

# EFFECTS OF CULTIVATION ON THE ORGANIC MATTER OF GRASSLAND SOILS AS DETERMINED BY FRACTIONATION AND RADIOCARBON DATING<sup>1</sup>

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The effects of cultivation on the net mineralization of carbon and nitrogen in a lacustrine Brown clay (Sceptre) and two Orthic Black soils on glacial till (Oxbow) were assessed with the aid of fractionation and radiocarbon dating techniques. Fractionation of the soil organic matter of comparative virgin and cultivated soils by acid hydrolysis and peptization in dilute NaOH showed that the distribution of carbon and nitrogen among fractions of these soils was similar. There was no measurable alteration in the mean residence time (MRT) of the soil during the first 15 to 20 yr of cultivation, during which time the Sceptre soil had lost 19% of its carbon and the Oxbow, 35%. However, the MRT increased from 250 yr before present (BP) to 710 years BP after 60 yr of cultivation of the Oxbow soil. The losses for nitrogen were 10% lower than for carbon in the Oxbow soil due to the recycling of nitrogen in the soil. The rate of loss of carbon from the Oxbow soil during the cultivation period was simulated by expressing it as the sum of two first order reactions using fractionation and carbon dating data as the variables.

L'effet de la culture sur la minéralisation du carbone et de l'azote de sols provenant de la région des sols Bruns Foncés (Sceptre) déposés sur argile lacustre et de la région des sols Orthiques Noirs (Oxbow) déposés sur till glaciaire a été déterminé à l'aide du fractionnement de la matière organique et de la datation au radiocarbone. Le phénomène connu des pertes rapides de carbone et d'azote au cours des premières années de culture suivies par des pertes plus lentes a été retrouvé sur ces sols. Le fractionnement de la matière organique du sol, soit par un hydrolyse acide ou une peptisation dans du NaOH dilué, démontre une distribution semblable du carbone et de l'azote dans les fractions obtenues avant et après l'avènement de la culture. Le temps moyen de séjour ne varie pas beaucoup durant les 15 à 20 premières années de culture mais il passe de 250 ans à 710 ans après 60 années de culture sur le sol Oxbow. Le taux de minéralisation du carbone peut être simulé durant la période de culture par la somme de deux fonctions exponentielles. Le taux de minéralisation de l'azote ne peut pas être simulé de la même manière puisque l'azote est cyclé dans le sol. Les résultats obtenus avec le sol Oxbow démontrent que les pertes d'azote diminuent de 10% par rapport au carbone après la culture.

Soil deterioration, attributable to cultivation, has always been a problem in agriculture because maintenance of soil structure and fertility is essential in any cropping system. Comparatively few studies (Shutt 1925;

Newton et al. 1945; Doughty et al. 1949) on the extent and mechanisms of the losses of soil organic matter in the grassland soils of the semiarid Canadian prairies have been made, although it is recognized that soil organic matter is a primary natural source of available plant nutrients and an important factor in maintaining the desirable physical properties of soils.

Contradictory data have been found concerning the effects of cultivation on the distribution of soil organic matter. Keeney and

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Bremner (1964) concluded that cultivation did not result in marked changes in the relative proportions of hydrolyzable forms of nitrogen. Porter et al. (1964), however, showed that cultivation led to a larger decrease in the hydrolyzable nitrogen, particularly the amino acid fraction. They suggested that the amino acid N accumulated under native conditions. Huntjens (1972) and Khan (1971) have shown a preferential increase in the amino acid components in soils sown to grass or in a legume rotation.

Radiocarbon dating has been successfully used in characterizing soil organic matter (Campbell et al. 1967; Scharpenseel 1971), but there does not appear to have been any attempt to relate the radiocarbon measurements of virgin and cultivated soils of similar parent materials. This should be a valuable tool in measuring soil deterioration attributable to cultivation. Carbon dating studies also can lead to better understanding of the turnover of soil organic matter.

Numerous mathematical equations have been proposed to describe the decrease of soil organic matter upon cultivation of virgin land (Stevenson 1965). They are generally based on the first order reaction,

$$dM/dt = -rM + A \quad (1)$$

in which  $dM/dt$  is the rate of decline of organic matter (as determined by carbon or nitrogen);  $r$ , the proportionality constant, is negative and represents the rate of decomposition of the organic matter,  $M$  is the amount of organic matter present in the soil, and  $A$  is the amount added to the soil annually. Jenkinson (1966) pointed out the difficulties of using such an equation: (1) the rate of decomposition likely changes with time, (2) the organic matter has components that decompose at different rates, (3) the rate of addition of new material is not necessarily constant.

Information from radiocarbon dating and fractionation of soil organic matter can be used to measure changing rates of decomposition in the various soil fractions, and Hill (1954) has shown that the addition of new material in cultivated prairie soils is relatively constant through time. Jenkinson is presently working with soils that have well documented agricultural histories; he is

attempting to use the carbon dating technique to determine the rates of incorporation of residual constituents in the organic matter fractions (personal communication).

The present study was undertaken to determine the effects of cultivation on the rate of decomposition and the distribution of carbon and nitrogen in different fractions of the organic matter of three grassland soils. The second purpose was to determine whether first order kinetics could be used in simulating the rate of decomposition of organic matter in soils.

## MATERIALS AND METHODS

### Characterization of Soils

Bulk samples of Ah and Ap horizons were taken at three sites in Saskatchewan. Two sites, Quinton and Hafford, were classified as Orthic Oxbow Black Chernozemic soils containing 25% clay. Virgin and adjacent 15-yr and 60-yr cultivated soils were sampled at Quinton; virgin and corresponding 30-yr cultivated soils were sampled at Hafford. The soils of the third site (Matador — International Biological Program station) were classified as Sceptre Brown Chernozemic soils containing 70% clay. Virgin and adjacent 20-yr cultivated soils were sampled.

Identifiable plant materials in the soil were removed by repeated floatations with 0.01 N HCl followed by handpicking under 20X magnification of a stereomicroscope. Soil carbonates were removed with 1 N HCl. Organic carbon was determined in duplicate by dry combustion (St. Arnaud 1956) utilizing a trap of KI solution (50%) to absorb the HCl. Total nitrogen was determined in duplicate by the Kjeldahl method (Bremner 1965a).

### Fractionation of Soil Organic Matter

The soil organic matter was extracted and fractionated according to the procedure used by Biederbeck (1969); NaOH 0.5 N was the extractant and  $\text{Na}_2\text{S}_2\text{O}_4$  followed by 0.1 N HCl was used to remove Fe (Fig. 1). To shorten the tables and the text, the term fluvic acid is often replaced by FA, acid extract by AE, and humic acid by HA. Soil organic matter was also fractionated by successive hydrolysis using 0.5 N and 6 N HCl (Fig. 2).

### Radiocarbon Dating

Approximately 3 g of soil carbon were required for the radiocarbon counting (Campbell et al. 1967). The mean residence time (MRT) and

the  $\delta^{13}\text{C}$  were calculated by using the equations,

$$\text{MRT} = 18,500 \log_{10} (A_0/A) \quad (2)$$

and

$$\delta^{13}\text{C} = [(A - A_0)/A_0] \times 1,000 \quad (3)$$

where  $A_0$  was the rate of disintegration of a modern standard (95% of the activity of NBS oxalic acid) and  $A$  was the activity of the unknown sample. The MRT was quoted in years before present (BP) i.e. before 1950, and the  $^{13}\text{C}$  was expressed in per mil ( $\text{‰}$ ). The detailed procedure used for radiocarbon dating measurements was described elsewhere (Martel 1971).

### RESULTS AND DISCUSSION

#### Characterization of Organic Matter

The NaOH extraction showed a similar distribution of carbon and nitrogen in the virgin and the cultivated Sceptre soils (Table 1). In both instances, a mean of 41% of the organic carbon and 34% of the total nitrogen were not dissolved by this procedure and remained in the humin. Most of the extractable carbon and nitrogen were found in the acid-soluble fractions (FA + AE). Other data (results not presented) showed that, in both the virgin and the cultivated soils, 85% of the nitrogen in the mobile humic acids and 75% of the nitrogen in the nonmobile humic acids were hydrolyzable.

Therefore, cultivation of the Sceptre soils did not change the general distribution of the organic materials in the fractions extracted by the NaOH.

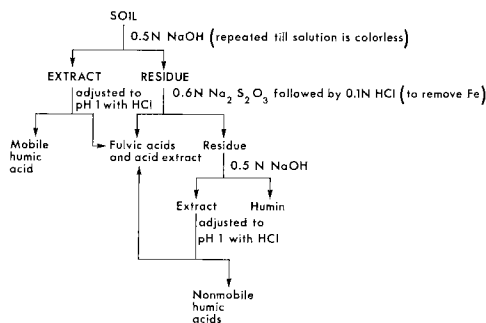


Fig. 1. Fractionation of soil organic matter by NaOH.

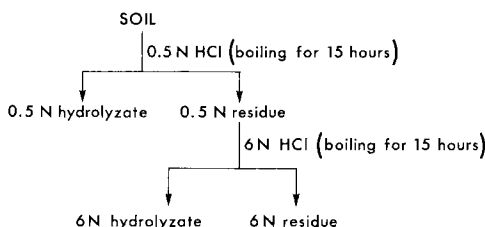


Fig. 2. Fractionation scheme of soil organic matter by HCl hydrolysis.

Table 1. Distribution of carbon and nitrogen in the fractions of soil organic matter

	Carbon		Nitrogen		C:N	
	Virgin	Cultivated	Virgin	Cultivated	Virgin	Cultivated
<i>NaOH Extraction</i>						
<i>Sceptre</i>						
FA + AE	100(2.6)†	100(2.1)	100(0.29)	100(0.26)	9.0	8.1
Mobile HA	36	33	44	43	7.3	6.2
Nonmobile HA	7	8	7	7	9.0	9.2
Humin	15	19	16	15	8.4	10.2
	42	40	33	35	11.4	9.2
<i>HCl Hydrolysis</i>						
<i>Sceptre</i>						
0.5N hydrolyzate	100(2.6)	100(2.1)	100(0.29)	100(0.26)	9.0	8.1
6N hydrolyzate	42	37	43	41	8.8	7.3
Residue	18	22	43	46	37	3.9
	40	41	14	13	26.0	26.0
<i>Oxbow from Quinton</i>						
0.5N hydrolyzate	100(5.4)	100(2.2)	100(0.45)	100(0.23)	12.0	9.6
6N hydrolyzate	35	31	51	46	7.1	6.4
Residue	12	14	25	30	4.9	4.5
	53	55	24	24	28.0	22.0

†Content, in percent, of organic carbon or total nitrogen in the soils.

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Table 2. Effects of cultivation on mean residence times and characteristics of the soil organic matter of oxbow and sceptre soils

	Orthic Oxbow from Quinton				Orthic Oxbow from Hafford						Sceptre		
	Virgin Ah 0-10 cm	15-yr cultivated Ap 0-10 cm	60-yr cultivated Ap 0-10 cm	Virgin Ah	Virgin Ah			30-yr cultivated Ap			Virgin Ah 0-10 cm	20-yr cultivated Ap 0-10 cm	
					Upper 0-7 cm	Middle 0-10 cm	Lower 0-12 cm	Upper 0-10 cm	Middle 0-12 cm	Lower 0-15 cm			
MRT (yr BP)	250±65	295±75	710±60	Modern	Modern	-	-	-	Modern	+21±7	-	545±70	430±70
$\delta^{14}\text{C}$ (‰)	-31±8	-36±9	-85±7	+33±7	-	-	-	-	-	-	-	-66±9	-52±9
Carbon (%)	5.4	3.5	2.2	7.3	9.7	2.7	4.3	4.6	2.6	2.1	0.26	2.6	2.1
Nitrogen (%)	0.45	0.34	0.23	0.60	0.81	0.27	0.38	0.41	0.29	0.26	0.26	0.29	0.26
C:N	12.0	10.4	9.6	12	12	10	11	11	9.0	8.1	8.1	9.0	8.1
Hydrolyzable — C†	47	49	45	50	40	56	52	45	60	59	60	60	59
Hydrolyzable — N†	76	78	76	71	66	80	78	73	86	87	86	86	87

†Percent of total soil content.

The standard deviation ( $1\sigma$ ) of the successive hydrolysis, measured as the amount of carbon extracted from the Sceptre soil, was 1%. The successive hydrolysis removed 9% more carbon than the single 6 N HCl hydrolysis. This difference was attributed to the formation of a black precipitate that was found on the walls of the flasks during the direct 6 N hydrolysis; no precipitate was detected during the successive hydrolysis. The latter was attributed to the removal of sugars and organic substances of low molecular weight by the initial acid treatment. These materials are said to condense with nitrogeneous compounds during direct boiling with 6 N acids to form insoluble complexes (Ogner 1969). The Sceptre soil had 15% more hydrolyzable carbon and 10% more hydrolyzable nitrogen than the Oxbow soil (Table 1). This supports Spørensens's (1967) laboratory findings that clays adsorb the newly formed humus and release it upon hydrolysis.

After 15 yr of cultivation, 65% of the organic carbon and 74% of the total original nitrogen were still present in the soil (Table 2). Only 41% of the original carbon and 51% of nitrogen were present after 60 yr of cultivation. These organic carbon and total nitrogen determinations, made in duplicate, had a coefficient of variation of less than 3%. The MRT of the soil organic matter remained unchanged after 15 yr, but increased from 250 to 710 years BP after 60 years cultivation. The percentage of hydrolyzable carbon and nitrogen did not change with cultivation and showed no relationship with MRT or with organic matter content. The MRT of the nonhydrolyzable materials of the Sceptre soil was  $1,100 \pm 100$  years BP (Martel and Paul 1970).

Three slope positions (upper, middle and lower) on the Orthic segments of virgin and cultivated soils at another Oxbow site (Hafford) were analyzed to evaluate the effects of drainage within the Orthic profile. The carbon and nitrogen contents in both the virgin and cultivated soils were greater in the lower slope positions of the catenary sequence (Table 2). Conversely, the hydrolyzability of carbon and nitrogen decreased by 15% on the virgin, and 10% on the

cultivated, as sampling progressed down the slope.

Cultivation of the land led to losses ranging from 33 to 52% for carbon and 31 to 49% for nitrogen, with largest losses occurring in the lower positions. Radiocarbon measurements of the soil showed positive  $\delta^{14}\text{C}$  values. This could be attributed to high levels of H-bomb-produced  $^{14}\text{C}$ , but the possibility of laboratory contamination is more likely; consequently the  $\delta^{14}\text{C}$  values for this soil are not further discussed.

Eighty-one percent of the organic carbon and 90% of the total nitrogen of the Sceptre virgin soil remained after 20 yr of cultivation. The percentages of hydrolyzable carbon and nitrogen showed no differences between the two sites. The virgin soils had an MRT of  $545 \pm 70$  yr BP and the cultivated  $430 \pm 70$ . Although a decreasing MRT is noticeable, the difference is not statistically significant.

#### Calculation of Carbon and Nitrogen Losses

Losses of carbon and nitrogen for cultivated soils at Quinton and Hafford were compared with data for another Black Chernozemic soil, the Indian Head Clay soil (Shutt 1925; Newton et al. 1945; McIver, personal communication). Similar losses of nitrogen from the Hafford and Quinton soils and the Indian Head clay soil were found, even though the nitrogen contents of the latter were based on the total soil organic matter rather than on the soil after flotation with 0.01 N HCl as conducted in this study.

Hydrolysis of the soil organic matter yielded hydrolyzable to nonhydrolyzable ratios close to 50:50 for carbon and 75:25 for nitrogen (Table 2). The hydrolyzable fraction comprised the active material of the soil organic matter (Martel and Paul 1970). Its MRT has previously been estimated at 25 yr (Campbell et al. 1967). On this basis, the first order reaction (equation 1) was divided into two parts, each one representing the active and the resistant fraction of the soil

$$\begin{aligned} dM/dt = & (-r_1M_1 + A_1) + \\ & (-r_2M_2 + A_2) \end{aligned} \quad (4)$$

and after integration

$$M = M_{1_0} e^{-r_1 t} + [(A_1/r_1) \times (1 - e^{-r_1 t})] + M_{2_0} e^{-r_2 t} + [(A_2/r_2) \times (1 - e^{-r_2 t})] \quad (5)$$

where  $M$  was the amount of materials (carbon or nitrogen) in soils expressed as a percentage of the materials present in virgin soils. The subscript  $i$  identified the hydrolyzable ( $i = 1$ ) and the nonhydrolyzable ( $i = 2$ ) materials,  $M_{i_0}$  was the percentage of the hydrolyzable and nonhydrolyzable materials in virgin soils,  $r_i$  was the rate of decomposition of the hydrolyzable and the nonhydrolyzable materials,  $A_i$  was the annual additions of new organic matter to the hydrolyzable and the nonhydrolyzable materials and  $t$  was the years of cultivation. The time zero represented the virgin soil and, at that time, the value of  $M$  was expressed as 100%. The calculations of the declines of carbon and nitrogen were made separately. Through the years of cultivation, 50% of the carbon is hydrolyzable, so  $M_{1_0} = M_{2_0} = 50$  for the carbon calculations; and 75% of the nitrogen was hydrolyzable so  $M_{1_0} = 75$ ;  $M_{2_0} = 25$  for the nitrogen calculations. The annual addition of new organic matter to the soil was estimated at 1 ton/acre every 2 yr or 1,000 kg/ha of residue annually (Shields and Paul 1973; McGill et al. 1974). On this basis, the annual addition of new residue carbon ( $A_1 + A_2$ ) was assumed to be 0.05 g C/100 g soil. Assuming that the C:N ratio of the new material was 30, we calculated an annual addition of 0.017 g N/100 g soil.

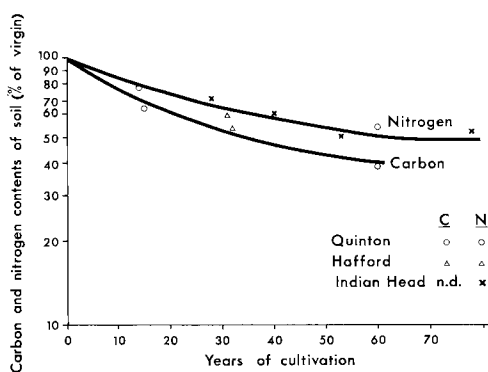


Fig. 3. Observed decline of carbon and nitrogen contents after cultivation of Oxbow and Indian Head Clay soils.

The decline of carbon and nitrogen was calculated (Fig. 4) taking into consideration that the MRT of the soil increased from 295 to 710 years BP after 15 yr of cultivation. The turnover rate ( $r$ ) was approximated from  $r_i = 1/\text{MRT}_i$  (Olson 1963). MRT's of 10, 15, 20 and 25 yr BP were assumed to be representative of the hydrolyzable fractions. The MRT's of the nonhydrolyzable fractions were calculated by using the MRT of the total soil and that of the hydrolyzable fraction (Paul et al. 1964). The  $r$  values were then used to calculate the decline of carbon and nitrogen.

Results showed that the decline of carbon based on 20 yr BP for the MRT of the hydrolyzable fraction gave a curve similar to the observed data of Fig. 3. It indicated that the sum of two first order equations yielded a calculated decline of the carbon content that was equivalent to the data obtained in the field after different lengths of cultivation.

The calculated decline of nitrogen (Fig. 4), based on a mean residence time of 20 yr BP indicated that only 20% of the nitrogen should be left after 60 yr of cultivation. This did not compare with the observed value of 50% (Fig. 3). One reason for the difference of 30% in the calculated value of nitrogen may be attributed to the release of organic compounds during hydrolysis (Bremner 1965b). This resulted in large values for hydrolyzable-N and a low calculated value of nitrogen after 60 yr of cultivation. The difference of 30% could not be attributed

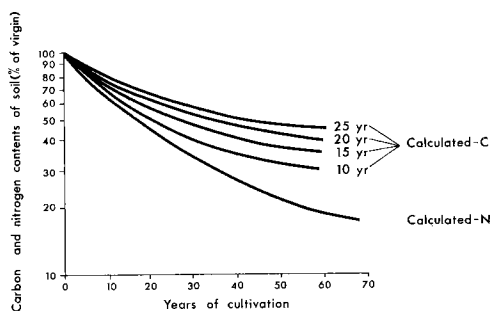


Fig. 4. Calculated decline of carbon and nitrogen using different mean residence times for the hydrolyzable carbon and 20 yr BP for the hydrolyzable nitrogen.

to fixed  $\text{NH}_4^+$  because calculation based on the data of Hinman (1964) showed that fixed ammonium accounted for only 3% of the 30% missing nitrogen.

The difference between the observed curves for carbon and nitrogen during 60 yr of cultivation was attributed to the recycling of nitrogen in soils. Carbon is decomposed to  $\text{CO}_2$  during decomposition, whereas the nitrogen is mineralized but reused by microorganisms and plants. This suggests that 10% of the amount of nitrogen left in the soil after 60 yr of cultivation is attributable to retention of mineral nitrogen by recycling.

### CONCLUSION

The large decrease of carbon and nitrogen during cultivation, with no measurable change in the distribution of the hydrolyzable and non-hydrolyzable materials, and the unchanged mean residence time of the organic matter during the first years of cultivation suggested that all forms of the organic matter fractions decomposed at a similar rate after cultivation independently of their individual stability in the virgin soil. The data suggested that the decline of carbon and nitrogen could be attributed to a change in the extent of physical protection of the soil organic matter, with recalcitrance of the humic material playing a smaller role. Cultivation also would enhance the contact of organic matter with extracellular enzymes.

The mechanisms related to the transformation of soil organic matter under field conditions are difficult to study. However, radiocarbon dating together with fractionation studies and the ability to represent carbon-loss mechanisms by a sum of first order reactions have made possible an interpretation of the dynamics of carbon. The high degree of hydrolyzability of nitrogen is not necessarily related to its turnover rate, and the internal cycling of nitrogen further makes it difficult to model the flow of this nutrient element.

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