

Soil organic carbon fractionation for improving agricultural soil quality assessment – a case study in Southern Belgium (Wallonia)

Sylvain Trigalet ⁽¹⁾, Caroline Chartin ⁽¹⁾, Inken Kruger ⁽²⁾, Monique Carnol ⁽²⁾,
Kristof Van Oost ^(1,3), Bas van Wesemael ⁽¹⁾

⁽¹⁾ Université catholique de Louvain. Georges Lemaître Centre for Earth and Climate Research. Earth and Life Institute. Place Louis Pasteur, 3. BE-1348 Louvain-la-Neuve (Belgium). E-mail: sylvain.trigalet@uclouvain.be

⁽²⁾ Université de Liège. Laboratoire d'Écologie végétale et microbienne. Botanique B22. Boulevard du Rectorat, 27. BE-4000 Liège (Belgique).

⁽³⁾ FNRS Research Associate

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Description of the subject. The paper presents and discusses a method for fractionating bulk soil organic carbon (SOC) in meaningful SOC fractions to better assess SOC status and its related soil ecosystem functions.

Objectives. The objective is to perform an evaluation of ecosystem functions of soil organic matter at plot scale and compare it to the normal operative range of the local agro-ecological region.

Method. By separating carbon associated with clay and fine silt particles (stable carbon with slow turnover rate, $< 20 \mu\text{m}$) and carbon non-associated with this fraction (labile and intermediate carbon with higher turnover rates, $\geq 20 \mu\text{m}$), effects of management can be detected more efficiently at different scales.

Conclusions. Soil organic carbon fractions, used as proxies for soil ecosystem functions, can be helpful because they represent SOC functional pools. This paper proposes to apply fractionation on samples taken at plot and regional scale. It is therefore possible to establish a normal operative range for a specific agro-region for comparison with the values in individual plots. This allows drawing a baseline for SOC fractions status in a specific agricultural unit. This approach provides valuable information to study and evaluate the impact of agricultural management in the context of enhancing soil quality and functions.

Keywords. Soil organic matter, fractionation, rural areas, Belgium.

Améliorer le diagnostic de la qualité des sols agricoles wallons par un fractionnement du carbone organique

Description du sujet. L'article traite d'une méthodologie de fractionnement du carbone organique du sol qui permet d'affiner l'interprétation de son statut au sein de parcelles agricoles dans des unités d'espace rural en Wallonie.

Objectifs. L'objectif est de réaliser un diagnostic des fonctions écosystémiques de la matière organique à l'échelle de la parcelle et de le comparer à l'unité rurale correspondante.

Méthode. Le fractionnement physico (-chimique) d'échantillons de sols agricoles permet de distinguer des *turnovers* contrastés entre les fractions stables (carbone associé aux limons fins et argiles, $< 20 \mu\text{m}$) et labiles (carbone non associé à ces derniers, $\geq 20 \mu\text{m}$) et de comparer ces fractions avec des gammes de référence au sein de l'unité rurale correspondante.

Conclusions. Un diagnostic des fractions de carbone organique, utilisées comme proxys des fonctions écosystémiques de la matière organique, permet d'établir des gammes de référence pour différents districts d'espace rural en Wallonie. Sur base de ce référentiel, le statut d'une parcelle individuelle peut être analysé dans le cadre local et mettre en avant la nécessité d'améliorer la gestion agricole.

Mots-clés. Matière organique du sol, fractionnement, zone rurale, Belgique.

1. INTRODUCTION

1.1. Soil organic matter

Soil plays a crucial role in food security, climate change mitigation, and other essential ecosystem services (Smith et al., 2000). Hence, efficient management and adapted policies are required for the maintenance and improvement of soil quality (Stoate et al., 2009). Among others, Karlen et al. (1997) defined soil quality as “the capacity of a specific kind of soil to function, within natural or managed ecosystem boundaries, to sustain plant and animal productivity, maintain or enhance water and air quality, and support human health and habitation”. In order to measure the preservation and improvement of soil quality, quantifiable meaningful parameters for soil quality assessment have to be monitored by suitable and efficient Soil Monitoring Networks (SMN) (Arrouays et al., 2008).

Soil organic matter (SOM) has been recognized as one of the most important attributes in terms of “soil quality”. Soil organic matter represents all organic components in the soil including decayed plant and animal tissues, their partially decomposed products, and the soil biomass (Baldock et al., 2000). Soil organic matter composition is extremely complex because of the nature of the various inputs and their different stages of decomposition (Chenu et al., 2014). Soil organic matter plays several roles in agro-ecosystems such as:

- the regulation of CO₂ fluxes between the soil and the atmosphere;
- mineral reserve and soil fertility;
- soil structure and hydrological behavior;
- soil stability and its resistance against erosion and compaction;
- biodiversity of soils.

It has beneficial effects on soil biological, physical and chemical properties, which influence the productive capacity of agricultural soils (Sollins et al., 1996).

Yet, the loss of SOM resulting from conversion of native vegetation to farmland has been confirmed and is one of the best-documented ecosystem consequences of our agricultural activities (Paul et al., 1997). The exchange between terrestrial environments and atmosphere is profoundly altered and increased amounts of SOM are exposed to oxidation and are transferred to the atmosphere as CO₂. As indicated by Lal (2015), indiscriminate ploughing, residue removal, negative soil organic carbon (SOC) or nutrients budgets and extractive farming can trigger the decline of SOM and therefore soil quality. A degradation of the soil structure leads to crust formation and compaction that increase runoff accelerating erosion. Loss of nutrients, SOC and water from ecosystems occurs and

is followed by a decrease in agricultural efficiency, a loss of soil resilience, and the reduction of ecosystem services (Lal, 2015). However, improved management practices can rebuild SOM levels and help mitigate CO₂ emissions and increase soil quality (Paustian et al., 2000). Practices such as the improvement of tillage management and cropping systems, the management to increase vegetation cover and the efficient use of production inputs, *e.g.* nutrients and water can help restoring SOM (Follett, 2001).

1.2. Current assessment of SOC levels in agricultural soils of Wallonia

In Southern Belgium (Wallonia), there is a long history of agriculture dating back centuries to millennia and this has affected SOM levels (Van Oost et al., 2012). It is then necessary to properly diagnose SOM status in Walloon agricultural soils. Soil organic carbon (SOC), the principal component of SOM (*ca.* 58%, depending on edaphic conditions), is often used as one of the major indicators of soil quality (Pribyl, 2010). Therefore, total organic carbon (TOC) is monitored in SMN to assess the impact of management practices or land use changes on agro-ecosystems. Belgium has one of the oldest, dense and high quality geo-referenced soil databases worldwide: the National Soil Survey (NSS) (Goidts, 2009). Through a SMN in Wallonia (CARBOSOL), Goidts et al. (2009a) studied SOC changes between 1955 and 2005 in different agricultural regions in Wallonia. In another study, Chartin et al. (2015) resampled topsoil profiles from this soil database in the framework of the CARBOSOL SMN. These SMN focus on bulk SOC evolution in southern Belgium by providing a controlled baseline to assess the impact of specific agricultural practices on SOC content at the regional scale over decade. There are ten agricultural regions within Wallonia with similar agricultural practices (rotations and/or livestock) and yield potentials, both directly linked to soil types (*e.g.*, texture, stoniness, drainage) and climatic characteristics (Lettens et al., 2004). These units are used as reference in most of the reporting activities linked to agricultural activities and diagnosis of soil quality in Wallonia (Genot et al., 2007). Goidts et al. (2009a) used this stratification to assess main driving forces of SOC changes. For each elementary unit considered (*i.e.* the soil sample, the microsite, the field or the landscape), the standard deviation (σ) of TOC and bulk density and its corresponding coefficient of variation ($CV = 100 \times \sigma/\text{mean}$, %) were assessed as a measure for the extent of the uncertainty of SOC stocks estimations. The spatial scale and associated variability had an important impact on uncertainties of SOC stock values as shown by the increase of the CV across scales (from 4% at the sample level to 34% at

the landscape scale; Goidts et al., 2009b). This work highlighted the difficulty to assess SOC dynamics at a regional scale due to:

- the small magnitude of changes,
- the important spatial and temporal variability of SOC,
- the lack of detailed information on present and past management practices.

In addition to the spatial variability and magnitude of uncertainties, intra- and inter-annual variability of SOC can affect detection of significant changes over time.

The regional analysis of SOC in agricultural soils through SMN allows detecting sensitive agricultural regions characterized with low levels of SOC where a lack of organic carbon may influence ecosystem services (Genot et al., 2007; Goidts et al., 2009a). Hence, the risk of falling below a threshold of 2% of OM (*i.e.*, less than *ca.* 1.15% SOC depending on edaphic conditions), often suggested as critical limit, appears high in the northern half of Wallonia, especially in the cropland soils of the loam region (Loveland et al., 2003; Colinet et al., 2005; Genot et al., 2007; Goidts et al., 2009a) (**Figure 1**). This approach was efficient to assess main driving forces of SOC changes and to highlight threatened agricultural zone. Nevertheless, due to its heterogeneity and uncertainties in bulk SOC assessment at various scales, the diagnosis of soil quality remains problematic and difficult to implement.

1.3. From bulk SOC to SOC fractions

An important issue of SOC dynamics assessment is related to the intrinsic complexity of SOC. Bulk SOC is not a simple pool with a homogenous dynamic as it consists of heterogeneous mixture with different turnover rates and stabilities. The turnover rate of the different SOC compounds varies due to the complex interactions between biological, chemical, and physical processes in the soil. Soil organic carbon is distributed in three components (*i.e.*, non-occluded organic matter with different recalcitrance levels, organic matter trapped in aggregates and organic matter associated with the silt and clay particles) and the system is driven by different physical, chemical and biological mechanisms (Sollins et al., 1996).

Several soil fractionation approaches recommended isolating functional SOC components such as the labile, intermediate and stable SOC components (von Lützwow et al., 2007). Here, SOC fractions are used as proxies to better assess ecosystem services of soils such as available organic matter to maintain its fertility and carbon sequestration for climate change mitigation.

Soil organic carbon fraction composition and evolution are influenced by a range of controlling

factors such as geology, geomorphology, pedology, land use and land management or climate. As these factors have effects on SOC at different scales, they must be taken into account for the establishment of a Walloon soil monitoring program. As it is challenging to detect SOC changes over space and time, the use of SOC fractions that are more sensitive indicators for SOC dynamics can be meaningful. Existing fractionation methods are built according to different scopes regarding the mechanisms of SOC stabilization (Poeplau et al., 2013). They are usually classified in physical or chemical fractionation. Although there is a continuum of progressively decomposing organic compounds, fractionation techniques are often used to define and delineate various theoretically discrete pools (Lehmann et al., 2015).

This paper proposes a method to improve the diagnosis of SOC, and the detection of its changes, based on the analysis of SOC fractions (as indicators for SOC dynamics and different ecosystem services) rather than bulk SOC. It shows how SOC fractions can be used to establish a diagnosis of SOC status in a field or a series of discontinuous areas representing a combination of soil type and land use in a specific unit at regional scale.

2. MATERIALS AND METHODS

2.1. Study sites

Regional scale. The Loam region is the most important of the ten agricultural regions in terms of surface area, with fertile silt to silt loam soils (USDA, 1993). Croplands with rotations of cereals, potatoes and sugar beets prevail. The sites were selected in the CARBOSOL database. Cropland soils in the Loam region contain between 1.1 and 2% of SOC. Then, diagnosing the state of SOC fractions in the region is needed as the lack of organic carbon may influence ecosystem services. The fractionation method was applied to soils in 12 sites under cropland in the Loam region to estimate the variability of different fractions at the regional scale (**Figures 1 and 3**).

Long-term experiment. In a long term experiment in Liroux (Gembloux), Buysse et al. (2013) highlighted that the inter-annual SOC changes limit the detection of significant decadal changes, even in a crop rotation experiment with contrasting residue management practices (**Figure 1**). Among other management practices, most crop residues are removed after harvest apart from inevitable leftover residues in the residue export plots (RE). In farmyard manure plots (FYM), 30 - 60 t·ha⁻¹ of fresh manure are applied every four years before sowing the sugar beet crop. In residue

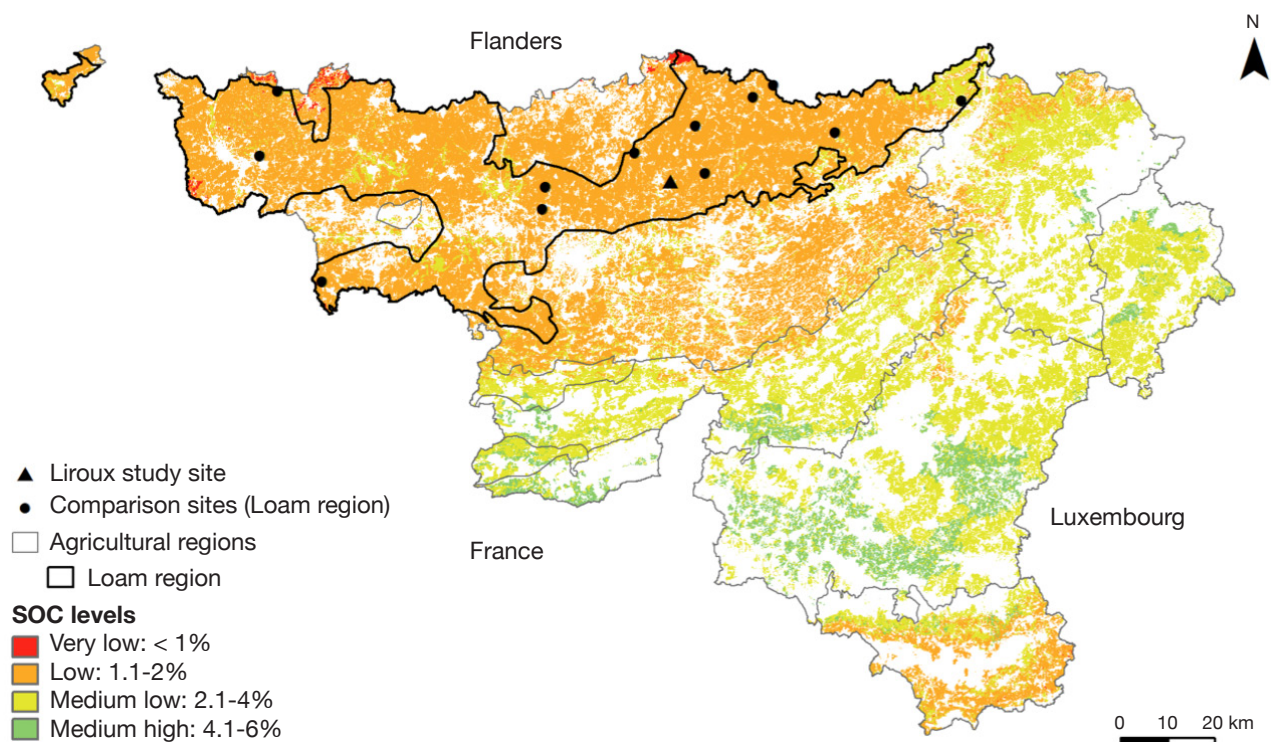


Figure 1. Soil organic carbon (SOC) content (%) in croplands and grasslands in Wallonia, based on the spatial analysis of SOC data collected by the Soil Monitoring Network CARBIOSOL — *Quantité de carbone organique (%) dans les cultures et prairies de Wallonie, basée sur l'analyse spatiale des données de carbone organique du réseau de monitoring du sol CARBIOSOL* (Chartin et al., 2015).

Sampling sites and delineations of the ten agricultural regions are overlaid — *Les sites d'échantillonnage de la présente étude et les régions agricoles de Wallonie sont superposés sur la carte.*

restitution plots (RR), crop residues are returned to the soil (+ straw) and green manure derived from intercrops (vetches or mustard) grown before sugar beet crop. Texture and mineralogy are relatively homogeneous between plots which allow comparison of stable SOC at the field scale (**Table 1**). Soil organic carbon fractions from the field scale were obtained in a previous study (Trigalet et al., 2014). Note that samples at regional scale were sampled in spring contrary to the samples collected in Liroux, which were taken in September–October. This could potentially affect the amount of labile carbon in samples.

2.2. Fractionation scheme

A combination of both physical and chemical approaches (**Figure 2**) to fractionate bulk SOC in meaningful fractions is proposed. This fractionation scheme is based on the particle-size distribution in which stable SOC is the organic carbon associated with the fine soil particles ($< 20 \mu\text{m}$) (Hassink, 1997). It refers to the SOC stabilized in organo-mineral associations which can be used as a proxy for carbon sequestration. Soil organic carbon in aggregates is used

as proxy for physically protected carbon (Six et al., 2002) and the amount of aggregates as a proxy for the soil structure state. In the complementary protocol, chemical and density separations better characterize functional SOC pools such as resistant SOC to microbial degradation made up of refractory and slow cycling carbon (rSOC) or a plant derived fraction, the particulate organic matter (POM) (Zimmermann et al., 2007). Free POM, non-occluded in aggregates, is primarily decomposed by micro-organisms providing nutrients for vegetation. Particulate organic matter is sensitive to management practices such as ploughing of crop residues or green manure. Measurable fractions, provided by the fractionation scheme, would allow improving the characterization of interactions between site conditions and SOC (Zimmermann et al., 2007).

Detailed applied protocol. Eighty ml of distilled water is added to 20 g of air-dried soil sieved at 2 mm and shaken horizontally 15 min ($175 \text{ revs}\cdot\text{min}^{-1}$) (**Figure 2**). Mechanical shaking disrupts a portion of macroaggregates but does not destroy microaggregates (Balesdent et al., 1998). The suspension is passed through a $50 \mu\text{m}$ sieve to remove aggregates (A),

Table 1. Characteristics of experimental plots in Liroux (mean \pm std. dev) — *Caractéristiques des parcelles expérimentales de Liroux (moyenne \pm écart-type).*

Type (ID)	Total organic carbon (g C·kg ⁻¹ soil)	Texture (%)		
	Bulk soil (0 - 2 mm)	Sand (50 μ m - 2 mm)	Coarse silt (20 - 50 μ m)	Clay and fine silt (< 20 μ m)
Residues export (RE)	9.2 \pm 1.4	7.0 \pm 0.9	52.5 \pm 2.5	40.5 \pm 2.0
Farmyard manure (FYM)	10.4 \pm 1.2	6.9 \pm 0.9	52.4 \pm 1.7	40.7 \pm 2.2
Residues restitution (RR)	10.1 \pm 0.8	6.9 \pm 0.4	52.2 \pm 2.1	40.9 \pm 2.2

For each management type, 6 replicate plots from which composite samples (consisting of 8 to 12 cores) were taken — *Pour chaque type de gestion agricole, 6 parcelles ont été utilisées comme répétitions dans lesquelles on a prélevé 8 à 12 échantillons rassemblés en un échantillon par parcelle.*

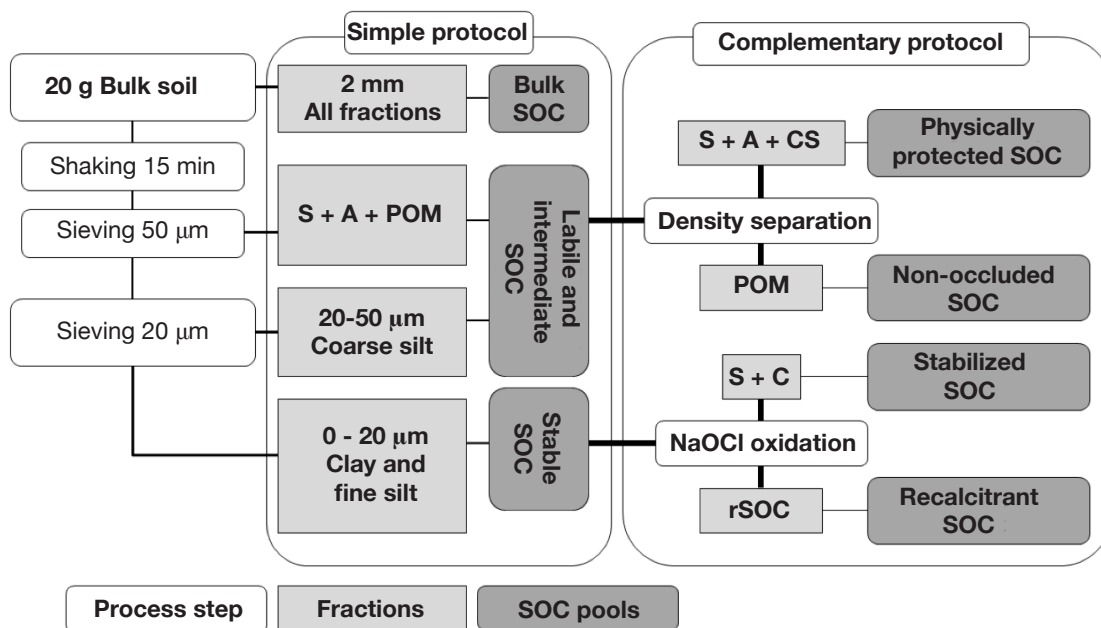


Figure 2. Fractionation scheme. Simple protocol used in the present study case: physical fractionation by wet sieving. In the complementary protocol (not done here), chemical (NaOCl oxidation) and density fractionation (heavy liquid separation) provide additional fractions used as proxies for conceptual functional pools — *Schéma de fractionnement. Protocole de base utilisé dans l'étude de cas : fractionnement physique par tamisage à l'eau. Dans le protocole complémentaire, des fractionnements chimiques (oxydation NaOCl) ou par densité (séparation avec une solution dense liquide) fournissent des fractions supplémentaires utilisées comme proxys des compartiments conceptuels de carbone organique.*

POM: particulate organic matter — *matière organique particulaire*; S + C: soil organic carbon bound to silt and clay — *carbone organique lié aux limons fins et argiles*; S + A: soil organic carbon attached to sand particles or occluded in aggregates — *carbone organique compris dans les sables ou en agrégats*; CS: coarse silt — *limons grossiers*; rSOC: chemically resistant fraction of carbon — *fraction de carbone chimiquement résistante*; SOC: soil organic carbon — *carbone organique du sol.*

sand (S) and free particulate organic matter (POM) in order to prevent carbon transfer to smallest fractions during sieving. Indeed, the use of an aerosol to ease sieving at 20 μ m may destroy additional aggregates and release POM trapped inside them. The coarse fraction (> 50 μ m) is dried at 50 °C, weighed and stored at room temperature. The remaining suspension is passed through a 20 μ m sieve with the help of an aerosol (Zimmermann et al., 2007) and the < 20 μ m

and 20-50 μ m fractions are dried at 50 °C, weighed and stored at room temperature. As 1 or 2 l of distilled water may be used for wet sieving, centrifuging the < 20 μ m suspension may speed up the drying step. When 3,000 ml of water were used for wet sieving, 10 min of centrifugation at 2,000 g apparently sufficed to separate the solid and the liquid phase (Poeplau et al., 2013). The 20-50 μ m fraction (coarse silt) can be mixed with the > 50 μ m fraction in order to get only

2 fractions reducing the SOC measurements after fractionation. The $\geq 20 \mu\text{m}$ fraction contains some C associated with coarse silt particles, free particulate organic matter (POM) and C in aggregates (intra-POM and C bound to silt and clay in aggregates). SOC content is the difference between the SOC in bulk soil and the C in $< 20 \mu\text{m}$ fraction (Hassink, 1997).

Complementary protocol. In order to get additional relevant proxies of functional pools, a short complementary protocol is presented (**Figure 2**, on the right). It was not applied to soil samples in the present case study but can help to better characterize SOC status. The separation of free particulate organic matter (POM) from coarse silt, sand and aggregates (S + A) is implemented by a density separation which is done by a heavy liquid *i.e.* sodium polytungstate at $2 \text{ g}\cdot\text{cm}^{-3}$ (Poeplau et al., 2013). Soil organic carbon concentration in cleared-POM $\geq 20 \mu\text{m}$ (CS + S + A) is used as a proxy for physical protection of organic matter (Six et al., 2002). Moreover, the proportion of aggregation in soil provides information on soil structure and soil stability.

The separation of resistant SOC (rSOC) and SOC bound to the S + C fraction by NaOCl oxidation can also be applied following Zimmermann et al. (2007). Here, NaOCl oxidation mimics, to a certain extent, the biological decomposition of organic matter.

2.3. SOC analyses

Soil organic carbon contained in fractions and bulk soil is determined using a Variomax dry combustion CN Analyzer (Elementar Analysensystem GmbH, Germany). The presence of inorganic C (IC) is tested by HCl (3%) and corrected for, if detected, following the analysis by modified pressure-calimeter method (Sherrod et al., 2002). Note that this method may not be appropriate for highly carbonated soils as the confidence on OC calculated by difference between total C and inorganic C might be too low for the OC objective. Particulate organic matter is assumed as the difference between bulk SOC and the sum of OC in all other fractions (rSOC, S + C, S + A).

3. CASE STUDY AND PRELIMINARY RESULTS

3.1. Comparison of SOC fractions at regional and field scales

Trigalet et al. (2014) showed that removing the inherent variability of the $\geq 20 \mu\text{m}$ fraction on SOC estimations allows the detection of significant changes

in stable carbon over decades between different management practices. In 1970, after 11 years of contrasted C inputs in the Liroux experiment, carbon content in the $< 20 \mu\text{m}$ fraction was not different between plots. Nevertheless, due to higher C inputs, progressively stabilized in FYM and RR plots, carbon associated with fine silt and clay was higher in 2012 (**Figure 3**). Carbon associated with clay and fine silt was an efficient indicator to assess decadal changes in contrasting management practices. Higher variability of $\geq 20 \mu\text{m}$ concentrations reduced the detection of differences between management practices.

Total and labile carbon concentrations were lower in Liroux compared to the 12 sites. Stable SOC shows strong variability among the 12 sites. This may be explained by the contrasting proportion of clay and fine silt mass in soil sample between sites which ranges from *ca.* 20 to 45%; SOC content in $< 20 \mu\text{m}$ fraction ($\text{g C}_{20\mu\text{m}}\cdot\text{kg}^{-1}$ soil) being positively correlated to the clay and fine silt content ($r^2 = 0.48$, $p < 0.05$) for those 12 sites. At the field scale, it was possible to detect differences between management practices due to a homogenous texture across the studied plots. This indicates the need to insert texture variability in our regional analysis or to consider other pedologic and geologic factors which may influence SOC dynamics at the local scale.

4. DISCUSSION

4.1. Refining regional scale analysis

As indicated by Legrain et al. (2011), despite the small size of its territory, Wallonia shows a varied and contrasted geopedological context. Therefore, the identification of agricultural units should be informed by the spatial heterogeneity of geopedological conditions. Contrary to the traditional ten agricultural regions, Legrain et al. (2011) used a finer stratification of the Walloon territory where landscape, land use, geology and pedology are combined in different units – the rural space units (RSU) – at different levels comprising 24 districts (Rural Space District - RSD) and 196 sectors (**Figure 4**). As the delineation of RSD takes into account the physical environment, the stratification should help to better assess the link between SOC fractions dynamics and soil properties for specific agricultural practices. Within these units, pedogenic processes are, to a certain extent, rather homogenous compared to the ten extensive agricultural regions. At this stratified spatial scale, we assume that the effect of geopedogenic factors is comparable within a specific RSD. This allows to link SOC fraction status more directly to management practices and land use.

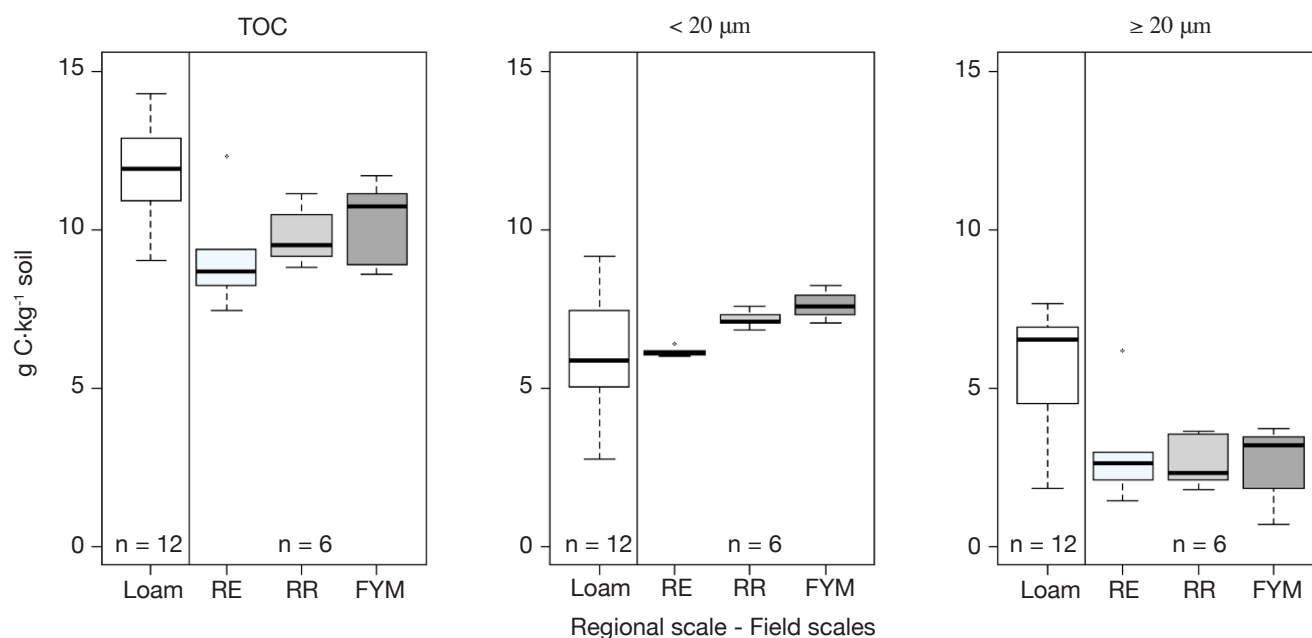


Figure 3. Comparison between regional scale (12 sites located in the central part of the “Loam Belt” in Belgium) and field scale (long term experiment in central Belgium, Gembloux, n = 6) for total organic carbon (TOC), < 20 μm and ≥ 20 μm soil organic carbon in 2012 — *Comparaison entre l'échelle régionale (12 sites localisés dans la région limoneuse en Belgique centrale) et l'échelle de la parcelle (expérience à long terme en Belgique centrale, Gembloux, n = 6) pour le carbone organique total (TOC), le carbone ≥ 20 μm et < 20 μm en 2012.*

RE: residue export — *export des résidus de cultures*; RR: residue restitution — *incorporation des résidus de cultures*; FYM: farmyard manure — *fumier*; n: number of plots — *nombre de parcelles*.

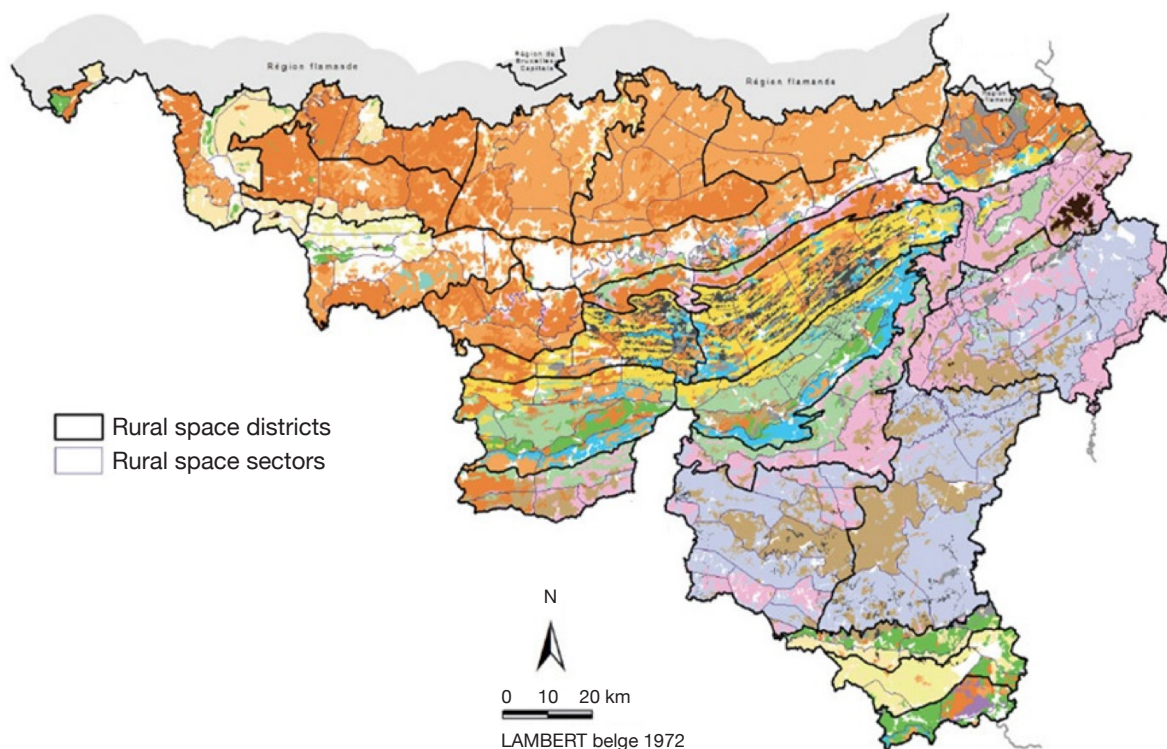


Figure 4. Rural space units (districts and sectors) in Wallonia, southern Belgium, overlaid on the principal soils — *Unités d'espace rural (districts et secteurs) en Wallonie, superposées sur les principaux sols de Wallonie.*

Legend — *légende*: see — *voir*: Legrain et al., 2011. *Carte des principaux types de sols de Wallonie à 1/250 000 (CNSW250)*, <http://www.pressesagro.be/base/index.php/base/article/view/583/570>, (11.16.2016).

4.2. Comparison of specific fields with corresponding RSDs

An assessment of SOC fractions for each specific rural space district (RSD) can be established. In Wallonia, information on bulk SOC is available to highlight RSD's where low levels of SOC can disturb soil ecosystem services (Colinet et al., 2005; Goidts et al., 2009a). In each of those RSD's, the distribution of SOC fraction is established after random sampling. Each fraction is summarized by statistical information determining the Normal Operative Range (NOR) (Pereira et al., 2013) such as mean and quantiles for cropland and grassland and different classes of fine fraction contribution (% of $< 20 \mu\text{m}$ fraction). These fractions are considered as proxies for soil ecosystem services. For a specific RSD, three soil functions (carbon sequestration, available carbon, soil structure), corresponding to our fractions, are evaluated. The soil function level of each district can then be divided into different classes based on relevant thresholds.

By means of this diagnostic, data from a soil plot can be compared with the NOR of its corresponding RSD. It will then be possible to position the observation in the context of the RSD and evaluate its relative position in relation to soil function status. Depending on the plot status, farmer and managers can highlight if improved management is needed. Decisions are based on comparison with the corresponding RSD, after interpreting geopedological parameters of the specific plots. The integration of local parameters in the diagnosis such as soil association, parental material or other available soil parameters is needed for interpreting information given by the NOR.

4.3. Sampling

Composite samples help to smooth spatial heterogeneity within analyzed plots. However, mixing and probe sampling may alter aggregates by enhancing compaction or disrupting macroaggregates. The application of sampling protocol between sites has to be identical in order to avoid any bias at the beginning of the procedure. The timing of sampling also influences the amount of labile carbon or the aggregation level due to seasonal climatic effects and crop management.

As indicated by Wuest (2014), seasonal fluctuations in soil C can be significant (2-8% of the mean over a 39 months period or 4-13% in a no-till system). This has implications for the accurate comparison of SOC between sites, treatments and even in the same plot over time. In Liroux, samples are always taken after the harvest, reducing the impact of intra-annual variation of SOC. Extensive campaigns covering an entire region, are normally not restricted to the post-harvest period only. As long-term experiments demonstrating

the effect of improved management options are scarce, paired field approaches can be the way forward in providing these indicators for each RSD (Poeplau et al., 2014).

4.4. Fractionation

In order to use soil quality and its related functions as a tool for sustainability of agro-ecosystems, physical, chemical and biological properties must be employed to verify which respond to land use and management practices or edaphic variability within a specific timescale (Cardoso et al., 2013; Wiesmeier et al., 2014). Fractionation is time-consuming compared to a bulk SOC analysis. However, the former provides an efficient means to monitor relevant soil functions through the quantification of SOC fractions used as proxies. Loveland et al. (2003) indicated that further investigation of relationship between "active" (*i.e.*, labile carbon) and "total" SOC and soil properties was needed to better understand soil behavior and improve our understanding of soil quality and related issues. Soil organic carbon fractions, as defined here, are an approximation of the theoretically existing labile-stable dichotomy. Indeed, in terms of their decomposability and turnover time, SOC pools correspond more to a continuum of SOC compounds.

Note that the fractionation scheme is not relevant in sandy or organic soils as aggregation and chemical processes differ from fine-grained mineral soils as mineral binding does not contribute significantly to SOM stabilization (Sleutel et al., 2010). However, sandy soils do not cover large areas in agricultural soils of Wallonia, except in the Hennuyere campine or Jurassic region (Lettens et al., 2004). Dissolved organic carbon (DOC) was not measured as it represents a small part of bulk SOC. Nevertheless, as DOC leaching plays an important role in SOC dynamics, investigating DOC changes could help to detect the reactivity of the soil system to environmental changes. Indeed, DOC sorption onto mineral surfaces is very likely to be an important mechanism in stabilizing organic matter (Kalbitz et al., 2008).

5. CONCLUSIONS

Soil organic matter plays an important role in agroecosystems. Agricultural practices can decrease SOM levels, and this requires efficient soil management. An assessment of the status of agricultural soils is therefore needed to identify the effect of different management practices. Methods based on bulk SOC are difficult to implement, as they are not sensitive enough to describe the complex soil system. Soil organic carbon fractions, used as proxies for soil ecosystem functions,

can be helpful because they represent SOC functional pools. In this study, we proposed to apply fractionation on samples taken at both the plot and regional scale. This approach enables to establish a normal operative range for a specific rural space district. From this, a baseline of SOC fractions state in agricultural units can be derived which can provide valuable information to study the impact of agricultural management on soil quality. In the literature, a wide range of indicators such as available water capacity, aggregate stability, potentially mineralizable nitrogen or soil chemical composition have been proposed for soil quality diagnosis (Gugino et al., 2009). Here, the approach based on SOC fractions has the potential to be applied for routine soil analysis and for which the regional baselines can be established. As a first step into this direction, this study showed that two simple fractions (< 20 and $\geq 20 \mu\text{m}$) in bulk soil could be used to determine the existing land use and management types in each RSD, allowing the Normal Operative Ranges to be defined. As a second step, indicator values reflecting management options in each RSD should be identified and linked to SOC fraction status. This information could then assist in formulating quantitative advice to improve soil quality.

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Bibliography

- Arrouays D. et al., 2008. *Environmental assessment of soil for monitoring: volume iib survey of national networks. EUR 23490 EN/2B*. Luxembourg: Office for the Official Publications of the European Communities.
- Baldock J.A. & Nelson P.N., 2000. Soil organic matter. In: Sumner M.E., ed. *Handbook of soil science*. Boca Raton, FL, USA: CRC Press, B25-B84.
- Balesdent J., Besnard E., Arrouays D. & Chenu C., 1998. The dynamics of carbon in particle-size fractions of soil in a forest-cultivation sequence. *Plant Soil*, **201**, 49-57.
- Buysse P., Roisin C. & Aubinet M., 2013. Fifty years of contrasted residue management of an agricultural crop: impacts on the soil carbon budget and on soil heterotrophic respiration. *Agric. Ecosyst. Environ.*, **167**, 52-59.
- Cardoso E. et al., 2013. Soil health: looking for suitable indicators. What should be considered to assess the effects of use and management on soil health? *Sci. Agricola*, **70**, 274-289.
- Chartin C. et al., 2015. *Carbone organique, biomasse et activité microbienne des sols : vers un indicateur de la qualité des sols en Wallonie. Rapport final de la subvention CARBIOSOL I*. Namur, Belgique : SPW-DGARNE.
- Chenu C., Rumpel C. & Lehman J., 2014. *Chapter 13 – Methods for studying soil organic matter: nature, dynamics, spatial accessibility, and interactions with minerals*. In: Eldor A.P., ed. *Soil microbiology, ecology and biochemistry*. 4th ed. Oxford, UK: Elsevier.
- Colinet G. et al., 2005. *Base de données sols de Requasud. 2^e synthèse*. Gembloux, Belgique : Asbl Requasud.
- Follett R.F., 2001. Soil management concepts and carbon sequestration in cropland soils. *Soil Tillage Res.*, **61**, 77-92.
- Genot V., Colinet G. & Bock L., 2007. *La fertilité des sols agricoles et forestiers en Région wallonne. Dossier scientifique réalisé dans le cadre de l'élaboration du rapport analytique 2006-2007 sur l'état de l'environnement wallon*. Gembloux, Belgique : Laboratoire de Géopédologie, Département Sol-Écologie-Territoire, Faculté universitaire des Sciences agronomiques de Gembloux.
- Goidts E., 2009. *Soil organic carbon evolution at the regional scale: overcoming uncertainties & quantifying driving forces*. PhD thesis: Université catholique de Louvain, Louvain-la-Neuve (Belgium).
- Goidts E., van Wesemael B. & Van Oost K., 2009a. Driving forces of soil organic carbon evolution at the landscape and regional scale using data from a stratified soil monitoring. *Global Change Biol.*, **15**, 2981-3000.
- Goidts E., van Wesemael B. & Crucifix M., 2009b. Magnitude and sources of uncertainties in soil organic carbon (SOC) stock assessments at various scales. *Eur. J. Soil Sci.*, **60**, 723-739.
- Gugino B.K. et al., 2009. *Cornell soil health assessment training manual. Edition 2.0*. Geneva, NY, USA: Cornell University.
- Hassink J., 1997. The capacity of soils to preserve organic C and N by their association with clay and silt particles. *Plant Soil*, **191**, 77-87.
- Kalbitz K. & Kaiser K., 2008. Contribution of dissolved organic matter to carbon storage in forest mineral soils. *J. Plant Nutr. Soil Sci.*, **171**, 52-60.
- Karlen D. et al., 1997. Soil quality: a concept, definition, and framework for evaluation (a guest editorial). *Soil Sci. Soc. Am. J.*, **61**, 4-10.
- Lal R., 2015. Restoring soil quality to mitigate soil degradation. *Sustainability*, **7**, 5875-5895.
- Legrain X. et al., 2011. Méthodologie de constitution d'une collection d'échantillons de sols en relation avec les principaux matériaux parentaux en Wallonie (Belgique méridionale). *Biotechnol. Agron. Soc. Environ.*, **15**, 683-697.

- Lehmann J. & Kebler M., 2015. The contentious nature of soil organic matter. *Nature*, **528**, 60-68.
- Letten S., Van Orshoven J., van Wesemael B. & Muys B., 2004. Soil organic and inorganic carbon contents of landscape units in Belgium derived using data from 1950 to 1970. *Soil Use Manage.*, **20**, 40-47.
- Loveland P. & Webb J., 2003. Is there a critical level of organic matter in the agricultural soils of temperate regions? a review. *Soil Tillage Res.*, **70**, 1-18.
- Paul E.A., Paustian K., Elliott E.T. & Cole C., 1997. *Soil organic matter in temperate ecosystems*. New York, NY, USA: CRC Press.
- Paustian et al., 2000. Management options for reducing CO₂ emissions from agricultural soils. *Biogeochemistry*, **48**, 147-163.
- Pereira e Silva M. et al., 2013. Microbe-mediated processes as indicators to establish the normal operating range of soil functioning. *Soil Biol. Biochem.*, **57**, 995-1002.
- Poeplau C. et al., 2013. Reproducibility of a soil organic carbon fractionation method to derive RothC carbon pools. *Eur. J. Soil Sci.*, **64**, 735-746.
- Poeplau C. & Don A., 2014. Soil carbon changes under *Miscanthus* driven by C4 accumulation and C3 decomposition - toward a default sequestration function. *Global Change Biol. Bioenergy*, **6**, 327-338.
- Pribyl D.W., 2010. A critical review of the conventional SOC to SOM conversion factor. *Geoderma*, **156**, 75-83.
- Sherrod L.A., Dunn G., Peterson G.A. & Kolberg R.L., 2002. Inorganic carbon analysis by modified pressure-calimeter method. *Soil Sci. Soc. Am. J.*, **66**, 299-305.
- Six J., Conant R.T., Paul E.A. & Paustian K., 2002. Stabilization mechanisms of soil organic matter: implications for C-saturation of soils. *Plant Soil*, **241**, 155-176.
- Sleutel S., Abdul Kader M., Ara Begum S. & De Neve S., 2010. Soil organic matter stability in sandy cropland soils is related to land-use history. *J. Plant Nutr. Soil Sci.*, **173**, 19-29.
- Smith P. et al., 2000. Meeting Europe's climate change commitments: quantitative estimates of the potential for carbon mitigation by agriculture. *Global Change Biol.*, **6**, 525-539.
- Sollins P., Homann P. & Caldwell B., 1996. Stabilization and destabilization of soil organic matter mechanisms and controls. *Geoderma*, **74**, 65-105.
- Stoate C. et al., 2009. Ecological impacts of early 21st century agricultural change in Europe – A review. *J. Environ. Manage.*, **91**, 22-46.
- Trigalet S., Van Oost K., Roisin C. & van Wesemael B., 2014. Carbon associated with clay and fine silt as an indicator for SOC decadal evolution under different residue management practices. *Agric. Ecosyst. Environ.*, **196**, 1-9.
- USDA, 1993. *Soil survey manual. Handbook 18*. Washington, DC: U.S. Department of Agriculture, Soil Conservation Service.
- Van Oost K. et al., 2012. Legacy of human-induced C erosion and burial on soil – atmosphere C exchange. *Proc. Natl. Acad. Sci. U.S.A.*, **109**(47), 19492-19497.
- Von Lützw M. et al., 2007. SOM fractionation methods: relevance to functional pools and to stabilization mechanisms. *Soil Biol. Biochem.*, **39**, 2183-2207.
- Wiesmeier M. et al., 2014. Quantification of functional soil organic carbon pools for major soil units and land uses in southeast Germany (Bavaria). *Agric. Ecosyst. Environ.*, **185**, 208-220.
- Wuest S., 2014. Seasonal variation in soil organic carbon. *Soil Sci. Soc. Am. J.*, **78**, 1442-1447.
- Zimmermann M. et al., 2007. Measured soil organic matter fractions can be related to pools in the RothC model. *Eur. J. Soil Sci.*, **58**, 658-667.

(31 ref.)