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## SOIL ORGANIC $^{14}\text{C}$ DYNAMICS: EFFECTS OF PASTURE INSTALLATION ON ARABLE LAND

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**ABSTRACT.** In a study addressing composition and recovery of soil carbon following pasture installation on arable land, radiocarbon isotope ratios were measured in size- and density-separated soil organic matter (SOM) fractions in a pasture and maize plot. The average soil carbon age increased with depth from 444 yr in the 0–30-cm layer to 2456 yr in the 60–80-cm layer in the pasture soils, and from 42 to 1625 yr in the maize-cultivated soil. Weight fractionation of the macro-organic matter (size  $>150\ \mu\text{m}$ ) in a light (density  $<1.17\ \text{g cm}^{-3}$ ) intermediate ( $1.17\ \text{g cm}^{-3} < \text{density} < 1.37\ \text{g cm}^{-3}$ ), and heavy fraction (density  $>1.37\ \text{g cm}^{-3}$ ) resulted in markedly different ages for different fractions with ages increasing from 2 yr in the light fraction to  $>3000$  yr in the heavy fractions.  $^{13}\text{C}$  and  $^{14}\text{C}$  (accelerator mass spectrometry (AMS)) isotope ratios in the  $<20\ \mu\text{m}$  fraction in the 60–80-cm layer indicated that vertical displacement of colloidal organic material occurred during maize cropping. The physical fractionation method, in combination with natural level  $^{13}\text{C}$  and  $^{14}\text{C}$  analysis, resulted in a better insight in carbon dynamics that occur after the conversion of arable land to pasture.

### INTRODUCTION

The conversion of arable land into forest or pasture causes considerable changes in the organic matter dynamics in soils and has a profound impact on soil physical and chemical properties (Römkens and de Vries 1995). Size, composition and turnover rate of the soil organic matter (SOM) pool are essential factors for the retention and transfer of trace metals (Schulin, Geiger and Furrer 1995). The study of SOM cycling, however, is difficult, and indirect approaches employing mathematical modeling and computer simulation have been used to simulate organic carbon dynamics in soil (van Veen 1992). Studies on the long-term dynamics of SOM using radiocarbon dating and  $^{14}\text{C}$  tracer methods have shown that the life span of different fractions varies from less than one to several thousand years (Jenkinson and Rayner 1977; Hsieh 1994). Based on these findings, mechanical long-term models have been developed that describe carbon dynamics using hypothetical carbon- or SOM fractions with varying degrees of stability (van Veen and Paul 1981; van Veen, Ladd and Amato 1985; Parton *et al.* 1987; Parton, Stewart and Cole 1988; Verberne *et al.* 1990). Meyboom, Hassink and van Noordijk (1995) developed a nondestructive method to separate these fractions. Recent results indicate that the method can separate young organic material with a high turnover rate from older, more stabilized material with much lower turnover rates (Hassink 1995b). The distinction between “young” and “old” carbon in the study by Hassink (1995b), however, was based entirely on the calculation of the average half-life time in each fraction, but was not verified with measurements of the absolute age of the samples. In this study, we used accelerator mass spectrometry (AMS)  $^{14}\text{C}$  dating to measure the natural  $^{14}\text{C}$  content in size- and density fractionated SOM. Until now,  $^{14}\text{C}$  dating has been applied mainly to non-fractionated soil samples (*e.g.*, Hsieh 1992; Jenkinson and Coleman 1994; Harrison, Post and Richter 1995). The results indicated that the average soil carbon “age”

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increased with depth from a few hundred years in the topsoil to >3000 yr in the subsoil. However, the SOM pool in each soil horizon contains fractions with varying  $^{14}\text{C}$  contents. Trumbore (1993) and Trumbore *et al.* (1995), using  $^{14}\text{C}$  data on SOM and fractionated SOM, revealed that at least three distinctly different fractions exist with turnover times ranging from <10 to >1000 yr.  $^{14}\text{C}$  data of a light (density <2.0 g cm $^{-3}$ ) and heavy (density >2.0 g cm $^{-3}$ ) SOM fraction showed that the average age increased with fraction density. Aside from the lack of background data on separate SOM fractions, the majority of recent studies are based on measurements of soil  $^{14}\text{C}$  activity levels following addition of  $^{14}\text{C}$ -enriched material (Nowak and Nowak 1990; Ladd *et al.* 1996; Magid, Gorissen and Giller 1996; van Gestel, Merckx and Vlassak 1996). This enabled the authors to determine the degree of incorporation of  $^{14}\text{C}$ -labeled carbon into SOM and its different (sized) fractions. However, carbon dynamics in pools with long turnover rates (*e.g.*, in the heavy or finest fractions) cannot be studied by this approach: the addition of labeled material does not give relevant information on the turnover of *native* soil carbon. The derivation of the turnover time of native soil carbon, therefore, can only be achieved by the measurement of the natural  $^{14}\text{C}$  content of soil samples or SOM fractions. AMS  $^{14}\text{C}$  dating allows for precise measurements of small amounts of  $^{14}\text{C}$ , which enables us to determine the natural  $^{14}\text{C}$  content in size- and density-separated SOM. We present data on natural level  $^{13}\text{C}$  and  $^{14}\text{C}$  of SOM and SOM fractions from mature pasture sites and maize fields. The combination of  $^{13}\text{C}$  and  $^{14}\text{C}$  analysis and the carbon content in the size and density SOM fractions will be used to elucidate the consequences of land use changes with respect to soil organic carbon dynamics.

## METHODS

### Site Description and Sampling Procedures

We used soil samples from the Cranendonck experimental station, located in the province of Noord-Brabant, the Netherlands. At the site, several long-term fertilization experiments (nitrogen, phosphorus and potassium) are being carried out. The site is divided into three large replicate blocks; within each block all experimental treatments are performed simultaneously within subplots. The treatments studied here include plots with continuous maize cropping (from 1968 onwards) vs. continuous pasture plots (referred to here as "maize" and "pasture" in the tables and figures). All sites received a similar amount of inorganic nitrogen fertilizer at a rate of 140 kg N ha $^{-1}$  yr $^{-1}$ . Further, cow manure was applied to all plots at a rate of 15–25 m $^3$  ha $^{-1}$  yr $^{-1}$  once every 2 yr. Samples were taken from each replicate block at three different depths: 0–30 cm, 40–60 cm and 60–80 cm. From each plot and depth layer, a mixed soil sample of 2 kg was taken. Coarse root material was removed manually. SOM fractionation was performed on field moist samples, after which the remaining soil was dried at 40°C for 24 h. The dried and sieved (<2 mm) soil samples were used for the analysis of the total organic matter content by combustion at 850°C. In Table 1, general soil characteristics for the topsoil in the grassland plot are shown as well as data of the organic matter content (whole soils) and the distribution of soil carbon among fractions for all depth layers and land use types. Textural differences between plots and blocks are negligible.

### Organic Matter Fractionation

SOM was fractionated according to Meyboom, Hassink and van Noordwijk (1995); it includes a size and density separation. The method is based on wet-sieving of soil samples into four fractions: >250  $\mu\text{m}$ , 150–250  $\mu\text{m}$ , 20–150  $\mu\text{m}$ , and the remaining fraction <20  $\mu\text{m}$ . Both the 150–250  $\mu\text{m}$  and the >250  $\mu\text{m}$  are further (weight) fractionated using an inert silica gel (Du Pont Ludox<sup>TM</sup>) with densities of 1.13 and 1.37 g cm $^{-3}$ . No density separation was performed on the finer fractions (20–150  $\mu\text{m}$  and <20  $\mu\text{m}$ ), which mainly contained strongly humified organic matter attached to soil particles.

TABLE 1. General Soil Characteristics (Pasture)\*

Depth (cm)	Texture				SOM (%)	C/N ratio	pH (1 N KCl)
	<2 μm	2–16 μm	16–50 μm	>50 μm			
0–30	3.2	1.6	16.4	78.8	2.6	18.2	5.4
60–80	1.4	1.4	17.1	80.1	0.9	24.1	4.9

\*Soil Type: Leptic Podzol (FAO 1990). Entic Haplorthod (Soil Survey Staff 1975)

### Carbon Isotope Analysis

Soil samples and size- and density-fractionated SOM (10–300 mg, depending on the carbon content) were combusted at 850°C under a continuous flow of oxygen. The CO<sub>2</sub> released was purified by CuO and Ag using standard methods. <sup>13</sup>C values were measured on a VG/SIRA 9 Mass Spectrometer at the Center for Isotope Research, Groningen. Following <sup>13</sup>C analysis, the CO<sub>2</sub> was trapped cryogenically and converted to graphite for subsequent AMS analysis. The <sup>14</sup>C activity was measured by the Groningen AMS facility (Gottdang, Mous and van der Plicht 1995; van der Plicht 1996). The amount of <sup>14</sup>C in SOM relative to the <sup>14</sup>C concentration in the atmosphere can be used to calculate the age of the material. In order to correct for the bomb peak from nuclear testing and the Suess effect, <sup>14</sup>C activities were converted to ages based on the atmospheric <sup>14</sup>CO<sub>2</sub> record from Groningen (Meijer *et al.* 1994). The <sup>14</sup>C results are given in ‰ excess, corrected for isotopic fractionation and radioactive decay

$$\Delta^{14}\text{C} = \left[ e^{\frac{-T}{8033}} e^{\frac{1950 - \text{year}}{8267}} - 1 \right] \cdot 1000 \text{ ‰} \quad (1)$$

where *T* is the conventional <sup>14</sup>C age (Stuiver and Polach 1977).

## RESULTS AND DISCUSSION

### Radiocarbon Dating Results

In Figure 1 and Table 2, the results from the <sup>14</sup>C analysis are shown for both the pasture and maize soils and SOM fractions (0–30 cm and 60–80 cm layers only). <sup>13</sup>C measurements are also shown (Table 2). Both the <sup>14</sup>C content and the corrected age are given. In general, the <sup>14</sup>C content in the pasture topsoil decreased in fraction size and increased in fraction density. This confirmed the hypothesis that the resistance to mineralization increased due to humification and association with sand, silt, or clay particles (Hassink 1995a). It also shows that the method used here physically separates fractions with a distinctly different character in terms of stability. The <sup>14</sup>C content of both the roots and >250 μm fraction in the pasture and maize topsoil did not differ much. The average fraction age in the maize plot (0–30 cm) was slightly lower compared to the fraction age in the pasture plot. This seems to be in contrast with previous results from Hsieh (1994) who showed that the average soil carbon age in undisturbed pasture plots was lower compared to cultivated plots due to a loss of young, macro-aggregate bound carbon upon cultivation. We observed also that the amount of carbon stored in macro-aggregates (>150 μm) decreased upon transformation of pasture to maize. However, in this study, 26 yr of maize cropping resulted in the input of young maize-derived carbon into all fractions. In another study (Römken, van der Plicht and Hassink ms.) it was shown that the incorporation (in percent of the total amount of carbon stored in each fraction) of maize-derived carbon was largest in the >150 μm fractions: after 26 yr of maize cropping, the contribution of maize-derived carbon ranged from

11% in the <20  $\mu\text{m}$ , to 90% in the >250  $\mu\text{m}$ -light fraction. This was calculated based on the differences in the  $\delta^{13}\text{C}$  value of the corresponding fractions in the maize and the pasture plots (also shown in Table 2). Assuming that the maize-derived carbon is of modern age, we used the distribution of pasture vs. maize-derived carbon to estimate the current age in each fraction in the maize soil according to

$$^{14}\text{a}(\text{C}_{\text{fraction-i-maize}}) = \% \text{-pasture} \times ^{14}\text{a}(\text{C}_{\text{pasture-fraction-i}}) + \% \text{-maize} \times ^{14}\text{a}(\text{C}_{\text{modern}}) \quad (2)$$

with:

$^{14}\text{a}(\text{C}_{\text{fraction-i}})$ :	$\text{a}^{14}\text{C}$ in fraction i (in pMC)
$^{14}\text{a}(\text{C}_{\text{pasture-fraction-i}})$ :	$\text{a}^{14}\text{C}$ in fraction i in the pasture soil (in pMC)
%-pasture:	percentage of pasture derived carbon in fraction i
%-maize:	percentage of maize derived carbon in fraction i
$^{14}\text{a}(\text{C}_{\text{modern}})$ :	activity of $^{14}\text{C}$ in recent carbon (in pMC).

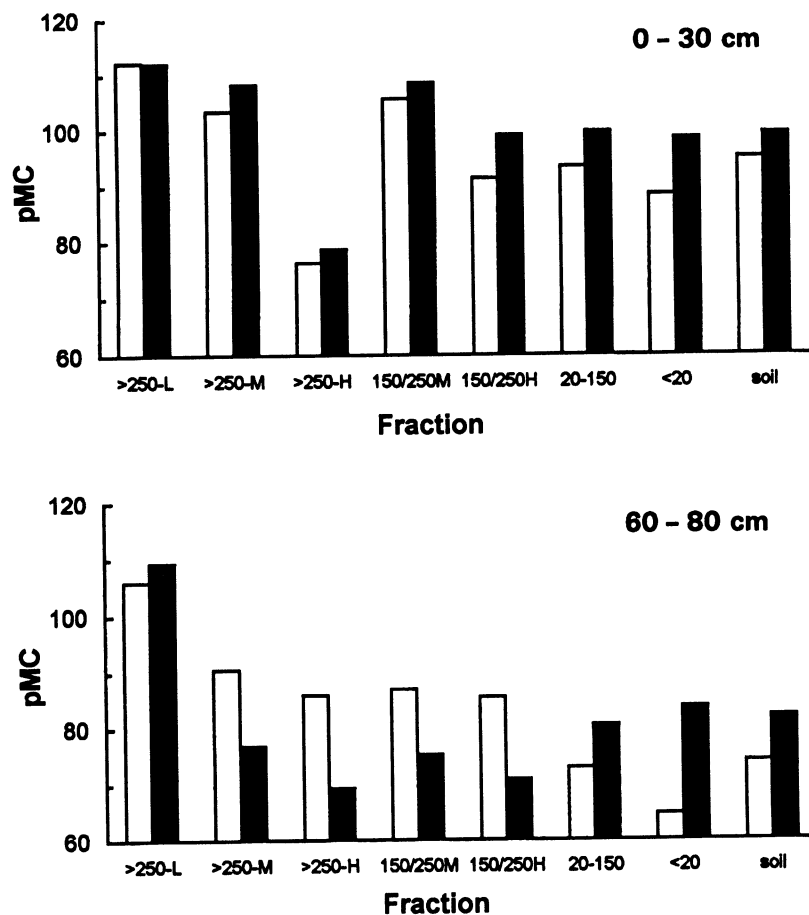


Fig. 1.  $^{14}\text{C}$  content in size- and density-separated SOM fractions and whole soils in pasture (open bars) and maize plots (closed bars)

TABLE 2A. AMS Data: Whole Soil Analysis

Depth (cm)	Pasture				Maize			
	<sup>14</sup> a (pMC)	<sup>14</sup> Δ (‰)	Age (yr)	δ <sup>13</sup> C (‰)	<sup>14</sup> a (pMC)	<sup>14</sup> Δ (‰)	Age (yr)	δ <sup>13</sup> C (‰)
0–30	95.15	-54	444	-27.63	99.42	-11	42	-25.88
60–80	74.07	-263	2456	-27.73	82.14	-183	1625	-27.48

TABLE 2B. AMS Data: Topsoil Samples Fractionated SOM (0–30 cm)

Fraction	Pasture				Maize			
	<sup>14</sup> a (pMC)	<sup>14</sup> Δ (‰)	Age (yr)	δ <sup>13</sup> C (‰)	<sup>14</sup> a (pMC)	<sup>14</sup> Δ (‰)	Age (yr)	δ <sup>13</sup> C (‰)
Roots	113.16	+125	3	-28.78	--	--	--	-13.00
>250-L	112.22	+116	2	-28.23	112.04	+114	3	-16.25
>250-M	103.41	+28	41	-27.48	108.28	+77	40	-21.52
>250-H	76.26	-242	2222	-27.30	78.73	-217	1966	-24.88
150/250-M	105.58	+50	41	-27.78	108.57	+80	38/2	-21.77
150/250-H	91.31	-92	775	-27.23	99.11	-14	42	-24.26
20/150	93.51	-70	584	-27.66	99.74	-8	42	-25.43
<20	88.34	-121	1041	-27.59	98.71	-18	43	-26.35

TABLE 2C. AMS Data: Subsoil Samples Fractionated SOM (60–80 cm)

Fraction	Pasture				Maize			
	<sup>14</sup> a (pMC)	<sup>14</sup> Δ (‰)	Age (yr)	δ <sup>13</sup> C (‰)	<sup>14</sup> a (pMC)	<sup>14</sup> Δ (‰)	Age (yr)	δ <sup>13</sup> C (‰)
Roots	--	--	--	--	--	--	--	--
>250-L	106.02	+54	40	-28.64	109.35	+88	2	-16.89
>250-M	90.41	-101	855	-28.04	76.82	-236	2163	-27.08
>250-H	85.98	-145	1258	-27.99	69.26	-311	2996	-27.30
150/250-M	86.98	-135	1166	-28.09	75.27	-251	2327	-27.61
150/250-H	85.56	-149	1298	-27.93	70.90	-295	2808	-27.50
20/150	72.89	-275	2585	-27.98	80.48	-200	1789	-27.34
<20	64.56	-358	3560	-27.50	83.83	-166	1462	-27.60

In Table 3, the results for each fraction as well as for the whole soil are shown using a value of 112 (pMC) for modern carbon. The predicted <sup>14</sup>C values agree well with the measured values, indicating that dilution of the original pasture-derived carbon with modern maize-derived carbon is the main reason for the “rejuvenation” of the coarse fractions (with the exception of the >250-heavy fraction, see below). The estimated <sup>14</sup>a levels for the finest fractions (<20 μm, 20–150 μm) are underestimated. This is probably because these fractions contain “protected” carbon that was incorporated into the fine fractions a couple of decades ago. At that time, natural <sup>14</sup>C levels exceeded those of today (<sup>14</sup>a >125 pMC), which resulted in a higher <sup>14</sup>a level in the 20–150 μm and <20 μm fractions. The measured <sup>14</sup>a levels in both the 15–250 μm-heavy and the 20–150 μm fraction can be predicted well using a value of 125 for <sup>14</sup>a (C<sub>modern</sub>). However, in order to explain the <sup>14</sup>a level in the <20-μm fraction a value of 200 for <sup>14</sup>a (C<sub>modern</sub>) is needed, which seems excessively high. This indicates that the measured <sup>14</sup>C level in the <20 μm fraction in the maize plot cannot be explained by dilution with modern carbon alone. A striking feature is also the very high age (ca. 2 ka) of the coarse-heavy fraction (>250

$\mu\text{m}$ , density  $>1.37 \text{ g cm}^{-3}$ ). The low carbon content in this fraction ( $<10 \text{ g OM per } 100 \text{ g fraction}$ , data not shown here) indicates that carbon in the  $>250\text{-}\mu\text{m}$ , heavy fraction is highly mineral-associated; this is in contrast to other density fractions in the  $>250\text{-}\mu\text{m}$  class that contained between 85 and 95 g OM per 100 g fraction (Römkens, van der Plicht and Hassink ms.).

TABLE 3. Measured vs. Calculated Activity Levels ( $^{14}\text{a}$  in pMC) in the Maize Plot (0–30 cm)

Fraction	Measured $^{14}\text{a}$	Calculated $^{14}\text{a}$	
		$^{14}\text{a}(\text{C}_{\text{input}}) = 112$	$^{14}\text{a}(\text{C}_{\text{input}}) = 125$
>250-L	112.04	112.02	--
>250-M	108.28	107.53	--
>250-H	78.73	83.41	--
150/250-M	108.57	108.60	--
150/250-H	99.11	96.28	99.40
20/150	99.74	96.84	99.18
<20	98.71	90.71	92.01

The close association between soil particles and organic material probably protects the carbon in the  $>250\text{-}\mu\text{m}$  heavy fraction from microbial degradation. However, the land use history of the study area may have contributed to this (potential) artifact as well: from 1500 to *ca.* 1900 AD the area was covered with heath ( $\text{C}_3$  species), and regular (natural and human-induced) burning was common. This resulted in the formation of charcoal, which is likely to be present in the  $>250 \mu\text{m}$  (heavy) fraction. Skjemstad, Le Feuvre and Prebble (1990) assumed charcoal to be responsible for the low  $^{13}\text{C}$  content of the  $<1.6 \text{ g cm}^{-3}$  fraction; it is therefore not unlikely that charcoal-carbon is depleted in  $^{14}\text{C}$  as well. Dilution of the total carbon with charcoal therefore will lead to an overestimation of the absolute age of a certain fraction. In the pasture soil, mineral-associated and fine-heavy SOM fractions were old (775–1041 yr) compared to the coarser fractions (3–41 yr), which points to advanced stabilization of the organic material in the finest fractions. However, in the maize top soil, the average age in both the 20–150  $\mu\text{m}$  and  $<20 \mu\text{m}$  fractions was much lower (*ca.* 40 yr). This is partly due to the incorporation of maize-derived (young) SOM, as was explained earlier. However, the young age of the  $<20 \mu\text{m}$  fraction remains unexplained. Possibly, a significant part of the carbon in the  $<20 \mu\text{m}$  fraction in the maize soil originates from larger aggregates (*e.g.*, the  $>250 \mu\text{m}$  fractions), which are physically degraded upon cultivation (maize cropping) after which the carbon becomes associated with smaller soil particles (*e.g.*, silt and clay particles).

Apart from carbon redistribution due to agricultural activities, manure application may also explain part of this apparent rejuvenation of the carbon content in the  $<20 \mu\text{m}$  fraction in the maize plots. But this explanation seems unlikely since the  $^{14}\text{C}$  enrichment of the finest fractions was not observed in the pasture soil which received similar loads of manure. From previous research on the stability of mineral-associated soil organic carbon, it was concluded that carbon associated with the fine fractions (*e.g.*, the  $<20 \mu\text{m}$ ) can be considered as “stable” or “inert” with ages ranging from 853 (Hsieh 1992) to  $>1 \text{ ka}$  (Balesdent, Wagner and Mariotti 1988). Buyanovsky, Aslam and Wagner (1994) observed a difference between the silt-bound SOM (400 yr) and clay-bound SOM (900 yr). In general, these findings agree well with our data on pasture soil, which indicates that, under stable conditions (*e.g.*, continuous pasture), carbon dynamics mainly take place in macro-aggregates ( $>150 \mu\text{m}$ ). Under stable conditions, redistribution of carbon towards the finer fractions (20–150  $\mu\text{m}$  and  $<20 \mu\text{m}$ ) will take several hundred years. However, in case of maize cropping, young,  $^{14}\text{C}$  enriched, pasture-derived carbon is rapidly transferred to both the 20–150  $\mu\text{m}$  and the  $<20 \mu\text{m}$  fractions, most

likely as a result of physical disintegration of the carbon originally stored in >150 μm fractions. The distinctly different ages in the finest fractions in the maize plot compared to the pasture plot indicate that the use of size fractions as a way to discriminate between “active” and “passive” carbon pools in soils under intensive cultivation seems to be invalid.

#### Transport of Colloidal Organic Material as Indicated by <sup>13</sup>C and <sup>14</sup>C Data

In a previous study, a significant increase in the amount of carbon stored in the finest fractions in the maize plots (20–150 μm and <20 μm) was observed (see also Table 1), which was attributed to colloidal displacement transport of carbon (Römkens, van der Plicht and Hassink ms.). This hypothesis was based on the distribution of carbon among fractions, and on <sup>13</sup>C isotope ratios of fractionated SOM, which indicated that the carbon stored in the finest fractions in the maize plots was derived mainly from C<sub>3</sub> species (pasture species). The <sup>14</sup>C data of the maize plots presented here reveal that carbon stored in the <150 μm fractions in the topsoil and the lower part of the soil is of recent origin (topsoil) or of a more recent age (subsoil) than in similar fractions in the pasture plots. Both the stable isotope and the <sup>14</sup>C analysis, therefore, imply that the carbon attached to the clay- and silt particles in the maize subsoil originates from other soil horizons. Vertical displacement of colloidal carbon that was destabilized upon cultivation from the topsoil still remains a plausible explanation for this phenomenon. A second possibility includes the vertical displacement of manure-derived carbon, but then it remains questionable why this did not occur in the pasture soils as well since all plots received similar loads of manure. The observed colloidal transport of carbon, whether from manure or pasture, indicates that vertical displacement of carbon can be enhanced due to cultivation. Especially in areas with high manure application rates such as those with intensive animal husbandry, the mechanism of colloidal transport of carbon may lead to facilitated transport of contaminants attached to SOM (e.g., trace metals or pesticides).

#### CONCLUSION

Application of a size and density SOM fractionation method resulted in the separation of the total SOM pool in distinctly different fractions representing different stages of humification. Maize cropping induced significant changes in the quantity and quality of the fractions distinguished. It also shows that a combination of SOM fractionation and <sup>13</sup>C and <sup>14</sup>C analysis yields a better insight in SOM dynamics under field conditions. It appeared that the dynamics in the so-called stable soil carbon pool in cultivated plots are much larger than previously believed. This stresses the potential role of the application of natural <sup>14</sup>C values instead of the addition of <sup>14</sup>C-enriched material. This is made possible by the AMS technique, which allows for measurements of milligram-sized samples. Especially carbon transfer between “pools” with a long turnover time cannot be studied in short-term experiments as a result of the slow incorporation of carbon into these pools. The observed transfer of young, pasture-derived carbon into the < 20 μm fractions in the maize-cultivated soil due to cultivation is of importance with respect to the behavior of contaminants: due to a preferential attachment of trace metals and pesticides onto SOM, an accelerated downward SOM-transport through the soil column can induce a faster downward transport of contaminants than would be expected from transport controlled by water flow only. The distinctly different ages of various fractions stress that it is necessary to use a fractionation method if attempting to understand SOM dynamics in cases of land use changes. The results clearly show that the application of average carbon turnover rates in simulation models cannot realistically represent SOM dynamics. Models predicting the development of the soil organic carbon with time based on lumped parameters (Jenkinson and Coleman 1994) may fit experimental data but are not adequate in giving insight in the contribution of the different components within the total SOM pool. Especially in the case of land use changes where many aspects of the



organic matter chemistry affect the behavior of contaminants, one must estimate the change in the total SOM pool, but especially the dynamics in the more reactive fractions since they will mainly control both the microbial activity as well as the binding of contaminants to the solid phase.

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