



Effects of mineral characteristics on content, composition, and stability of organic matter fractions separated from seven forest topsoils of different pedogenesis

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ABSTRACT

Mineral topsoils possess large organic carbon (OC) contents but there is only limited knowledge on the mechanisms controlling the preservation of organic matter (OM) against microbial decay. Samples were taken from the uppermost mineral topsoil horizon (0 to 5 cm) of seven sites under mature deciduous forest showing OC contents between 69 and 164 g kg⁻¹ and a wide range in mineral characteristics. At first, organic particles and the water-extractable OM were removed from the soil samples. Thereafter, Na-pyrophosphate extractable organic matter (OM(PY)), assumed to be indicative for OM bound via cation mediated interactions, and the OM remaining in the extraction residue (OM(ER)), supposed to be indicative for OM occluded in mechanically highly stable micro-aggregates, were sequentially separated and quantified. The composition of OM(PY) and OM(ER) was analyzed by FTIR and their stability by ¹⁴C measurements. The OC remaining in the extraction residues accounted for 38 to 59% of the bulk soil OC (SOC) suggesting a much larger relevance of OM(ER) for the OM dynamic in the analyzed soils as compared with OM(PY) that accounted for 1.6 to 7.5% of the SOC. The FTIR analyses revealed a lower relative proportion of C=O groups in OM(ER) compared to OM(PY) indicating differences in the degree of microbial processing between these fractions. Correlation analyses suggest an increase in the stability of OM(PY) with the soil pH and contents of Na-pyrophosphate soluble Fe, Al, and Mg and an increase in the stability of OM(ER) with the soil pH and the contents of clay and oxalate-soluble Fe and Al. Despite the detected influence of soil mineral characteristics on the turnover of OM(PY) and OM(ER), the $\Delta^{14}\text{C}$ signatures indicated mean residence times less than 100 years. The presence of less stabilized OM in these fractions can be derived from methodological uncertainties and/or the fast cycling compartment of mineral-associated OM.

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1. Introduction

Approximately 50% of the estimated global soil organic carbon (SOC) stock (1500 Pg in 0–100 cm depth) are stored in forest soils, although only 40% of the soil-covered land surface is forested (Jobbágy and Jackson, 2000). The afforestation of arable soils and improved forest management have been proposed as options for C-sequestration but there is conflicting evidence on the efficacy of these measures (Paul et al., 2002; Jandl et al., 2007; Nave et al., 2013). Gains in C stocks that can be related to changes in land use or management have been often found to be limited to the organic and uppermost mineral topsoil horizons (Vesterdal et al., 2002; DeGryze et al., 2004; Hedde et al., 2008; Nave et al., 2013) but such soil horizons may quickly turn from C sinks into C sources under changing site conditions (Jandl et al., 2007).

In contrast to the organic litter layers, the uppermost mineral topsoil horizon possesses less organic than inorganic phases, which are crucial for the protection of organic matter (OM) against microbial decomposition by occlusion of OM within aggregates and the formation of organo-mineral/cation interactions (Kögel-Knabner et al., 2008; Schmidt et al., 2011). Interactions between organic and inorganic soil constituents comprise several mechanisms, such as the complexation of OM at surfaces of Fe- and Al-(oxy)hydroxides via ligand exchange reactions and the bridging of OM to negatively charged mineral surfaces via polyvalent cations (i.e., OM-cation-mineral) (e.g., Kögel-Knabner et al., 2008) as well as the cation-mediated crosslinking of OM (i.e., -OM-cation-OM-) and the formation of metal-organic complexes (i.e., chelates, OM-cation) (Senesi, 1992; Senesi and Loffredo, 2005; Kunhi Mouvenchery et al., 2012).

For a more acidic soil environment, ligand exchange reactions at surfaces of Fe- and Al-oxides have been hypothesized to be the dominating organo-mineral interaction (Parfitt et al., 1977; Gu et al., 1994). Close positive correlations between contents of stabilized OM and poorly

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crystalline Fe and Al phases suggest an indirect evidence for this hypothesis for acidic forest subsoils (Eusterhues et al., 2005; Kleber et al., 2005; Mikutta et al., 2006). However, such relationships have not been found for forest mineral topsoils, which is partially explained by the relatively larger C loadings on Fe oxides in SOC-rich forest topsoils (Eusterhues et al., 2005). Kaiser et al. (2012b) argued that OM in acidic forest topsoils might be bound to mineral surfaces in a multilayer architecture, referring to the conceptual model of Kleber et al. (2007), and Hobbie et al. (2007) found C mineralization rates to be closely negatively correlated to quantities of exchangeable Al and Fe cations. They speculated that Al^{3+} and Fe^{3+} ions in soil may promote SOM flocculation and bridging processes resulting in reduced C mineralization. Such cation-mediated processes of SOM stabilization in forest topsoils have also been hypothesized by other authors (Mueller et al., 2012; Clarholm and Skjyllberg, 2013). However, the relative importance of the above suggested mechanisms remains unclear.

To better understand soil C dynamics in forest ecosystems and to assess potential changes caused by disturbances or land use change an improved knowledge on the OM stabilization mechanisms in mineral topsoils is necessary. Selectively separated OM fractions that are indicative for different types of OM protection (e.g., OM–mineral interactions, OM occlusion in stable aggregates) may help to determine the extent of the above-mentioned mechanisms for OM protection against microbial decomposition. The alkaline Na-pyrophosphate ($Na_4P_2O_7$) solution is expected to extract (i) OM bound to mineral surfaces via cation bridging (OM–cation–mineral) (Bremner and Lees, 1949; Edwards and Bremner, 1967; Wattel-Koekkoek et al., 2003), (ii) OM crosslinked via polyvalent cations (OM–cation–OM) (Yang et al., 2001), and (iii) OM bound in metal-organic complexes (OM–cation) (Bremner et al., 1946; Masiello et al., 2004). Alkaline $Na_4P_2O_7$ solution has also been shown to extract OM sorbed to synthetic Al- and Fe-oxides (Kaiser and Zech, 1996) and has been proposed to separate OM bound to mineral surfaces via ligand exchange reactions in acidic forest subsoils (Kleber et al., 2005).

However, the separation of fractions indicative for mineral-associated OM from soil requires the isolation of interfering fractions indicative for not mineral-associated OM. Accordingly, Kaiser et al. (2011, 2012b) employed a sequential fractionation procedure to separate less mineral-associated physically uncomplexed and macro- as well as micro-aggregate occluded organic particles and water-extractable OM prior to the extraction of mineral-associated OM using $Na_4P_2O_7$ solution. The OM remaining in the residue after Na-pyrophosphate extraction is supposed to be mainly occluded in silt- and clay-sized micro-aggregates of high mechanical stability preventing the occluded OM from separation (Kayler et al., 2011).

The objectives of this paper were (i) to quantify the amounts of sequentially Na-pyrophosphate extractable OM and of the OM remaining in the extraction residue, (ii) to analyze the composition and turnover of these two fractions by FTIR spectroscopy and ^{14}C analysis, and (iii) to analyze the correlation between the amount, composition, and stability of these OM fractions and soil mineral characteristics such as the contents of oxides and cations.

2. Material and methods

2.1. Site description and soil sampling

Seven deciduous forest sites in Germany featuring different soil types (Table 1) and covering a wide range in soil mineral characteristics shown to be relevant for OM stabilization (Table 2) were selected. The sites have been under continuous forest land use for minimum periods between 71 and 150 years (Table 1). Soil types, geographical location and climatic data for the selected sites are summarized in Table 1. Soils were sampled in 2005 and 2006. At each selected site one soil pit was excavated for soil classification according to the WRB (2006). Ten sampling points around each soil pit were chosen, the organic layers removed, and the mineral surface horizon was sampled using a cylindrical auger. For each site, these ten pseudo-replicates were bulked, air-dried, sieved to pass 2 mm and stored until the analyses.

2.2. Bulk soil analyses

Prior to all analyses of C ratios of the bulk soil samples (<2 mm), coarse organic particles were removed by electrostatic attraction (Kaiser et al., 2009). Bulk soil pH ($CaCl_2$, 0.01 M), the contents of organic C as well as the contents of clay, silt, and sand size particles were determined as described in Kaiser et al. (2011). Furthermore, the contents of oxalate-soluble Fe and Al were used to assess the contents of poorly crystalline Fe- and Al-oxides (Fe_{ox} , Al_{ox}) (Rasmussen et al., 2005) and were determined according to Kaiser et al. (2011). The bulk soil cation exchange capacity (CEC) as well as the contents of exchangeable Ca (Ca_{ex}) and Mg (Mg_{ex}) cations were determined according to Kaiser et al. (2008).

2.3. Sequential separation of the Na-pyrophosphate extractable organic matter (OM(PY)) and the organic matter remaining in the extraction residue (OM(ER))

- (i) In a first step, physically uncomplexed, macro- and micro-aggregate occluded organic particles and water-extractable OM

Table 1
Soil type (WRB, 2006), parent material, mean annual precipitation (P) and temperature (T), depth ranges of the analyzed forest mineral surface soil horizons (Depth), and periods of minimum constant forest land use (Land use) of the study sites and contents of soil organic carbon (SOC) and clay, pH values, cation exchange capacities (CEC), contents of exchangeable cations (Ca_{ex} , Mg_{ex}) and of oxalate-extractable Fe and Al (Fe_{ox} , Al_{ox}) for the analyzed forest mineral surface soils. Values for the soil chemical and mineral parameters are means calculated from two analytical replicates.

Soil type	Parent material		P ^a	T ^a	Depth	Land use	SOC		pH	CEC	Ca _{ex}	Mg _{ex}	Fe _{ox}	Al _{ox}
			(mm)	(°C)	(cm)	(yr.)	(g kg ⁻¹)	clay	(CaCl ₂)	(cmol _c kg ⁻¹)			(g kg ⁻¹)	
Colluvic Regosol	CR	Colluvial material	532 ^b	8.3 ^b	0–3 to 4	150	69	76	3.5	19	3.5	0.5	1.6	0.6
Ortsteinic Podzol	OP	Pleistocene sand	784 ^h	8.7 ^h	0–3 to 5	71	94	26	3.6	30	4.7	1.3	0.5	0.2
Albic Luvisol	AL	Till	536 ^c	7.9 ^c	0–1 to 2	150	164	88	4.3	52	13.2	14.8	1.5	0.6
Haplic Stagnosol	HSt	Till	566 ^d	8.0 ^d	0–2 to 3	119	79	97	4.5	22	7.7	1.8	1.6	0.8
Haplic Luvisol	HL	Loess	577 ^e	9.4 ^e	0–1 to 2	150	110	153	4.7	46	16.7	3.1	3.7	0.9
Haplic Cambisol	HC	Limestone	1069 ^f	6.8 ^f	0–0.5 to 1	105	122	372	5.5	60	32	9.2	3.1	2.2
Vertic Stagnosol	VS	Clay stone	1024 ^g	8.8 ^g	0–1 to 3	150	103	513	6.1	52	36.8	2.4	8.3	1.7

^a Climatic parameters are mean values for different timescales recorded from the following climate stations.

^b Angermünde: 1961–1990.

^c Neubrandenburg: 1961–1990.

^d Greifswald: 1961–1990.

^e Würzburg: 1971–2000.

^f Geislingen-Stötten: 1961–1990.

^g Schwäbisch Gmünd: 1961–1990.

^h Soltau: 1971–2000.

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