Particulate and mineral-associated organic matter in water-stable aggregates as affected by mineral fertilizer and manure applications

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Aoyama, M., Angers, D. A. and N'Dayegamiye, A. 1999. **Particulate and mineral-associated organic matter in water-stable aggregates as affected by mineral fertilizer and manure applications.** Can. J. Soil Sci. **79**: 295–302. Application of cattle manure generally improves soil structure and organic matter (OM) content. However, changes in forms and location of OM within the aggregate structure are less well known. The effects of long-term (18-yr) applications of cattle manure (20 Mg ha^{-1} yr^{-1}) and NPK fertilizer on the distribution of particulate and mineral-associated organic matter in water-stable aggregates were investigated in a Le Bras loam (Humic Gleysol). Soil samples from the 0- to 10-cm depth were taken from the untreated control, NPK, manure and NPK + manure treatments. They were separated into four aggregate-size fractions (>1000 µm, 250–1000 µm, 53–250 µm, and <53 µm) by slaking air-dried soil, followed by wet sieving. Particulate (>53 µm) and mineral-associated OM (<53 µm) were separated from water-stable aggregates >53 µm by sieving after mechanical dispersion. After 18 yr, manure increased the OM level of the whole soil and favored formation of slaking-resistant macroaggregates (250–1000 µm). This effect was primarily a result of the OM added by the manure. In contrast, NPK fertilizer did not affect soil OM level or macroaggregation. The increase in OM induced by manure application was observed primarily in macroaggregates, and both as mineral-associated and particulate OM. However, manure did not change OM located in the fraction <53 µm confirming that recently deposited OM preferentially accumulates within the aggregate structure and not in the finely or non-aggregated material. Since previous studies have shown that most of the C in cattle manure is composed of coarse particles, we hypothesize that manure-derived OM first enters the soil primarily as particulate material, then, during decomposition, is transformed within the aggregate structure into mineral-associated material thereby contributing to aggregate stabilization.

Key words: Cattle manure, mineral fertilization, particulate organic matter, soil structure

Aoyama, M., Angers, D. A. et N'Dayegamiye, A. 1999. **Influence de l'application de fertilisants mineraux et de fumier sur les formes de matière organique dans les agrégats stable à l'eau.** Can. J. Soil Sci. **79**: 295–302. L'incorporation de fumier de bovin au sol conduit généralement à l'amélioration de sa structure et de sa teneur en matière organique (MO). Cependant, les effets sur la nature et la localisation de la MO sont moins bien connus. Nous avons étudié les conséquences à long terme (18 années) de l'application de fumier de bovin (20 Mg ha^{-1} année^{-1}) et de fertilisants minéraux sur la distribution de la MO fine et grossière dans les agrégats d'un loam Le Bras (Gleysol humique). Des échantillons de sol ont été prélevés de l'horizon 0–10 cm des parcelles témoin (sans amendement) et des parcelles traitées soit avec des fertilisants minéraux (NPK), du fumier de bovin, ou NPK + fumier. Les échantillons de sol ont été soumis à l'éclatement et séparés par tamisage sous eau en quatre fractions d'agrégat (>1000 µm, 250–1000 µm, 53–250 µm, <53 µm). Les fractions granulométriques grossières (>53 µm) et fine (<53 µm) de la MO ont ensuite été obtenues pour chaque fraction d'agrégat après dispersion mécanique et tamisage sous eau. L'application de fumier a permis une augmentation marquée de la teneur en MO et la formation d'agrégats stables de 250–1000 µm. Cependant, la fertilisation minérale seule n'a pas eu d'effet notable. L'augmentation de MO à la suite de l'application de fumier était apparente surtout dans les macroagrégats et autant sous forme fine que grossière. L'application de fumier n'a pas eu d'influence sur la MO localisée dans la fraction non-agrégée, ce qui confirme que la MO d'origine récente s'accumule dans la fraction agrégée (>53 µm) du sol et non dans la fraction finement ou non agrégée. Etant donné que des études antérieures ont montré que la MO des fumiers est plutôt de taille grossière, nous émettons l'hypothèse que la MO des fumiers est déposée dans le sol sous forme particulaire et est ensuite transformée en matériel fin associé aux particules minérales, contribuant ainsi à la stabilisation du sol.

Mots clés: Fumier de bovin, fertilisation minérale, matière organique particulaire, structure du sol

Long-term application of manure increases the level of OM in soil (Jenkinson and Rayner 1977). Soil OM accumulation induced by manure application is derived directly from the applied manure, as well as indirectly from the increased plant residues returned to the soil if manure application increases crop yields (Angers and N'Dayegamiye 1991). Particle-size fractionation allows the separation of soil OM according to its origin and degree of transformation. Organic matter of recent plant origin is believed to be preferentially recovered in the sand-size fraction, whereas more microbially processed material can be found in the silt- and

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clay-size fractions (Cheshire and Mundie 1981). However, Christensen (1988) noticed that changes in soil OM induced by long-term manure applications were observed only in fine fractions (clay- and silt-size particles). Angers and N'Dayegamiye (1991) observed that the application of solid cattle manure increased the OM content of all the soil particle size fractions and increased the soil carbohydrates of both plant and microbial origins. Aoyama (1992) also reported an increase in OM of all the particle size fractions separated from soils receiving farmyard manure.

Besides increasing soil OM, manure applications improve soil structure (Williams and Cooke 1961; Hafez 1974; N'Dayegamiye and Angers 1990). Soil OM has long been recognized as playing an important role in the formation of water-stable aggregates as a binding agent of soil particles (Tisdall and Oades 1982). According to the hierarchical model proposed by Tisdall and Oades (1982), stable microaggregates (<250 µm) are bound together to form stable macroaggregates (>250 µm). Elliott (1986) has shown that the intermicroaggregate binding agents in macroaggregates are composed of relatively labile OM. Cambardella and Elliott (1992, 1993, 1994) suggested that the labile organic pool within macroaggregates of grassland soils is either particulate OM or relatively low density, mineral-associated OM, probably of microbial origin. On the other hand, OM in microaggregates is more resistant to microbial decomposition than that in macroaggregates (Elliott 1986). In microaggregates, OM is present not only in mineral-associated form but also, to a lesser extent, as particulate OM, which is encrusted with mineral particles (Oades and Waters 1991).

In this study, we analyzed the distribution of particulate and mineral-associated OM among size classes of water-stable aggregates in soils amended with dairy cattle manure and/or NPK fertilizer for 18 yr. Our objective was to determine the effects of NPK and manure applications on the form and location of the accumulated OM within the aggregate structure.

MATERIALS AND METHODS

Site and Soils

The long-term field trial was established in 1978 at the St-Lambert-de-Lévis Soil Research Station of the Ministère de l'Agriculture, des Pêcheries et de l'Alimentation du Québec (46°34′N, 71°13′W). The soil is a Le Bras silty loam to loam (Humic Gleysol) with an average of 30% sand, 46% silt and 24% clay, and a $pH_{(H_2O)}$ of 6.2 at the beginning of the experiment. The field trial was arranged in a split-plot design with dairy cattle manure as the main plots and fertilizer treatments as subplots in three replicates. Each subplot measured 8 × 10 m. Each of the subplots was subdivided to accommodate the four phases of the rotation. Crops included silage corn (*Zea mays* L.), wheat (*Triticum aestivum* L.), beetroot (*Beta vulgaris* L.), canola (*Brassica campestris* L.) and barley (*Hordeum vulgaris* L.). Rotation sequences were corn–corn–wheat–beetroot from 1978 to 1985, corn–corn–wheat–canola from 1986 to 1989 and corn–corn–wheat–barley since 1990. For the cereals and for corn, aboveground residues were removed from the field. Dairy cattle manure was applied each fall prior to 1987 and thereafter each spring at rates of 0 and 20 Mg ha^{-1} on a wet-weight basis. On average, the manure contained 9.4% C and 0.44% N on a dry basis. Manure provided an average of about 90 kg N ha^{-1} yr^{-1}. Fertilizer treatments were NK, PK, NP, NPK, NPKMg and a control without fertilizer. Fertilizer rates were as recommended for the different crops in Québec following soil analysis. Tillage was performed annually and involved autumn moldboard plowing to a depth of 20 cm and spring harrowing. Every 5 yr, lime was applied on all plots at 3 Mg ha^{-1}. Further details of the trial have been reported by N'Dayegamiye (1996) and N'Dayegamiye et al. (1997).

For the present study, soil samples from the untreated control, NPK, manure, and NPK + manure treatments were taken in the 0- to 10-cm soil layer before manure application in the spring of 1996. Ten subsamples were taken from each of the three replicated plots per treatment and pooled to give one sample per plot. The soil samples were completely and gently passed through a 6-mm sieve in the field and air-dried in the laboratory.

Water-stable Aggregates and C and N Analyses

Size fractions of water-stable aggregates were separated by slaking of air-dried soil followed by wet sieving. One hundred grams of each air-dried soil sample was spread on the top of a nest of sieves with openings of 1000 and 250 µm. The sieves were shaken in deionized water for 5 min using an apparatus similar to that described by Bourget and Kemp (1957), and slaking was allowed to occur as aggregates were directly immersed in water without prewetting. After shaking, the aggregates on each sieve were collected by washing with deionized water to yield fractions >1000, 250–1000 and <250 µm. A few pieces of floating plant residues were present and were discarded as our primary interest was in aggregated material. The material <250 µm was poured on a 53-µm sieve and washed with deionized water until the washing water became clear. This yielded a microaggregate fraction of 53–250 µm and material <53 µm. The fraction <53 µm was collected by centrifugation at 1000 × g for 15 min. The <53 µm fraction contained both small microaggregates and non-aggregated particles. Each fraction was dried in a forced air oven at 40°C and weighed. A subsample from each fraction was oven-dried at 105°C to allow correction to a final dry weight.

Subsamples of aggregate size fractions were ground in a mortar and pestle to pass a 250-mm sieve. Total C and N were simultaneously determined using a LECO CNS-1000 automatic analyzer (Leco Corp., St. Joseph, MI). Since carbonates were not present, total C was equivalent to organic C.

Particulate and Mineral-associated OM in Aggregrate Size Fractions

Particulate (>53 µm) and mineral-associated OM (<53 µm) were separated from each water-stable aggregate fraction by sieving after mechanical dispersion of the soil by agitation in water with glass beads (Balesdent et al. 1991). For the aggregate size fractions >1000, 250–1000 and 53–250 µm, subsamples (5–10 g) were shaken with 50 mL deionized water and five glass beads (6 mm in diameter) for 16 h on a reciprocal shaker. The dispersed particulate OM plus sand were collected on a 53-µm sieve and washed with deionized water. The material <53 µm was dried in a forced-air oven at 40°C and weighed. The weight of particles >53 µm in

Table 1. Soil C and N contents in the 0–10 cm depth from the field plots

	Total C	Total N	C/N
	g kg^{-1} soil		
Treatment			
Control	21.6	1.33	16.2
NPK	22.9	1.49	15.4
Manure	31.4	1.91	16.4
Manure + NPK	28.0	1.84	15.2
F-probability			
Manure	<0.001	<0.001	0.99
NPK	0.28	0.34	0.05
Manure × NPK	0.03	0.05	0.68

Table 4. Carbon-to-N ratios of the water-stable aggregates (sand-free basis)

	Aggregate size			
Treatment	>1000 μm	250–1000 μm	53–250 μm	<53 μm
C-to-N ratio				
Control	23.6	17.6	16.4	14.7
NPK	22.4	16.8	16.1	14.3
Manure	17.9	16.2	16.3	15.5
Manure + NPK	17.8	15.9	16.1	14.9
F-probability				
Manure	<0.001	0.08	0.92	0.15
NPK	0.29	0.28	0.72	0.26
Manure × NPK	0.26	0.62	0.82	0.70

Table 2. Distribution of aggregate size fractions in whole soil

	>1000 μm		250–1000 μm		53–250 μm		
Treatment	Whole	Sand-free	Whole	Sand-free	Whole	Sand-free	<53 μm
Water-stable aggregates (% of whole soil dry weight)							
Control	9.8	4.0	14.9	7.9	47.3	24.8	28.7
NPK	12.1	4.9	21.0	11.2	40.7	18.7	25.8
Manure	11.0	5.4	26.6	16.2	40.3	21.1	21.7
Manure + NPK	11.0	5.2	27.4	16.1	39.3	18.7	22.1
F-probability							
Manure	0.96	0.08	0.04	<0.01	0.02	0.44	0.23
NPK	0.47	0.41	0.35	0.35	0.03	0.11	0.77
Manure × NPK	0.47	0.21	0.48	0.31	0.09	0.43	0.69

Table 3. Carbon and N contents of water-stable aggregates (sand-free basis)

	C				N			
	>1000 μm	250–1000 μm	53–250 μm	<53 μm	>1000 μm	250–1000 μm	53–250 μm	<53 μm
	g C kg^{-1} sand-free aggregates				g N kg^{-1} sand-free aggregates			
Treatment								
Control	40.1	39.1	38.6	22.1	1.70	2.22	2.36	1.50
NPK	44.6	44.5	45.1	25.2	1.99	2.65	2.80	1.76
Manure	60.2	56.6	55.4	28.1	3.37	3.49	3.41	1.81
Manure + NPK	56.1	52.1	54.3	26.4	3.15	3.28	3.37	1.77
F-probability								
Manure	<0.01	<0.01	<0.001	0.07	<0.001	<0.01	<0.001	0.34
NPK	0.97	0.87	0.15	0.71	0.90	0.63	0.25	0.50
Manure × NPK	0.29	0.12	0.06	0.19	0.23	0.18	0.16	0.38

each aggregate size fraction was determined by the difference between the weight of undispersed aggregates and that of the material <53 μm. The amounts of total C and N in the mineral-associated OM were directly determined by the LECO CNS-1000 automatic analyzer. The total amounts of C and N in the particulate OM were estimated by subtracting the total contents of C and N in the mineral-associated OM from those in the intact aggregate size fraction (Cambardella and Elliott 1992).

Statistical Analysis

Treatment effects were tested using an analysis of variance (ANOVA) as a split-plot with manure as the main factor and NPK as the sub-plot factor.

RESULTS

Manure application significantly increased the concentrations of C and N in whole soils as indicated by the highly significant main effect of manure (Table 1). However, the main effect of NPK was not significant. The NPK × manure interactions were only weakly significant and were due to the fact that NPK had a very small, but opposite, effect on C and N contents in the presence and in the absence of manure (Table 1). Overall, the effect of NPK was considered to be not significant. The C/N ratios are relatively high at this site and are consistent with previous work at the same location (Angers and N'Dayegamiye 1991; N'Dayegamiye et al. 1997). The only significant effect of the treatments on the C/N ratio was a slight decrease with NPK fertilization.

Table 5. Particulate and mineral-associated C and N contents of aggregate size fractions (sand-free basis)

	>1000 μm		250–1000 μm		53–250 μm	
Treatment	Particulate	Mineral-associated	Particulate	Mineral-associated	Particulate	Mineral-associated
Carbon (g C kg^{-1} sand-free aggregates)						
Control	20.6	19.5	11.4	27.7	8.0	30.6
NPK	22.8	21.7	13.0	31.5	10.7	34.4
Manure	25.5	34.7	16.0	40.5	13.3	42.2
Manure + NPK	26.7	29.4	15.1	37.0	14.9	39.3
F-probability						
Manure	0.02	<0.01	0.07	<0.01	0.02	0.01
NPK	0.28	0.57	0.84	0.95	0.20	0.87
Manure × NPK	0.71	0.19	0.43	0.19	0.75	0.25
Nitrogen (g N kg^{-1} sand-free aggregates)						
Control	0.56	1.14	0.34	1.89	0.34	2.02
NPK	0.67	1.32	0.44	2.21	0.43	2.37
Manure	1.13	2.24	0.82	2.67	0.63	2.78
Manure + NPK	1.20	1.95	0.74	2.53	0.62	2.75
F-probability						
Manure	<0.001	<0.01	<0.01	0.04	0.13	0.04
NPK	0.33	0.76	0.85	0.68	0.79	0.49
Manure × NPK	0.80	0.19	0.31	0.33	0.72	0.41

Table 6. Carbon to N ratios of particulate and mineral-associated organic matter in aggregate size fraction (sand-free basis)

	>1000 μm		250–1000 μm		53–250 μm	
Treatment	Particulate	Mineral-associated	Particulate	Mineral-associated	Particulate	Mineral-associated
C-to-N ratio						
Control	36.8	17.1	33.5	14.7	23.5	15.2
NPK	34.0	16.4	29.6	14.3	24.9	14.5
Manure	22.6	15.5	19.5	15.2	21.1	15.2
Manure + NPK	22.3	15.1	20.4	14.6	24.0	14.3
F-probability						
Manure	<0.01	<0.01	<0.001	0.28	0.91	0.81
NPK	0.62	0.19	0.47	0.28	0.35	0.96
Manure × NPK	0.64	0.57	0.25	0.85	0.93	0.81

More than 99.6% of whole soil dry weight was recovered after wet-sieving, indicating that losses during the fractionation process were negligible. For all treatments, the quantity of soil material was greatest in the 53–250 μm fraction and least in the fraction >1000 μm (Table 2). As sand particles (>53 μm) accumulated differently in the various aggregate fractions, we normalized each aggregate fraction to a sand-free basis (Elliott et al. 1991; Beare et al. 1994). On a sand-free basis, the application of manure significantly increased the proportion of small macroaggregates (250–1000 μm) and tended to increase ($P = 0.08$) the large macroaggregates (>1000 μm), whereas the NPK fertilization did not influence the aggregate size distribution.

The concentrations of C and N in sand-free aggregates generally increased with aggregate size (Table 3). The element concentrations also significantly increased with manure application, except for the size class <53 μm, whereas NPK fertilization had no significant effect on any fraction (Table 3). The C/N ratio in the large macroaggregates (>1000 μm) was lower when manure was applied; a similar tendency ($P = 0.08$) was observed in the small macroaggregates (250–1000 μm) (Table 4).

Manure application also significantly increased the contents of both particulate and mineral-associated C and N (Table 5) in the sand-free aggregates >53 μm; however, the application of NPK had no effect. Concentrations of particulate C and N were lower than those of mineral-associated C and N, except for C in the large macroaggregates (>1000 μm) from the control and NPK treatments. Further, manure application caused a significant decrease of the C/N ratio in both the particulate and mineral-associated OM in the large macroaggregates (>1000 μm) and in the particulate OM in the small macroaggregates (250–1000 μm) (Table 6).

The amounts of C and N in each size fraction were calculated by multiplying the content of C and N (g kg^{-1} of whole aggregate) by the quantity of soil in each whole aggregate size fraction. The largest portion of the OM occurred in the large microaggregates (53–250 μm) (Figs. 1 and 2). The amounts of both the particulate and mineral-associated C and N were greater in treatments receiving manure than in the non-manured one. This was particularly true in the small macroaggregates (250–1000 μm) reflecting the greater amounts of soil material and greater C and N concentrations in this fraction from the manured soils. The application of

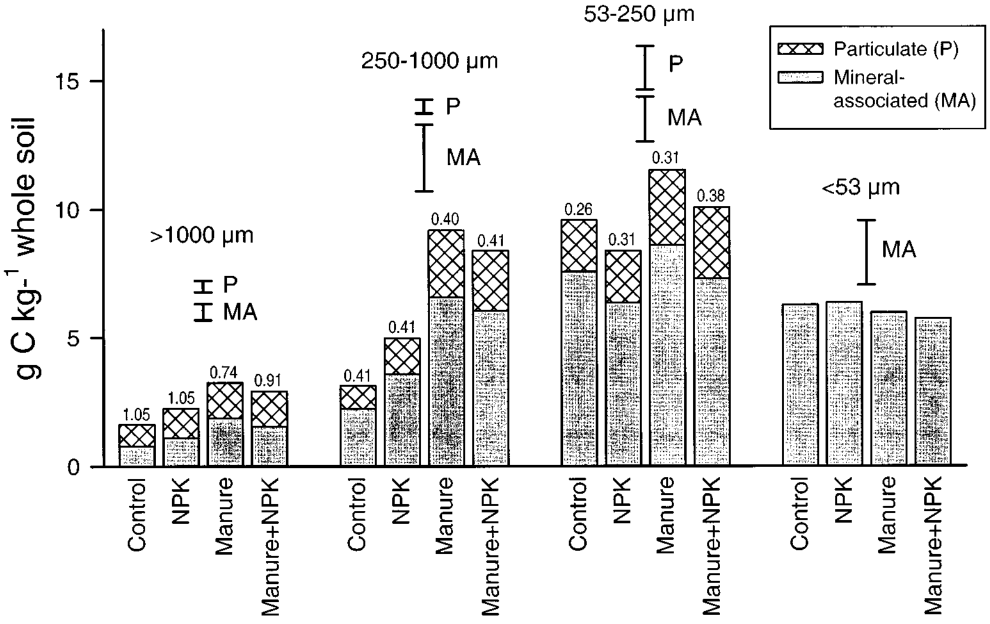

Fig. 1. Amount of particulate and mineral-associated C per kg of whole soil. Values above bars indicate the ratio of particulate C to mineral-associated C. Vertical lines represent **least significant differences (LSD)** at the 0.05 probability level.

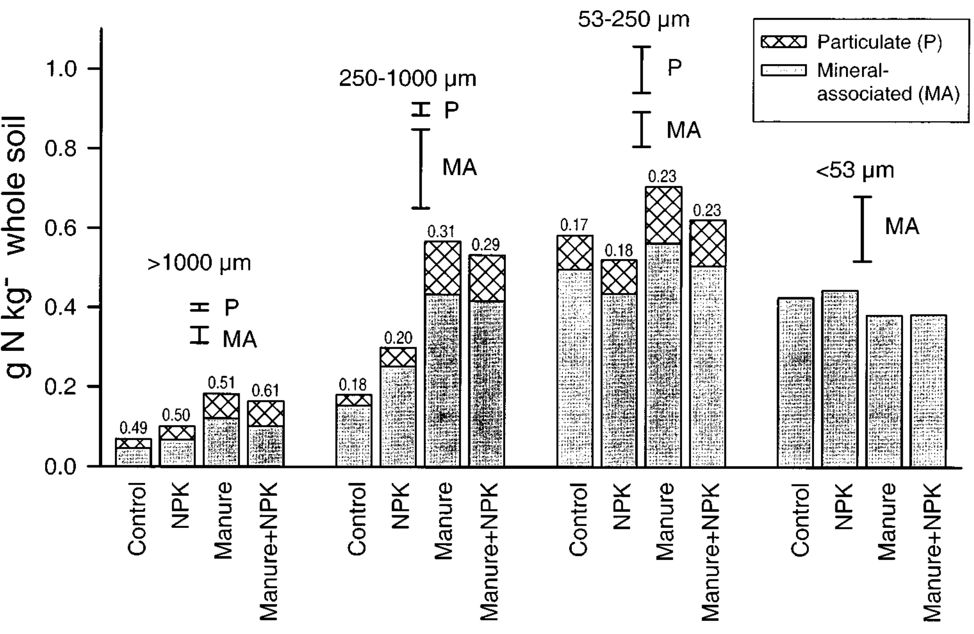

Fig. 2. Amount of particulate and mineral-associated N per kg of whole soil. Values above bars indicate the ratio of particulate N to mineral-associated N. Vertical lines represent LSD at the 0.05 probability level.

NPK also increased somewhat the particulate and mineral-associated C and N in macroaggregates. The amount of particulate C and N in any aggregate fraction was usually less than those of the mineral-associated, except for C in the large macroaggregates (>1000 µm). The ratio of particulate/mineral-associated C or N decreased with decreasing aggregate size class. The different treatments had a significant effect on this ratio only for the aggregates >1000 µm.

DISCUSSION

The application of cattle manure increased the level of OM and favored the formation of water-stable macroaggregates, especially the aggregate size class 250–1000 µm, compared with the other treatments. The formation of water-stable macroaggregates >2 mm concurrently with the increase in the soil OM have been observed in these and other field plots at the same location with application of cattle manure (N'Dayegamiye and Angers 1990; Angers and N'Dayegamiye 1991; N'Dayegamiye et al. 1997). These previously reported findings are only partly in line with our present results. The discrepancy is in the size of the water-stable aggregates resulting from the manure application. We attributed this to the fact that N'Dayegamiye and Angers (1990) and N'Dayegamiye et al. (1997) used field-moist instead of air-dried soils for separating water-stable aggregates. As demonstrated by Elliott (1986), wet sieving of air-dried soils induces slaking with considerable reduction in macroaggregate size.

The increase in soil OM content resulting from the application of manure can originate directly from the applied manure as well as from the plant biomass when manure increases crop yields and residue input to the soil. The average yields of silage corn and wheat recorded from 1987 to 1991 in the plots under study, were in the order of NPK + manure > manure = NPK > control (N'Dayegamiye 1996). This suggests that the amount of OM added as belowground plant biomass was probably similar in the NPK and the manure-alone treatment. Yet soil OM was greater in the manure than in the NPK treatment. This suggests that the additional accumulated OM in the soils receiving manure application was mostly derived from the applied manure itself, and not from an increased plant biomass input. The differences in C and N contents between the NPK-alone and the control treatments were very small. This is contrary to some other studies in which long-term application of mineral fertilizer resulted in increased soil C content due to an increase in plant productivity and residue input (Campbell and Zentner 1993; Gregorich et al. 1996) but in line with others where the effect was negligible (e.g. Campbell et al. 1991). The absence of such an effect in our study can be explained partly by the fact that aboveground residues were removed from the field for most crops of the rotations.

We showed that manure application increased the concentration of both particulate and mineral-associated OM in aggregate fractions. Christensen (1988) observed that the application of farmyard manure increased the clay- and silt-associated OM of the whole soil relative to the unmanured soil, while no changes were detected in the sand-size fraction. In contrast, Angers and N'Dayegamiye (1991) and Aoyama (1992) reported that manure application increased not only the clay- and silt-associated OM, but also the sand-size OM. The latter findings are in agreement with our present results. Cattle manure OM is largely composed of coarse particles in which carbohydrates are of plant origin, and fine and water-soluble fractions comprised only a small portion (Aoyama 1985, 1991). Thus, we hypothesized that when manure is incorporated into soil, the particulate OM gradually decomposes to produce microbial biomass, metabolic products and humic substances associated with clay- and silt-size particles.

In addition to increasing their concentration in particulate and mineral-associated OM, manure application increased the amount of macroaggregates of 250–500 μm. It is thought that part of the mineral-associated OM derived from the decomposition of particulate OM from manure is participating in the formation and stabilization of macroaggregates. Cambardella and Elliott (1993, 1994) suggested that, together with particulate OM, mineral-associated OM of relatively low density and probably of microbial origin, acted as inter-microaggregate binding agents in cultivated grasslands. Decomposition of particulate OM leads to the production of fungal hyphae (Tisdall 1994) and microbial polysaccharides (Haynes and Francis 1993), which contribute to the stability of soil aggregates. However, Golchin et al. (1997) proposed that macroaggregates that formed and stabilized around decomposing particulate OM are only transient due to the rapid decomposition and fragmentation of their organic cores. Nevertheless, in our study, manure application had its greatest effect on macroaggregates. It is likely that the constant supply of OM through yearly manure applications maintained a high level of microbial activity and binding agent production in the macroaggregates.

An increase in both particulate and mineral-associated OM concentration in the large microaggregate fraction (53–250 μm) was also induced by the long-term application of manure, but without a concurrent increase in the amount of aggregates in that fraction. Based on electron microscopic observations (Oades and Waters 1991) and spectroscopic analyses of organo-mineral fractions, Golchin et al. (1994, 1997) proposed that stable aggregates of 20–250 μm consist of small fragments of partially degraded plant debris bound in a matrix of mucilages and mineral materials. They further suggested that organic cores of microaggregates decompose slowly and are protected from rapid decomposition by encrustation with inorganic materials. Besnard et al. (1996) showed that the particulate OM occluded within microaggregates 50–200 mm had a slow turnover rate compared with particulate OM in other locations. They proposed that this may be due either to its recalcitrant chemical nature or to its physical protection within microaggregates. The fact that the manure-induced accumulation of OM in the large microaggregates did not result in an increase in the amount of aggregates in that fraction would suggest that part of this OM is already partially decomposed and biologically recalcitrant, resulting in low production of metabolites and mucilages capable of binding soil particles (Golchin et al. 1997).

In contrast to the large microaggregates (53–250 μm) or the macroaggregates, the OM of the smallest aggregate fraction (<53 μm) was not affected by manure application. The mineral-associated OM produced during the decomposition of manure-derived particulate OM would therefore only be found in the macroaggregates and large microaggregates. This finding confirms the results of several recent isotopic studies using aggregate separation (without complete soil dispersion), which have shown that recently deposited OM is located preferentially in the aggregate structure and not in the finely or non-aggregated material (Buyanovsky et al. 1994; Puget et al. 1995; Angers and Giroux 1996; Angers et al. 1997).

Carbon and nitrogen generally showed a fairly similar response to manure and fertilizer applications. However, one noticeable difference was in the large macroaggregates (Table 3) where manure application resulted in proportionally greater increase in N than in C contents which was reflected by a much lower C/N ratio in the macroaggregates from the manured plots (Table 4). This difference was mostly apparent in the particulate fraction (Table 6), which would suggest that the particulate OM originating from the manure had a lower C/N ratio than that from crop residues. Also, N from manure may be recalcitrant and accumulate in macroaggregates. It may also be that N was better preserved in macroaggregates when manure was applied due to the greater microbial biomass and activity (M. Aoyama et al., unpublished data) and consequent greater immobilization and internal cycling of N.

CONCLUSIONS

This study showed that the long-term application of cattle manure resulted in an increase in both particulate and mineral-associated OM within water-stable aggregates >53 μm. At the same time, the manure application improved the formation of water-stable macroaggregates and the manure-derived OM accumulated preferentially in these macroaggregates, especially in the small macroaggregates (250–1000 μm). This effect was primarily a result of the OM added by the manure and not crop residue inputs. Since previous work has shown that the C in cattle manure was mostly coarse and of plant origin, we hypothesized that manure-derived OM first enters the soil primarily as particulate material and during decomposition is transformed within the aggregate structure in mineral-associated material of microbial origin, thereby contributing to aggregate stabilization.

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