

Organo-Mineral Complexes and Their Study by Radiocarbon Dating¹

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ABSTRACT

The objective of this study was to investigate the long-term cycling of organic C in cultivated soils by radiocarbon dating. Samples from a clayey Haploboroll soil (Indian Head) taken in 1963 and 1978 showed a general reduction in radiocarbon age of the whole soil of 20%. This was attributed to the enrichment of ¹⁴C in the atmosphere because of nuclear explosions in the 1960s, and its subsequent incorporation into organic matter. Relative decreases in age were least for nonhydrolyzable C and the aromatic HA-A, the oldest and least active fractions. The apparent age of the humin and clay-associated HA-B decreased much more, indicating their greater involvement in short-term cycling. A second clayey Haploboroll soil (Melfort) was studied by chemical fractionation and separation into size fractions after ultrasonic dispersion. The HA-A and coarse clay-associated organic C were oldest, with a much younger date for fine-clay-associated organic C. Fulvic acids purified by dialysis to eliminate materials less than 2000 molecular weight had an old date of 1140 ± 35 yr. Size fractionation of two other Boroll soils indicated young radiocarbon ages (radiocarbon enrichment) of fine clay-associated organic C. These findings are discussed in terms of the cycling of organic matter and nutrients. Particular attention is given to clay-humus interactions and their importance in protecting otherwise labile humic materials to provide a substantial pool of moderately available nutrients.

Additional Index Words: clay-humus complexes, organic matter, Mollisol.

Anderson, D. W., and E. A. Paul. 1984. Organo-mineral complexes and their study by radiocarbon dating. *Soil Sci. Soc. Am. J.* 48:298-301.

SOIL ORGANIC MATTER is a multicomponent system through which carbon and associated nutrients may cycle rather rapidly or be tied up in resistant forms for long periods of time. Knowledge of the more rapid turnover, with time spans of weeks to a few years, has benefited from studies utilizing isotopically labelled substrates (Paul and Van Veen, 1978; Ladd et al., 1981). Most of the organic matter, however, occurs in biologically resistant forms often in association with mineral colloids which, because of the sheer size of the pools involved, provide a substantial portion of the nutrients mineralized and made available to plants (Paul and Juma, 1981; McGill et al., 1981). Radiocarbon dating has been used to study the longer term turnover of organic matter (Martel and Paul, 1974a; Jenkinson and Rayner, 1977; Stout et al., 1981). There are, however, certain questions that require further elucidation. Massive enrichment of the atmosphere with ¹⁴C resulted from the detonation of thermonuclear devices (Stout et al., 1981). Its effect on the radiocarbon age of soil organic carbon and knowledge of the rate at which this pulse of ¹⁴C moves through different fractions can add to our knowledge of organic matter processes.

¹ Contribution from the Saskatchewan Inst. of Pedology, Univ. of Saskatchewan, Saskatoon, Sask. S7N 0W0 Journal Paper no. R331. Received 7 Mar. 1983. Approved 1 Nov. 1983.

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The significance of clay-humus complexes in both the short and long-term cycling of organic matter requires further assessment. One concept suggests that organic matter-mineral colloid interactions are the mechanism for the relative resistance of organic matter to microbiological degradation (O'Brien and Stout, 1978). A second hypothesis also emphasizes humus-clay interactions, but considers the resultant pool to be important to the medium-term cycling of organic matter (Jenkinson and Rayner, 1977; Anderson, 1979) and therefore controlling the rates of nutrients cycling in soil. These considerations, coupled with the relatively recent assessments of the importance of soil organic matter as a carbon sink on a global basis (Wilson, 1978), indicate the need for more information in these areas. This paper reports on the radiocarbon dating of several Mollisol soils from the Northern Great Plains. Comparisons of soils sampled in 1963 and 1978 measure the effect of bomb enrichment on different organic C fractions. The radiocarbon age of the organic C of size fractions gives information as to the significance of clay-humus complexes in organic carbon cycling.

MATERIALS AND METHODS

Soils

All soils studied were Ap horizons of cultivated soils of former grassland vegetation (Table 1). The Indian Head soil was from the Indian Head Experimental Farm, with two sampling dates studied. Two plots were sampled in 1963 and the soil stored air-dry in sealed polyethylene bags until 1978. The 1978 samples were obtained from the same plots sampled in 1963. The Melfort soil was obtained in 1977 from the same plots sampled in 1964 at the Melfort Research Station (Campbell et al., 1967). The Oxbow and Amulet soils were sampled in 1979 from cultivated fields in southeastern Saskatchewan to relate radiocarbon age of organic C in different size fractions to the previously documented properties of the organic C (Anderson et al., 1981).

Samples were processed and stored in laboratory buildings where ¹⁴C radiotracers had never been used, to minimize the possibility of contamination with radiotracer C. Samples were air-dried and ground to less than 2 mm. Larger roots and residue fragments were removed first. Finer plant residue was removed under a 20 × binocular microscope. Finally, smallest plant fragments were removed by flotation in 0.01N H₂SO₄ (Anderson et al., 1974).

Fractionation Methods

The alkali-pyrophosphate fractionation method (Anderson et al., 1974) involves a principal extraction with 0.1M NaOH-0.1M Na₄P₂O₇. The principal extract was separated from the soil by centrifugation at 1000 g. The sedimented soil residue then was dispersed in water with sonification, followed by high speed centrifugation (10 400 g) to yield a dark-colored humus-very fine clay suspension as the supernatant. The organic C still remaining with the sedimented soil was termed the humin fraction. Both the alkaline pyrophosphate extract and the humus-clay system were acidified to pH 1.5. This precipitated the humic acids—designated HA-A for the alkaline pyrophosphate extract, and HA-B for the humus-clay system. The fulvic acids remained in solution.

Table 1—Classification and properties of the soils.

Soil	Classification U.S./Canadian	Texture	Org. C mg g ⁻¹	Cultural history
Indian Head	Udic Haploborolls Orthic Black	Clay	(1963)-24.1 (1978)-22.8	Grain, legume-grass, fallow rotation
Melfort	Udic Haploborolls Orthic Black	Silty clay	(1963)-55.8 (1978)-54.3	Wheat-fallow rotation, straw returned
Oxbow	Udic Haploborolls Orthic Black	Loam	33.2	Cultivated, generally grain-fallow rotation
Amulet	Typic Argiborolls Orthic Dark Brown	Clay loam	17.1	Cultivated, generally grain-fallow rotation

The fulvic acid sample that was to be radiocarbon dated was concentrated by dialysis using a membrane (Spectraphor) which retained materials above approximately 2000 molecular weight. The low molecular weight materials were discarded.

The particle size fractionation followed ultrasonic dispersion of a 1:10 soil/water suspension using 300 W power for 8 min, with cooling of the sample in an ice-bath (Anderson et al., 1981). Sand (>50 μm) was separated by wet sieving, coarse silt (50–5 μm) by sedimentation and decantation, and fine silt (5–2 μm) and coarse clay (2–0.2 μm) by centrifugation. The fine clay (<0.2 μm) was flocculated from dilute suspension by adding CaCl_2 solution; water soluble C in the solution phase was discarded. The fractions were freeze-dried and ground prior to analyses and radiocarbon dating.

Chemical Methods

Organic C was determined by wet combustion (Nelson and Sommers, 1975). Acid hydrolysis treatments involved boiling samples in 0.5M HCl under reflux for 16 h, then in 6M HCl for 16 h.

Radiocarbon Dating

Radiocarbon-dating measurements of the Melfort and Indian Head samples were done in the Radiocarbon Laboratory of the Saskatchewan Research Council. Corrections for ¹³C were not done because past experience has indicated that these corrections are small, about 0 to 20 yr on the average (Martel and Paul, 1974a). The Oxbow and Amulet samples were analyzed by Geochron Laboratories, Cambridge, MA, and included ¹³C correction. The considerable amount of radiocarbon enrichment in many samples required expression of data as both radiocarbon age (years B.P.) and percent of modern (1950) ¹⁴C activity.

RESULTS

A comparison of the Indian Head soils sampled in 1963 and the same soils sampled in 1978 shows that the radiocarbon ages decreased about 20% over the 15 yr period (Table 2). This was attributed to a general enrichment of ¹⁴C in the atmosphere due to the detonation of nuclear devices (bomb effect). The ¹⁴C content of plant material peaked at about 68% above the radiocarbon pre-bomb levels in 1964 (Rafter, 1965). This decrease in age compares with a 50% reduction reported for soils at Rothamsted (Jenkinson and Rayner, 1977). It is likely that the bomb effect had already decreased the radiocarbon age of the Indian Head samples somewhat by 1963.

The nonhydrolyzable organic C had the oldest radiocarbon age in the 1963 sample; the relative decrease in age, or degree of enrichment, in the 1978 samples was least. The resistant nature or biological immobility of this fraction is consistent with earlier work (Campbell et al., 1967; Martel and Paul, 1974a).

Table 2—Radiocarbon age of Indian Head soils sampled in 1963 and 1978.

Fraction	Proportion of organic C†	1963		1978		1963–1978 change
		% modern	Equivalent age yr	% modern	Equivalent age yr	
Soil	1.0	78.7	1910 ± 45	82.8	1515 ± 40	–21
Nonhydrolyzable organic carbon	nd	70.4	2820 ± 45	77.0	2095 ± 40	–26
HA-A	0.21	73.6	2455 ± 45	80.7	1720 ± 45	–30
HA-B	0.30	83.9	1325 ± 45	92.9	590 ± 40	–55
Humin	0.33	73.3	2495 ± 50	86.8	1140 ± 45	–54
Soil‡	1.0	82.7	1520 ± 40	85.5	1255 ± 65	–17

† Fulvic acids (not dated) accounted for 0.17 of the carbon. 1963 and 1978 distribution was similar.

‡ A second pair of samples from an adjacent plot.

Table 3—Radiocarbon age of Melfort soil and fractions.

Fraction	Proportion of organic C	Age, % modern	Equivalent age, yr
Soil	1.00	90.6	795 ± 55
Chemical fractionation†			
HA-A	0.32	80.5	1425 ± 95
HA-B	0.21	91.6	705 ± 70
Fulvic acids	0.09	86.8	1140 ± 55
Size fractionation‡			
Coarse silt	0.25	90.6	800 ± 50
Fine silt	0.29	88.7	965 ± 45
Coarse clay	0.31	85.5	1255 ± 60
Fine clay	0.08	97.9	170 ± 50

† Not dated: Humin (0.26) and fulvic acids < 2000 molecular weight (0.18).

‡ Not dated: Organic carbon with sand (0.03).

The alkali-pyrophosphate extractable HA-A had a comparatively old radiocarbon age, and the reduction in age in the 1963–78 period was similar to the nonhydrolyzable organic matter. In comparison, the clay-adsorbed HA-B had a radiocarbon age of 1325 ± 45 yr, decreasing substantially to 590 ± 40 yr by 1978. This indicates the faster rates of turnover and greater involvement of these fine clay-associated materials in the cycling of organic C. The humin fraction, with a mean age of 2495 ± 50 yr in 1963, decreased to 1140 ± 45 yr. This combination of considerable age coupled with a substantial decrease in age due to the bomb enrichment is evidence for the variable nature of the humin or non-extractable organic C. Humin consists of chemically resistant, aromatic humic materials, and clay-adsorbed organic carbon much like the HA-B (Anderson et al., 1974), which has young organic C in it. It also contains nonextractable recent plant residues which were not removed by flotation. The lesser amounts of ¹⁴C derived from recent plant material in the humic acid and nonhydrolyzable fractions, and its concentration in the humin are consistent with findings for a cultivated Gleysolic soil in Eastern Canada (Martel and Lasalle, 1977).

The Melfort soil was studied using both chemical and size fractionation methods (Table 3). The HA-A, with a radiocarbon age of 1425 ± 95 years, was the oldest fraction observed, consistent with its strongly aromatic nature. The fine clay-adsorbed HA-B had a radiocarbon age of 705 ± 70 yr, consistent with its less aromatic, more nutrient-rich or less humified characteristics (Anderson et al., 1974). The radiocarbon age of 1140 ± 55 yr for the fulvic acids is of

Table 4—Radiocarbon age of size fractions of Oxbow and Amulet soils.

Soil	Fraction	% modern	Equivalent age, yr
Oxbow	Whole soil	94.2	480 ± 115
	Coarse silt	92.3	645 ± 115
	Fine silt	94.0	495 ± 125
	Coarse clay	97.5	200 ± 125
	Fine clay	102.2	Modern
Amulet	Whole soil	105.4	Modern
	Coarse silt	105.4	Modern
	Fine silt	101.1	Modern
	Coarse clay	101.4	Modern
	Fine clay	106.2	Modern

particular significance. This fraction, retained in a dialysis chamber of 2000 molecular weight cutoff, accounted for 32% of the fulvic acids and 9% of the soil organic C. In many soils the dates for fulvic acids are modern; in this particular soil the whole fulvic acid fraction was 525 ± 55 yr in 1964 (Campbell et al., 1967) when the whole soil dated 870 ± 50 as compared to 795 ± 55 in 1978. This older mean age of 1140 yr for the higher molecular weight fulvic acids, which are more like the humic acids, indicates the continuous nature of the spectrum of humic materials, and the arbitrary nature of the humic-fulvic split once the low molecular weight materials have been removed. It also suggests that much of the higher molecular weight fulvic acids may represent strongly humified materials that were formerly a part of the humic acids; however, because of progressive decreases in molecular weight and increases in aromaticity with advancing humification (Swift et al., 1970), these materials may now be part of the fulvic acid fraction.

Size fractionation after ultrasonic dispersion and subsequent dating of the organic C yielded a maximum age of 1255 ± 60 yr for the coarse clay (0.2 to 2 μm). Segregation of the fine clay-associated organic C yielded a much younger radiocarbon age (170 ± 50 yr), indicating that this organic C was considerably more active than that associated with the coarse clay. The coarse silt was dated at 800 ± 50 yr and the fine silt at 965 ± 45 yr. Characteristically, the organic C of silt fractions consists of partially to strongly humified particulate organic residues (Tiessen and Stewart, 1983), charcoal fragments and, as observed in a similar soil, microaggregates not disrupted by the ultrasonic treatment (Turchenek and Oades, 1979).

The size fractionation method was also applied to analysis of the Oxbow and Amulet soils (Table 4). The fine clay-organic C was the most enriched in ^{14}C . Older or less active material was associated with the fine silt and coarse clay in the Amulet soil, and with the silt in the Oxbow soil.

DISCUSSION

The marked differences between the fine clay-associated organic C and that associated with the coarse clay and fine silt shows a discontinuity between particle size and soil organic matter stability. The organic C of the coarse clay-fine silt was consistently the oldest fraction, concurring with the predominance of strongly aromatic humic acids in these sizes, and the higher degree of resistance of the humic material to acid hydrolysis (Anderson et al., 1981). Electron micrographs

of the organic materials concentrated in the coarse clay fractions of several soils showed that humified microbial cell walls were a major component (Turchenek and Oades, 1979). Strongly aromatic humic acids were dominant in alkali extractions of those fractions. McGill and Paul (1976) reported that microbial cell wall materials accumulated in the coarse clay fraction. Work with model systems demonstrated that reactions between microbial cell wall components and phenolic polymers resulted in the formation of biologically resistant humic materials (Nelson et al., 1979).

Microbial cell walls are more resistant to decomposition than cytoplasmic materials (Kassim et al., 1981), and linkages between cell wall components and the phenolic polymers of the existing humus or adsorption to clay could form complex structures that are even more recalcitrant. These observations suggest that the original resistance of the organic materials, complexing with phenolic polymers to form still more resistant moieties, increased polycondensation of aromatic components, and clay protection all may be involved in maintaining humic materials in soils for thousands of years.

The organic materials associated with the fine clay are mostly fulvic acids and high molecular weight, largely aliphatic humic acids that are rich in nutrients such as N and S (Anderson et al., 1981). In a study of a grassland soil that had been cultivated for 60 yr a proportionately greater depletion of fine clay associated organic matter than other fractions indicated its importance to nutrient supply (Tiessen and Stewart, 1983). Work with labelled substrates showed that products of microbial synthesis, presumably cytoplasmic materials or metabolites, were concentrated in fine clay fractions (McGill and Paul, 1976). Stabilization of amino acids by adsorption to clay has been demonstrated (Sorenson, 1967). Later work (Sorenson, 1972) showed that the addition of montmorillonite did not influence the rate of decomposition of ^{14}C -labelled carbohydrates but increased the stability of the products thus formed during decomposition. This was attributed to the formation of biostable complexes and aggregates with clay.

Examination of ultra-thin sections of soils showed the coating of carbohydrates on clay platelets and in crevices of submicron size. This explains, in part, why some carbohydrates are resistant to degradation, and why small quantities of microbial polysaccharides are able to stabilize clay aggregates (Foster, 1981).

The high degree of radiocarbon enrichment of the Amulet soil, 105.4% of modern, is difficult to explain. All size fractions were enriched and had a pattern of relative ages similar to the Oxbow and Melfort soils. Contamination in the laboratory or a recent, massive addition of enriched organic residues do not appear to be the cause. The organic C content of the soil was normal for this region. This cultivated, former semi-arid grassland soil may be enriched in bomb C because of fast rates of decomposition of native organic C under the enhanced temperature and moisture conditions of alternate fallow cultivation. Coupled with this there may have been higher than normal additions of crop residues enriched in bomb C over the past two

decades, resulting in the age distribution and degree of enrichment observed.

It is difficult to relate radiocarbon age from one soil to another, both within soil catenae and across soil zones. Young or modern radiocarbon ages have been reported for the organic C of Oxbow soils sampled in different areas and for clayey Brown soils (Martel and Paul, 1974b). The surface horizons of similar soils in nearby areas had much older radiocarbon ages. Lower slope soils have often dated modern, indicating higher than usual additions of ^{14}C in recent years. Recent work with another bomb-produced radionuclide showed that ^{137}Cs was concentrated in the light, partially decomposed, recent organic material in soil, which was preferentially moved by soil erosion (H. Tiessen, personal communication, 1982). Similar processes may have concentrated ^{14}C in different landscape positions.

Substantial amounts of stable, silt-sized aggregates are characteristic of grassland soils (Turchenek and Oades, 1979). It is likely that fine silt, coarse clay and fine clay occur in the same aggregate units. The associated organic C, including that complexed with the fine clay, can be expected to be of similar age. The relatively young radiocarbon ages of the fine clay-associated organic C observed in this study suggest that the fine clay acting most effectively as loci for short-to medium-term protection of labile humic materials occurs as individual particles, on aggregate surfaces or in crevices in aggregates. Increasingly more effective disruption of aggregates and release of fine clay-associated organic C complexed within aggregates can be expected to decrease the differences in relative age of the fine silt-coarse clay and fine clay-associated organic C.

Clay protection of organic materials by means of organo-mineral complexing appears to be an important process in soils: (i) maintaining nutrient supply by stabilizing a large pool of nutrient-rich, labile materials, (ii) slowing the progressive disposition of nutrients into the strongly aromatic forms and their virtual removal from active cycling, and (iii) probably increasing the efficiency of substrate utilization by microorganisms. Thus, clay protection plays its major role in reacting with the otherwise labile amino acid and carbohydrate metabolites resulting in turnover times of months, years or decades rather than days. The net effect of this process is to dampen fluctuations in available nutrient levels, smoothing out shortfalls when residue additions are small or very deficient in nutrients, and tying up labile microbial products when activity is high and mineralized nutrients and C are subject to loss by leaching, luxuriant consumption by plants or gaseous evolution into the atmosphere.

ACKNOWLEDGMENT

The authors would like to acknowledge the scientists of the Research Branch, Agriculture Canada, for providing the long-term plots for sampling, particularly E.D. Spratt and R.N. McIvor, who obtained and stored the 1963 samples.

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