# The Use of Radiocarbon Dating of Organic Matter in the Study of Soil Genesis<sup>1</sup>

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#### **ABSTRACT**

Radiocarbon dating was used to study the nature and the stability of the organic matter in a range of Chernozemic soils (Borolls) from Saskatchewan. Two catenas, one cultivated, the other not, were studied in detail; the bottom soils of the uncultivated catena contained buried horizons.

From the crest of the knoll to the depression, the mean residence time of the surface horizon of the cultivated catena decreased from 545 years before present (B.P.) to modern, the organic matter content increased and the percentage of hydrolyzable carbon decreased. The content of organic matter decreased but the mean residence time increased by 700 to 4,000 years in the B horizons. Two buried horizons located at different depths underneath the lower members of the uncultivated catena showed mean residence times of 5,950 and 8,410 years B.P. indicating that mass movement of soil had periodically occurred since the last glaciation.

The <sup>14</sup>C content of a field soil, measured by radiocarbon dating, was compared with the <sup>14</sup>C distribution in fractions of another sample of the same soil after amendment with <sup>14</sup>C-acetate and 350 days incubation in the laboratory. The artificially labelled soil accumulated more of the tracer in the acid hydrolyzable fractions than was found by radiocarbon dating the field soil. In turn, the distribution of soil nitrogen was more closely associated with the <sup>14</sup>C than with the unlabelled carbon of the soil.

Additional Index Words: humus, catena, mean residence time.

R ADIOCARBON DATING, utilizing naturally occurring <sup>14</sup>C, has been used in soil work since 1950, and measurements on buried soil as well as an organic relics found adjacent to the solum have been studied relative to the chronologic problems of soil genesis (4). The organic matter of most surface soils has been found to be sufficiently old to

yield usable radiocarbon measurements (6, 12, 17). Most soil profiles show increasing radiocarbon dates with depth (18), and radiocarbon dating of soil organic matter extracts has provided information on the time sequence of their formation (2, 12, 18). Although this technique has been the subject of recent reviews (15, 16, 19), data are needed to relate radiocarbon dates with information on pedogenic factors such as those responsible for the formation of a catena.

Tracer techniques utilizing <sup>14</sup>C have made possible the study of the relative stability of different humic fractions of soil organic matter labelled with <sup>14</sup>C during incubation experiments. Paul (11) assembled data from a number of incubation experiments with <sup>14</sup>C-labelled materials and concluded that the traditional fractions (humic acid, fulvic acid, and humin) showed a rather uniform distribution of the labelled carbon. Work with acid hydrolysis has been more successful in obtaining fractions of different specific activities (13). Radiocarbon dating gives an opportunity to study the distribution of naturally occurring <sup>14</sup>C in soils and to compare it with the results of experiments where labelled <sup>14</sup>C-material was added to soil incubated in the laboratory.

The study was undertaken: (i) to investigate the variation in the stability and the characteristics of the soil organic matter as affected by the position on the slopes of a catena, by the depth in the profiles and by the burial of old soils and (ii) to investigate the distribution of naturally occurring <sup>14</sup>C in the fractions of soil organic matter in comparison with the distribution of <sup>14</sup>C-labelled material added to the soil in the laboratory. This made it possible to test the hypothesis that successive hydrolysis of the soil organic matter provides meaningful information about its stability in nature.

### MATERIALS AND METHODS

#### Characterization of Soils

The soils at Site no. 1 were cultivated and located at Waldheim in the upper part of the Black Chernozemic (Udic Boroll) soil zone. The uncultivated soils at Site no. 2 were located in the same soil zone at Quinton, 219 km (130 miles) southeast of the first site. The soils of both sites contain about 25% clay and have been derived from the same calcareous glacial till and

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under the same climate. The two catenerary sequences, classified as Oxbow, have the sequence of Rego, Orthic, and Eluviated Chernozemic profiles and a Gleysolic profile.

On the upper slope, the shallow development of the soil is recognized as a Rego Chernozemic (Udorthentic Haploroll) profile. The better developed soil on the intermediate slope was described as an Orthic Chernozemic (Udic Haploboroll) profile. On the lower slopes, the effect of the runoff water leads to an Eluviated Chernozemic (Udic Argiboroll) profile. In the lowest topographic position, excess water accumulates and the wet and poorly drained soil is classified as an Eluviated Gleysol (Argic Cryaquoll). A Humic Eluviated Gleysol (Argic Cryaquoll) occurs where excess water remains in the profile all year preventing the decomposition of the organic matter and a Low Humic Eluviated Gleysol (Argic Cyraquoll) occurs where the water retreats occasionally, allowing some decomposition of organic matter to take place.

The soil of Site no. 3 was cultivated and located at Matador in the Brown Chernozemic (Aridic Boroll) zone; it contains about 70% clay and has developed on calcareous lacustrine deposits.

A bulk sample from each selected horizon (Table 1) was used for analysis. Identifiable plant materials in the soil samples were removed by repeated flotations with 0.01N HCl and handpicking under 20× magnification of a stereomicroscope. Soil carbonates were destroyed with 1N HCl. Organic carbon was determined by a dry-combustion method (22) using a 50% KI solution to absorb the HCl present in soil fractions. Total nitrogen was determined by the Kjeldahl method (1). Amounts of soil varying between 50 and 800g were necessary to obtain 3g of C for radiocarbon dating.

### Fractionation of Soil Organic Matter

The light materials were obtained after a densimetric fractionation (10) using ZnBr<sub>2</sub> with a density of 2.0g/cm<sup>3</sup> (Fig. 1). A portion of the organic matter remaining after flotation was extracted by successive 0.5N and 6N HCl hydrolysis (13). The NaOH extraction, after acid hydrolyses, was basically the method of Tyurin (7). The residue of the NaOH extraction was treated with water and yielded a water extract and a residue called humin.

## **Radiocarbon Dating**

The mean residence time (MRT) and the  $\delta^{14}C$  were calculated by using the equations

$$MRT = 18,500 \log_{10} (Ao/A)$$
 [1]

and

$$\delta^{14}C = [(A - Ao)/Ao] \times 1,000$$
 [2]

where Ao, the activity of the modern standard, was based on 95% of the activity of NBS oxalic acid, and A was the activity of the unknown sample (8). The mean residence time was expressed as years before present (B.P.) i.e. before 1950, and the  $\delta^{14}$ C was expressed in per mil (°/00). The value of 5,570 years was utilized as the half life of  $^{14}$ C.

# Discrimination Against 14C

The discrimination against  $^{14}$ C during photosynthesis and the subsequent biological transformations was determined by measuring the  $\delta^{13}$ C value of samples relative to the NBS Solenhofen standard no. 20 such that

$$\delta^{13}C = \frac{{}^{13}C/{}^{12}C \text{ sample} - {}^{13}C/{}^{12}C \text{ standard}}{{}^{13}C/{}^{12}C \text{ standard}} \times 1,000 [3]$$

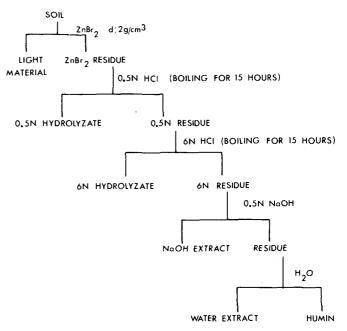


Fig. 1—Fractionation scheme utilizing acid hydrolysis.

The  $\delta^{13}$ C value was expressed in per mil (°/oo) and the mass spectrometer employed was an Atlas GD 150 (9).

## **Incubation Experiment**

Sörensen and Paul (21) studied, in the laboratory, the transformation of acetate  $^{14}$ C (100  $\mu$ c/g) into carbohydrate and amino acid metabolites during decomposition. They used a 0 to 15 cm depth sample of virgin Chernozemic soil (Aridic Boroll) and removed most of the roots by passing the soil through a 2 mm sieve. Incubation was continued for 350 days after which period a subsample containing 2.1% carbon was fractioned. The  $^{14}$ C present in the fractions was absorbed in NaOH after dry combustion. It was then counted in a scintillation counter (23).

# RESULTS AND DISCUSSION

The radiocarbon dating of the cultivated Black Chernozemic Ap horizons (Site 1) showed that the slope position of the organic matter in a catena affects the mean residence time (Table 1). The dating of the Ap horizon on the upper slope yeilded the oldest surface mean residence time of 575 years B.P. The mean residence times decreased on the slope to 270 years B.P. and 216 years B.P. In the depression, where the runoff water and its debris accumulated and where wet conditions were present, the mean residence time was modern. The decrease of the mean residence time from the intermediate slope toward the depression is associated with an increase in the amount of organic matter in the profile and a decrease in the percentage of hydrolyzable carbon. This shows that the amount, the nature, and the stability of the organic matter are affected by the position on the slope of the catena.

The mean residence times of two Ah horizons of the virgin Black Chernozemic soils (Site 2) were modern. These modern values may be attributed to the presence of a great amount of young material in the soil. The 0.01N HCl flotation of the soil removed most of the undecom-

Table 1-Mean residence times and char	acteristics of the organic matt	er sampled on Site 1 and Site 2
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Profile and							Hydrolyzable	
and horizons	Depth	MRT	∂ <sup>14</sup> C	∂ <sup>13</sup> C	Carbon	Nitrogen	C	N N
	em	(yr B, P, ±1σ)	(0/00±1σ)	(0/00)	9	% — <del>—</del>	% of	total
	8	Site 1: Oxbow Caten	a from Waldhei	m				
Rego Black Chernozemic	-			_				
Ap _	0-10	575 ±80	-69 ±13		2. 4	0.19	71	86
Orthic Black Chernozemic								
Ap	0-15	270 ±45	-33 ±10	-23.9	2. 1	0. 20	61	86
Eluviated Black Chernozemic								
Ap	0-15	216 ±45	-27 ±11	-24.0	2.7	0. 26	59	83
Bt	22-52	930 ±55	-109 ±11	-28.1	0.42	0.06	72	83
Low Humic Eluviated Gleysolic								
Ap	0-18	Modern	-4 ±13	-26.8	2, 4	0. 22	51	78
Bt	22-35	700 ±55	-83 ±11	-26.8	0.52	0.08	79	84
Btg <sub>3</sub>	60-70	4,870 ±60	-455 ±11		0.45	0. 07	33	58
		Site 2: Oxbow Cate	na from Quintor	1		•		
Rego Black Chernozemic	•			-				
Ah	0-8				5. 7	0.50	54	79
Orthic Black Chernozemic								
Ah	0-8	Modern	+8 ±10	-24.0	6.0	0.53	46	78
Bt	18-32	1,340 ±100	-154 ±15	-23.8	0, 68	0.08	70	87
Eluviated Black Chernozemic								
Ah	0-10				6, 1	0. 52	50	76
IIA-Cb	133-143	_8,410 ±115	-649 ±15		0,69	0, 08		
Humic Eluviated Gleysolic		•						
Ah	0-10	78 ±97	-10 ±16	-27, 8	6.4	0. 67	36	58
Bt	34-55	3,990 ±110	-391 ±15	-24.5	0,66	0.08	47	54
Ahb	86-96	5,950 ±140	-523 ±19	-24.7	0. 55	0.06		
Bgb	96-132				0.46			

posed materials, but visual observation under the 20× stereomicroscope showed the presence of black particles likely comprised of partially decomposed materials that were not removed by the flotation. This partially decomposed material lying on the surface of the soil is not in close contact with the microorganisms, and it may remain in that form until it is washed down the profile by rain water or mechanically buried by soil animals. This material would lower the mean residence of the total soil organic matter from values already low (270 years in the case of the orthic profile of Site 1) to values lower than 100 years B.P. and therefore too young for radiocarbon dating. The effect of undecomposed materials is especially strong because of the high levels of bomb-produced 14C in growing plants and recent residues. On Site 1 plowing likely accelerated the rate of decomposition of the newly added materials by mechanically bringing the organic matter of the surface soil in contact with the bulk of the microorganisms. For this reason. an Ah horizon would normally date younger than a corresponding Ap horizon.

The nature of the stability of the organic matter varied with the increasing depth. The mean residence times of the B horizons were all over 700 years B.P. The percentage of nonhydrolyzable carbon was smaller than in the A horizons with little change in the hydrolyzability of the nitrogen. This suggests, in agreement with the soil genesis concepts for those soils, that the nonhydrolyzable materials of the B horizons likely were formed in situ rather than being the result of a movement of the nonhydrolyzable material from the A horizons. The mean residence times of the organic matter present in the four Bt and the Btg<sub>3</sub> horizons of Table I were correlated with the percentage of nonhydrolyzable carbon (r = 0.99\*\*) and of the nonhydrolyzable nitrogen (r = 0.95\*\*). Although these calculations are based only on five results, the high correlation values suggest that the chemical nature of the nonhydrolyzable material contributes to increased stability of the soil organic matter in these lower horizons.

Two prominent darker layers occurred at the 86 to 96 cm depth of the Gleysolic profile and at the 133 to 143 cm depth of the Eluviated Chernozemic profile of Site 2 (Table 1). The material found at 86 cm dated 5,950 years B.P. and at 133 cm dated 8,410 years B.P. The high mean residence time obtained from radiocarbon dating, therefore, suggest the presence of two old buried horizons. This, also, is supported by the relatively high carbon content 0.69% of the material found at 133 cm. The mean residence time of a buried soil is a minimum estimation of the time since burial because the soil may be greater than zero at the time of burial and/or decomposition of recently added materials may continue after burial as long as the moisture and aeration conditions are not severely affected. One may, therefore, speculate that 8,410 years, the mean residence time of the deepest buried horizon, is the minimum time elapsed since an old land surface was covered by a layer of sediments presumably coming from the upper slope of that time. The profile characterized by an Ahb and a Bgb was buried at least 5,950 years ago by material forming today's surface.

The organic matter of the Ap 0 to 10 cm depth of the Brown Chernozemic soil (Site 3) which contained 1.9% of carbon and 0.20% of nitrogen was fractioned (Table 2). Each fraction was radiocarbon dated except the light material which was too young for proper counting and the 0.5N and the 6N hydrolyzates which demanded too long a manipulation for accumulating the necessary 3g of carbon. However, Paul et al. (12) showed that the mean residence time of such fractions could be calculated by difference, if the carbon content and the activity of the 14C of the other fractions and of the total soil are known. By assembling the fractions in three groups comprised of (i) total soil, light material, and ZnBr<sub>2</sub> residue, (ii) total soil, 0.5N hydrolyzate, and 0.5N residue, and (iii) total soil, 6N hydrolyzate, and 6N residue, one can calculate the unknown <sup>14</sup>C activity (<sup>14</sup>C) of the nonradiocarbon dated fractions by using the equation

Table 2-Mean residence times and δ14C of the fractions of the Brown Chernozemic soil (Site 3) and distribution of total nitrogen, organic carbon and <sup>14</sup>C in the fractions as it occurs in nature and 350 days after the addition of labeled <sup>14</sup>C-acetate

		Radiocarbon dated soil Ap 0-10 cm*							Incubated soil Ah 0-15 cm†		
	MRT	∂14 C	N	14 C	Soil-C	14 C/Soil-C	14 C	Soil-C	14 C/Soil-C		
	yr B. P. ±1σ	0/00 ±1σ		- % of total -			— % of	total —			
Total soil	350 ±65	-43 ±9	100	100	100	1.0	100	100	1,0		
Light material	Modern	+243 ±18‡	3.0	8.0	6.0	0, 3	5. 0	5.0	1.0		
ZnBr <sub>2</sub> residue	505 ±105	-61 *16	97	97	94	0, 98	95	95	1.0		
0. 5N hrdrolyzate‡	Modern	+7 ±15	41	37	35	1.1	49	35	1.4.		
0. 5N residue	855 ±70	-101 ±15	56	55	59	0. 93	46	60	0.78		
6N hydrolyzate‡	Modern	+61 ±22	43	24	22	1. 2	21	20	1.1		
6N residue	1,765 ±65	-197 ±16	13	31	37	0.84	25	40	0.63		
NaOH extract	1,910 ±105	-212 ±16	9, 0	22	27	0. 82	16	23	0.70		
Water extract	1,790 ±120	-200 ±15	1, 5	2, 6	3, 1	0.84	3.4	7.0	0.48		
Humin	1,330 ±100	-153 ±17	2, 5	6.4	6.9	0, 93	5. 6	10.0	0.56		

<sup>\*</sup> Carbon content 1.9%; nitrogen content 0.20%.

t Data were calculated by difference

<sup>14</sup>C (unknown fraction)

$$= \frac{{}^{14}\text{C (soil)} - [{}^{14}\text{C} \times \% \text{ of total C (known fraction)}]}{\% \text{ of total C (unknown fraction)}} [4]$$

From this <sup>14</sup>C, the mean residence time and the  $\delta$ <sup>14</sup>C of the light material, the 0.5N, and of the 6N hydrolyzates may be obtained by using Eq. [1] and [2].

The mean residence time was 350 years B.P. for the total soil but it varied from modern to 1,910 years B.P. for the fractions (Table 2). The youngest fraction of the soil was the light material with a  $\delta^{14}$ C of +243; its nature was not fully characterized but from the C/N ratio of 17 and a visual observation under a microscope, it appeared to be comprised of partly decomposed plant materials. This fraction enriched with <sup>14</sup>C, coming from nuclear tests that have taken place since 1950, showed that contamination of the soil organic matter with nuclear 14C existed even though 6% of the original carbon of the soil had been previously removed by the flotation of the original soil sample in 0.01N HCl. This contamination is important since 6% of carbon with  $\delta^{14}$ C of  $+243^{\circ}$ /oo would have the same effect on the mean residence time as 16% of carbon coming from prebomb plant material.

The  $\delta^{14}$ C values of the modern 0.5N and 6N HCl hydrolyzates were low, +7°/oo and +61°/oo respectively, but their amount, totaling 57% of the total carbon, contributed to lower the mean residence time of the 6N hydrolysis residue by 1,260 years—from 1,765 years for the 6N residue to 505 years for the ZnBr<sub>2</sub> residue. In turn, the mean residence times of the nonhydrolyzable fractions were high and ranged between 1,330 and 1,910 years B.P. This suggests that the 0.5N and 6N HCl hydrolyses can separate the young and/or the older materials in soil.

The <sup>14</sup>C content of a field soil from Site 3, measured by radiocarbon dating, was compared with the 14C distribution in fractions of another sample of the same soil after amendment with <sup>14</sup>C-acetate and 350 days incubation in the laboratory (Table 2). The laboratory labelled soil accumulated 70% of the <sup>14</sup>C-substances in the hydrolyzable fractions compared with 61% for the radiocarbon dated soil. This difference in the 14C distribution came from the substances dissolved by the 0.5N HCl hydrolysis which extracted 12% more 14C from the incubated soil.

The correlation between the <sup>14</sup>C and the soil-C within each experiment and between the two experiments are all significant, showing a similarity in the distribution of the natural 14C, the artificial 14C and the soil-C in the different fractions (Table 3). However, the δ<sup>14</sup>C values did not correlate as closely (r = 0.66) with the <sup>14</sup>C/soil-C ratios in the incubated soil as in the radiocarbon dated soil (r =0.98\*\*). This indicates that the <sup>14</sup>C/soil-C ratios of the incubated soil can not be used to determine the relative stability of each fraction. This lack of correlation likely originated from the different forms of 14C materials added to the soil; the <sup>14</sup>C-acetate, incorporated as microbial substrate, decomposed rapidly whereas plant material naturally added in the field underwent slow microbial degradation with a greater proportion of the materials going into the fractions with high mean residence times.

The distribution of nitrogen was more closely associated with the  $^{14}$ C materials (r = 0.86\*) than with the distribution of soil carbon (r = 0.77) in the radiocarbon dated soil. Assuming that the distribution of nitrogen of the dated soil is applicable to the incubated experiment, one sees that the nitrogen was also in better correlation with the 14C (r = 0.83\*) than with the soil carbon (r = 0.78). Simon-

Table 3—Correlation between the measurements made on the fractions of the radiocarbon dated soil and the incubated soil labelled with 14C-acetate

	Radiocarbon dated soil					Incubated soil			
	∂ <sup>1 4</sup> C	14 C	Soil-C	N	14 C/Soil-C	14 C	Soll-C	14 C/Soil-C	
Incubated Soil		<del></del> -							
<sup>14</sup> C/Soll-C Soll-C	0. 66	‡ 0. 97**	 0. 98**	0.78	0.73	0. 95**	1	1	
<sup>14</sup> C Radlocarbon dated soll		0. 95**	0.90**	0.83*		1			
<sup>14</sup> C/Soll-C N Soll-C	0. 98** 	0. 86* 0. 98**	0. 77 1	1	1				
14 C		1		-					
∂14 C	1								

<sup>†</sup> Carbon content 2. 1%.

<sup>\*, \*\*</sup> Statistical significance .05 and .01, respectively.
† Calculations were based on the results of the following six fractions of Table 2: light materials, 0.5N hydrolyzate, 6N hydrolyzate, NaOH extract, water extract, humin.
‡ Correlation is not possible.

art and Mayaudon (20) have reported that when <sup>14</sup>C-substrates such as glucose, hemicellulose, or cellulose decomposed in soils, the <sup>14</sup>C and the nitrogen were similarly distributed in the fractions. This suggests that, in studying the transformation of soil organic matter, the active fraction may be associated with nitrogen and can be analysed using <sup>15</sup>N, but the resistant material should be investigated with carbon by either radiocarbon dating or a <sup>14</sup>C isotope used in conjunction with long term field experiments.

The carbon isotopic composition of the soil organic matter can affect the mean residence time because the radiocarbon dating method was first standardized against pre-1900 wood which had a δ<sup>13</sup>C of -25°/oo relative to the University of Chicago PDB standard or -23.9°/oo with respect to the NBS Solenhofen standard which was used in this work (5). Samples with  $\delta^{13}$ C values that are not -23.9°/oo need correction for having an isotopic fractionation different from that of pre-1900 wood. The  $\delta^{13}$ C values of the total soil organic matter from samples of Site 1 and Site 2 varied between -23.8°/oo and -28.1°/oo (Table 1) and averaged -25.4°/oo. These are similar to the values reported by Campbell et al. (3) for other soils in Saskatchewan. The corrections of mean residence time for isotopic discrimination against <sup>14</sup>C are made possible by considering that the <sup>14</sup>C/<sup>12</sup>C ratio is twice the <sup>13</sup>C/<sup>12</sup>C ratio (14). The average  $\delta^{13}$ C value of  $-25.4^{\circ}/oo$  for soil organic matter indicates a discrimination of 3.0°/00 against <sup>14</sup>C compared to the isotopic composition of pre-1900 wood and leads to an error of +25 years for the radiocarbon measurement. This error is more than cancelled out by the decrease in mean residence time caused by the incorporation of plant materials containing 14C derived from the atmospheric testing of thermonuclear weapons; consequently, it does not appear as an important factor in the radiocarbon dating of the soil organic matter used in this study.

### **CONCLUSION**

The radiocarbon dating of organic matter of a catena verified the statement that the stability of organic matter is closely related to the pedogenic factors under which the soil is formed. The change of the mean residence time with position in the catena reflects the increased turnover of organic matter in the lower slope positions without a major alteration of the total organic matter content. It is not the purpose of this work to describe the dynamics of slope movement since too many parameters are involved, but the properties of the soil organic matter, as characterized by the radiocarbon measurements, the C/N ratios and the hydrolysis of the total soil, show that important differences occurring along the slopes greatly affect the sampling requirements and the interpretation of soil organic matter investigations in soil genesis.

In these grassland soils, it appears that the organic matter of the A horizons normally turns over more rapidly than that of the Bt horizons where the physical effects of climate and animal mixing are greatly reduced. This is shown by a drastic change in the amount, the nature and the mean residence time of the organic matter between the A and the Bt horizons. The decreasing C/N ratios of the nonhydro-

lyzable materials from A to the Bt horizons suggest that relatively more nitrogen is stored in the resistant nonproteinaceous forms in the Bt horizons than in the A horizons. Alternatively, other nonorganic forms of nitrogen resistant to hydrolysis would have to be retained in this fraction.

This work also has indicated that a great percentage of carbon and nitrogen in soil, containing a large amount of clay minerals, is biologically active in that it dates modern. The 0.5N and 6N acid hydrolysis dissolved the young organic constituents of the soil in both the naturally <sup>14</sup>C-labelled field soil and under laboratory incubation where <sup>14</sup>C entered the soil organic matter as microbial residues. It is difficult to precisely define the structure of the humus from a dynamics standpoint, but the organic molecules that are not hydrolyzable certainly form a major portion of the resistant organic components in soil. These resistant components although active in that they affect soil physical and chemical properties play only a small role in the carbon cycle of these soils.

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