near a magnet and attracted into the magnetic field) cause a broadening of spectral lines and reduce the signal-to-noise ratio, which in effect makes it difficult to obtain a clear characterization of the sample. Hemminga and Buurman (1997) recommended that SOM from Fe-rich soils be extracted and purified. The authors suggested that Fe may possibly be removed by dithionite reduction and $SnCl_2$. Additionally, they suggested that clayey samples be pretreated, otherwise portions of organic material may be obscured. Dai and Johnson (1999) provide a review of techniques to remove paramagnetic species found in Spodosols. Preston (1996) indicated that the problems associated with CP/MAS are not insuperable, suggesting that for many studies comparing relative intensities across a series of similar samples may be adequate.

Baldock et al. (1992) used ^{13}C NMR and size fractions to outline a model of oxidative decomposition of plant materials in mineral soils. The model expresses decomposition as a continuum from fresh plant residues that exist in larger size fractions (>20 Fm) to partially degraded material in intermediate sized fractions (2 to 20 Fm), while the most humified material and microbial synthesized substances reside in the clay fraction. The authors proposed that during the initial stages of decay CO_2 evolution occurs while a portion of C is assimilated by microbial populations. This results in the decrease of the O-alkyl

signal, although microbial produced O-alkyl will also form. The next phase along the continuum is the degradation of lignin, causing a reduction in the aromatic signal. Finally, alkyl C remains in the fine fractions, which is thought to represent polymethylene structures. Baldock et al. (1992) suggested that the accumulation of alkyl C may be attributed to microbial synthesis and preservation by clays. Subsequently, several others have confirmed this general decomposition model in forest floors, litter, and peats (Beyer et al. 1993; de Montigney et al. 1993; Baldock and Preston 1995; Gressel et al. 1995). Studies have also demonstrated the importance of fire on SOM structure, such that heating SOM results in an increase in aromaticity at the expense of carboxyl and aliphatic structures (Almendros et al. 1992; Haumaier and Zech 1995).

Zech et al. (1997) used NMR to study chemical structural changes with depth in an undisturbed soil profile. Distinct horizons at various stages of humification allowed the authors to characterize SOM at progressive stages of decay. Zech et al. (1997) similarly found that alkyl C and carboxyl C signals increased with increasing decomposition, while O-alkyl decreased. Kögel-Knabner et al. (1992) used dipolar-dephasing (DD) to study the rigidity of alkyl chains in relation to depth of the forest floor. Dipolar dephasing is a variation of cross-polarization, whereby a delay (typically 40 to 100 Fs) is inserted in the process (Preston 1996). Both nonprotonated C and C species that undergo molecular motion in the solid state (acetate, methoxyl, and methyl) may be revealed. Dipolar dephasing eliminated the signal of rigid alkyls, leaving a signal for mobile portions that decreased with the humification process.

Other studies have used NMR to characterize fractionated C pools. Kögel-Knabner et al. (1991) and Kögel-Knabner (1993) used the ratio of more oxidized lignin monomers (phenolic acids) to aldehyde monomers released in the oxidation of CuO as an index of lignin alteration. CuO oxidation and ^{13}C NMR were used in conjunction to reveal the structural changes in lignin during decay in forest soils. They found a decrease of phenolic and methoxyl C with increasing degree of humification, accompanied by decreasing yields of lignin-derived CuO oxidation products. Guggenberger et al. (1995) used the same procedure on particle-sized fractions of soil (A horizon). In addition, characterization of C fractions derived from HCl fractions and from the Georing and Van Soest (1970) fractionation scheme has been described by Kögel-Knabner (1997).

Golchin et al. (1994a, 1994b, 1995) used ^{13}C NMR to characterize the light fraction protected within aggregates, providing insights pertaining to C quality and soil structure. Heavy and light fractions were initially separated. The heavy fraction was subsequently dispersed using ultrasonication, releasing an occluded light fraction. The "free" light fraction was dominated by polysaccharides (O-alkyl C compounds). The occluded light fraction, protected from leaching and existing in relatively anaerobic conditions, contained more alkyl C than the free light fraction. When passing from the free light fraction to the occluded light fraction, O-alkyl C decreased and alkyl C (polymethylene) increased. These differences were thought to be associated with the relative degree of decomposition within these fractions. Increased proportions of alkyl C in the occluded light fraction may result from the utilization of easily decomposable carbohydrates and selective preservation of recalcitrant alkyl C by soil microorganisms. The authors concluded that occluded C represents a relatively old C pool that is protected within aggregates, whereas the free light fraction C represents a less protected and more labile C pool.

Pyrolysis-field ionization mass spectrometry

Pyrolysis-field ionization mass spectrometry (Py-FIMS) allows for the determination of SOM on a molecular basis (e.g., Simmleit and Schulten 1989; Beyer et al. 1993; Schulten and Leinweber 1993; Leinweber and Shulten 1993, 1995). Py-FIMS involves heating a sample evenly in a pyrolysis chamber, which is attached to a field ionization (FI) mass spectrometer. The mass spectrometer measures the mass of the compounds that are released as the sample is thermally degraded. Computer-based calculations identify the most probable compounds based on the mass of the compound. Whole soil samples that contain at least 1.5% to 2% SOM may be analyzed by Py-FIMS (Schulten and Schnitzer 1992). Py-FIMS may be superior to CP/MAS ^{13}C NMR because the exact identification of compounds is possible; however, the structure of more complex humic portions released by pyrolysis from the smaller compounds can not be determined directly (Anderson 1995).

Simmleit and Schulten (1989) provide a review of Py-FIMS techniques as they apply to environmental research. They produced mass spectral characterization of healthy and damaged spruce needles. Py-FIMS has been employed to study the dynamics of litter decomposition and SOM humification (Hempfling et al. 1991; Beyer et al. 1993). Leinweber and Schulten (1995) used Py-FIMS to study the retention of SOM by Fe- and Al-oxides, and swelling clay minerals.

Shulten and Leinweber (1993) investigated particle-size fractions with Py-FIMS, and demonstrated strong differences between thermal energies required to extract SOM constituents from bonding sites exist. Higher energy was required to release lignin subunits, lipids, and alkylaromatics from clay than from silt. In addition, they found that materials that require a low energy of degradation in medium silts are lost or transferred to finer-sized fractions. Leinweber and Shulten (1995) compared C turnover models created with data from CP/MAS and Py-FIMS and suggested that advanced cross-linking of molecular structures and stronger bonding to mineral surfaces occurs in the finer size fractions.

Fourier transform infrared spectroscopy

Fourier transform infrared spectroscopy (FTIR) may be used to analyze soils qualitatively and semi-qualitatively (Gressel et al 1995; McColl and Gressel 1995). Infrared light is used to increase the stretching and bending vibrations of chemical bonds. Because each group resonates at different frequencies and absorbs infrared light at specific wavelengths, different functional groups may be identified. Diffuse reflectance infrared Fourier transform spectroscopy (DRIFT) and cylindrical internal reflectance (CIR) are two techniques that are being investigated to judge their usefulness in characterizing SOM (McColl and Gressel 1995). DRIFT requires little sample preparation for finely ground samples of soil and litter, whereas CIR is used to analyze SOM in solution.

SUMMARY

This review summarizes many of the currently available techniques used to measure the character of C in soils. These techniques offer a wide range of tools to better understand the nature of forest soil C and the consequences of ecosystem perturbations on long-term C sequestration. The challenge for practitioners will be to define common and consistent methodologies that are easy to employ operationally and functionally meaningful with respect to ecosystem C condition. New techniques being developed offer the promise of greater insight on the character of forest soil C and the rate of turnover of C fractions as methodologically defined. It is likely that no single tool will be ideal for both monitoring and research activities dealing with C sequestration in forest soils, and all new initiatives should be pursued in the context of those techniques and experiences from the scientific

record. We hope that this document contributes to that understanding.

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