https://doi.org/10.17221/101/2022-SWR

$\delta^{13}C$ as a tool to determine the origin of soil organic carbon: Case study of a restored sloping orchard

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Citation: Muršec M., Leveque J. (2023): $\delta^{13}C$ as a tool to determine the origin of soil organic carbon: Case study of a restored sloping orchard. Soil & Water Res., 18: 81–88.

Abstract: The effect of drip irrigation on the origin and size fraction of soil organic carbon was studied in the soils of an apple orchard (*Malus domestica* Borkh.) on hilly (20%) terrain in northeastern Slovenia in three slope positions (upslope, midslope and downslope), comparing irrigated with non-irrigated soils. Physical fractionation of soil organic carbon was performed on three soil layers (0–0.05, 0.05–0.15 and 0.15–0.30 m) in three size fractions: fraction A (> 0.0002 m), fraction B (0.0002–0.00005 m) and fraction C (< 0.00005 m). Fraction A was the richest in soil organic carbon (7.7%), but fraction C was the dominant fraction in the total soil volume (86–92%), making it the largest source of soil organic carbon (73%). The $\delta^{13}C$ signature was performed to determine the existence of two different types (origins) of soil organic carbon: fresh and sedimentary. Fresh organic carbon dominates in the A fraction, while sedimentary organic carbon dominates in the C fraction and may contribute to higher structural stability, besides higher carbonates in the finest fraction. Irrigation mainly contributes to the higher stock of soil organic carbon (predominantly fresh and less sedimentary) in the coarse A fraction (21.14 t/ha in irrigated and 14.17 t/ha in non-irrigated soils).

Keywords: $CaCO_3$; carbon isotopes; drip irrigation; physical fractionation; soil organic matter

Fractionation of soil organic carbon (SOC) is crucial for understanding the decomposition and stabilization processes of soil organic matter (Poeplau et al. 2018). A combination of physical and chemical methods for SOC separation may be most effective in terms of turnover rates (Poeplau et al. 2018). There is a wide range of applied fractionation methods with different goals in measuring SOC (von Lützow et al. 2007). Most of the recently developed SOC fractionation methods are physical (particle size or density separation), which highlights the fundamental interactions between organic and inorganic soil components in the turnover of SOC (Christensen 2001). Stabilization of SOC in fine fractionated soils (silt and clay) is usually the result of physical protection of SOC by organo-mineral complexes and by aggregates (Six et al. 2002; Kaiser & Guggenberger 2003).

Fractionation-derived SOC pools are used to characterize SOC in terms of their formation and stability (Baldock et al. 2013; Cotrufo et al. 2015). For studies on environmental changes (land use, soil management, etc.) of the accumulation and turnover rates of SOC, ^{13}C abundance or other biomarkers are useful (Di Giovanni et al. 1997; Del Galdo et al. 2003; Dondini et al. 2009). In recent decades, stable carbon isotopes have been widely used to quantitatively assess the generation, stability, and turnover of SOC (Poeplau et al. 2018). This is the most effective method, which allows the identification of the sources and processes of carbon fractions (Liu et al.

Supported by Biogéosciences, University Bourgogne Franche-Comté, UMR CNRS 6282, France.

2018; Wang et al. 2020), which relative contribution of new SOC and old SOC can be estimated (Zhang et al. 2015; Dou et al. 2017; Jiang et al. 2019). Stable carbon isotopes can also link aggregate dynamics with organic matter, revealing long-term changes in SOC composition (Wang et al. 2020; Xiao et al. 2022).

Physical fractionation (size or density) can be used to separate different classes of SOC pools. Using both methods, it is found that the coarse fractions are light, and consist mainly of particulate organic matter (POM) with short turnover times. The fine fractions are heavy and consist of organic matter bound to mineral components such as clays. These organic materials are stabilized because they are difficult to decompose, with long turnover times (Christensen 1992; von Lützow et al. 2007). The use of the δ^{13}C signature and density fractionation provided information on the age of the SOC. A high δ^{13}C signature found in heavy soil fractions with low SOC is older, while light carbon fractions display a low δ^{13}C value, reflecting recent organic matter (Baisden et al. 2002; Sollins et al. 2009). Finally, the origin of soil organic matter is generally attributed to vegetation or plant succession present in the soil, assuming that the organic carbon contribution of the parent material is not significant. Several authors have shown that this is not always the case in optical and Rock-Eval pyrolysis observations, especially when dealing with recent and unbound sedimentary material (Sebag et al. 2006; Graz et al. 2010).

Our hypothesis was that we are dealing with two types of SOC in the studied soils: young SOC (consisting largely of fresh material from apple trees and grass) and old sedimentary SOC, associated with marl (bedrock). Our question was how much SOC originated from fresh organic matter and how much from the bedrock. This was highlighted with the δ^{13}C isotope data, which provide information on the type and rate of stabilisation of organic matter and the structural stability measurements obtained for different topological positions of irrigated and non-irrigated areas.

The main objectives of this study were: (1) to perform a physical (size) fractionation of SOC and separation of SOC according to its origin by measuring the δ^{13}C signature; (2) to establish correlations between the total SOC, δC^{13}, $CaCO_3$, C/N and structural stability; (3) to determine which type of SOC contributes more to structural stability; (4) to investigate the effect of drip irrigation on the content of fresh and sedimentary SOC stock content.

MATERIAL AND METHODS

The study was conducted at the Gačnik Experimental Station in NE Slovenia (46°61'N, 15°68'E, 300 m a.s.l.) in an apple orchard (*Malus domestica* Borkh.) on hilly (20%) terrain on three slope positions (upslope, midslope and downslope), comparing irrigated (IRR) with non-irrigated (NIR) soils. The original soil type was Eutric Cambisol (FAO 2006), developed on Helvetic marl bedrock with a homogeneous silty clay texture (47% clay, 50% silt, 3% sand). Upslope, the hard unweathered marl bedrock occurred at 0.6 m depth with some blocks of marl before (0.3–0.6 m), while downslope evidence of deep ploughing till 0.6 m with the soft weathered saprolithe from 1 m depth was observed. The structure of the topsoil horizon varied with depth from granular (0–0.05 m upslope and 0–0.1 m downslope) to subangular polyedric (0.05–0.15 m) and fine polyedric 0.15–0.3 m) to coarse polyedric (deeper than 0.3 m). Upslope soil was shallower, drier (including some vertical cracks) with less organic matter (0.6% SOC in 0–0.3 m soil layer with colour 10 YR 5/3) upslope, comparing to downslope soil (1.6 % SOC in 0–0.3 m with colour 10 YR 3/2 and 10 YR 4/4). Soils in this orchard are very heterogenic and anthropogenic, resulting of terracing, land levelling and deep ploughing.

Prior to the current planting (in the year 2004), the trees were planted in terraces; the orchard was then restored vertically along the slope, largely disturbing the initial organisation and distribution of organic matter and parent material. The soil surface between the rows was covered with natural grass. During the first six years, drip irrigation was applied from May to September at a rate of 2 L/tree/day, with a maximum of 70 days of irrigation per season. The climate is temperate, with a mean annual precipitation of 1.047 m and a mean annual temperature of 9.7 °C for the 1961–1990 reference period. Fresh soil samples were collected in triplicate at the end of the irrigation period in the IRR and NIR rows from three soil layers (0–0.05, 0.05–0.15 and 0.15–0.30 m) at three locations along the slope: at the top of the slope (UP), in the middle of the slope (MID), and at the bottom of the slope (DOWN).

The percentage of water stable aggregates (WSA) was determined according to the method described by Kemper and Rosenau (1986) and adapted by Bartoli et al. (1991). Total calcium carbonate ($CaCO_3$%) content was measured by analysing the volume of CO_2 released after the addition of HCl (ISO 10693:1995).

For physical fractionation of SOC, soil samples were dried at 60 °C before sieving (0.002 m mesh size). The soil samples were separated by wet sieving into three size fractions: fraction A (> 0.0002 m), fraction B (0.0002 m > B > 0.00005 m), and fraction C (< 0.00005 m). Fraction A represents a coarse sand fraction, fraction B represents a fine sand fraction, while fraction C represent a fraction of silt and clay together. A possible gravimetric separation procedure of silt and clay could not be considered due to the flocculation effect of clays associated with the presence of a very high amount of active lime in the studied soil.

To remove carbonate, the samples were treated with 7% HCl prior to analysis. Organic carbon, nitrogen, and isotope 13C were measured using a Micromass isotope ratio mass spectrometer (VG Isochrom-EA, Micromass FISONS, UK) coupled to a Carlo Erba 1500 CNS elemental analyzer (VG Isochrom-EA, Micromass FISONS, UK). The natural abundance of the stable isotope ^{13}C was determined for each sample and expressed in δ-units (‰) with reference to the international PDB standard (Craig 1957) according to the following Equation (1).

$$\delta\ ^{13}\mathrm{C}(‰) = 10^3 \times \left[\left(\frac{^{13}\mathrm{C}}{^{12}\mathrm{C}}\right)\mathrm{sample} - \frac{\left(\frac{^{13}\mathrm{C}}{^{13}\mathrm{C}}\right)\mathrm{PDB}}{\left(\frac{^{13}\mathrm{C}}{^{12}\mathrm{C}}\right)\mathrm{PDB}} \right] \quad (1)$$

C and N contents were determined using the sulphanilamide standard (41.84% C and 16.27% N). Calibration for δ^{13}C was performed using the USGS24 international graphite standard (δ^{13}C = –16.0 ± 0.1‰). Duplicates and triplicates provided a precision of higher than ± 0.15 ‰ δ-units. The level of fresh ($\mathrm{SOC_{fresh}}$) and sedimentary SOC ($\mathrm{SOC_{sed}}$) in the soil samples was calculated separately for each fraction using the following mixing model and expressed as a percentage of total SOC ($\mathrm{\%SOC_{fresh}}$ and $\mathrm{\%SOC_{sed}}$) as Equations (2–5):

$$\mathrm{SOC_{fresh}} = \mathrm{SOC_{total}} \times \left(\frac{(\delta_{\mathrm{total}} - \delta_{\mathrm{sed}})}{(\delta_{\mathrm{fresh}} - \delta_{\mathrm{sed}})}\right) \quad (2)$$

$$\mathrm{SOC_{sed}} = \mathrm{SOC_{total}} - \mathrm{SOC_{fresh}} \quad (3)$$

$$\mathrm{\%SOC_{fresh}} = 100 \times \mathrm{SOC_{total}} \times \left(\frac{(\delta_{\mathrm{total}} - \delta_{\mathrm{sed}})}{(\delta_{\mathrm{fresh}} - \delta_{\mathrm{sed}})}\right) \quad (4)$$

$$\mathrm{\%SOC_{sed}} = 100 - \mathrm{\%SOC_{fresh}} \quad (5)$$

where:

$\mathrm{SOC_{total}}$ – sum of sediment and fresh SOC;

δ_{total} – δ^{13}C value of the total SOC in the soil samples;

δ_{sed} – δ^{13}C value of old sedimentary SOC (–23.32 ± 0.15‰ measured in the marl saprolite);

δ_{fresh} – δ^{13}C value of fresh SOC (–28.18 ± 0.15‰ measured in the mixture of fresh apple tree residues such as branches, leaves and grass).

Total, sedimentary, and fresh stocks of SOC in a 0–0.30 m layer (all three previously mentioned layers together to represent the whole soil bulk) are calculated by considering the layer thickness, bulk density (BD), and the percent of rock fragments in each layer (frag/100) according to the IPCC LULUCF guidelines (Penman et al. 2003) in Equation (6).

$$\mathrm{SOC_{stock}}\ (\mathrm{t/ha}) = \mathrm{SOC}\ (\%) \times \mathrm{BD}\ (\mathrm{g/m^3}) \times \\ \times \text{soil layer (cm)} \times (1 - (\mathrm{frag}/100)) \quad (6)$$

Statistical analysis of the data was performed using the Statgraphics Centurion XV statistical program (Statgraphics 2005), with multifactor ANOVA, considering statistical significance $P < 0.05$. The LSD multiple test was used for the mean separation of statistically significant factors. The significance of linear relationships between pairs of data parameters was expressed in terms of Pearson correlation coefficients ($P < 0.05$, $P < 0.01$, $P < 0.001$).

RESULTS AND DISCUSSION

Correlations between total SOC, $\mathrm{CaCO_3}$, δ^{13}C, C/N and structural stability (WSA) were calculated over the 0.3 m soil depth cumulating the 3 levels of soil layers. The Pearson correlation coefficients between these parameters in the total soil bulk are shown in Table 1. Strong positive correlations ($P < 0.01$) were found between WSA and $\mathrm{CaCO_3}$, WSA and δ^{13}C and between C/N and SOC. On the other hand, a strong negative correlation was found between WSA and SOC. These correlations indicate that high SOC content does not result in improved structural stability. It is the carbonate content that controls the WSA with systematically higher δ^{13}C and lower SOC. Poeplau et al. (2018) and Volk et al. (2018) reported that higher δ^{13}C values tend to be associated with older, stable SOC with a longer turnover rate and associated with initial mineral matter.

Table 1. Pearson correlation coefficients (*R*) between pairs of measured parameters in the 0–0.3 m soil layer

	$CaCO_3$ (%)	C/N	δC^{13} (‰)	SOC (mg/kg)	WSA (%)
$CaCO_3$ (%)		–0.37	+0.33	–0.43	+0.55***
C/N	–0.37		–0.23	+0.74***	–0.35
δC^{13} (‰)	+0.33	–0.23		–0.39	+0.61***
SOC (mg/kg)	–0.43	+0.74***	–0.39		–0.58***
WSA (%)	+0.55***	–0.35	+0.61***	–0.58***	

SOC – total organic carbon; WSA – water structural stability; ***$P < 0.01$; $n = 18$

Lower $\delta^{13}C$ values are associated with fresh, mainly particulate SOC, which is more labile with a shorter turnover time.

On the slope, the distribution between WSA and $CaCO_3$ content indicates that we are dealing with two types of SOC: fresh SOC, associated with fewer carbonates and lower WSA at the bottom of the slope, and sedimentary SOC, associated with higher carbonates and higher WSA at the top of the slope, indicating the presence of Helvetian marls (Figure 1). Stable sedimentary SOC, related to higher $\delta^{13}C$ values, may potentially contribute to higher WSA. Sedimentary SOC is probably related with heavy occluded and mineral SOC (with high density), while fresh SOC with free particulate and light occluded SOC (with low density) (Gunina & Kuzyakov 2014). Within the aggregate size classes, $\delta^{13}C$ values increase from macroaggregates to the silt + clay-sized fraction, while within the SOC fractions is increased from free light fractions, to heavy occluded and mineral fractions, under the different treatments (Chen et al. 2022). The SOC in heavy fractions is the product of a process with stronger or more frequent microbial transformations, concluded that occluded SOC is more physically protected and thus difficult to decompose (Chen et al. 2022).

Table 2 shows the statistically significant differences (LSD test, $P < 0.05$) in C/N, SOC, $\delta^{13}C$, sedimentary and fresh SOC, according to the different factors. Comparing irrigated (IRR) with non-irrigated (NIR) soils, SOC, C/N and $\delta^{13}C$ values are higher in IRR soils which have a lower, fresh SOC. Wu et al. (2008) reported that long-term irrigation can significantly increase SOC and soil inorganic carbon (SIC) by converting soil CO_2 to carbonates. In Gačnik, some carbonate is supplied in the form of active lime from the collection of runoff and infiltration water in the pond that feeds the irrigation system. Along the slope, C/N, SOC and the proportion of fresh SOC are higher in mid and downslope, due to higher soil moisture content increasing the growth of the grass cover downwards. SOC and the proportion of fresh SOC decreases with deeper soil layers, while $\delta^{13}C$ increases upslope and with deeper layers. Using the mixing equation to distinguish the respective contributions of the organic sources (fresh/current against older/sedimentary), the coarse fraction A is mainly composed of recent SOC, and the

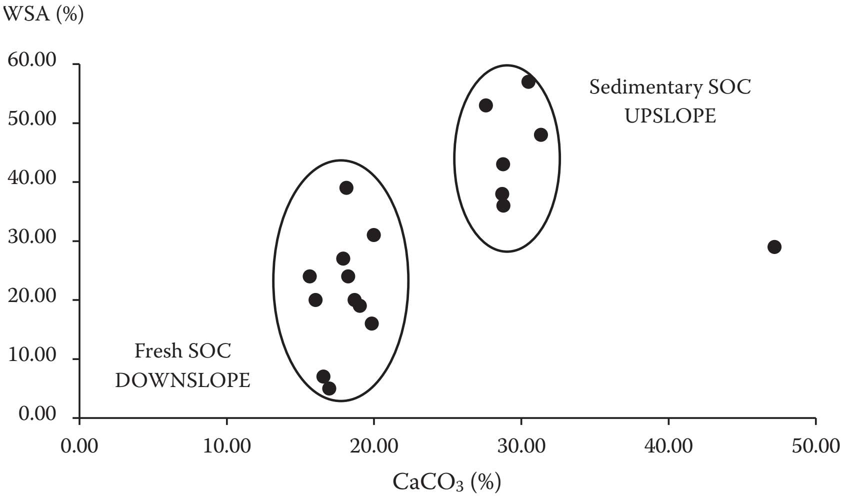

Figure 1. Relation of water structural stability (WSA) and $CaCO_3$

SOC – soil organic carbon

Table 2. Review of C/N, total soil organic carbon (SOC) content, δC^{13}‰, percentage of fresh and sedimentary SOC in total SOC in the in the 0–0.3 m soil layer

Factor		C/N	SOC (%)	δC^{13} (‰)	SOC_{fresh}/SOC	SOC_{sed}/SOC
					(%)	
Water treatment (n = 81)	IRR	12.9 ± 0.9^{a}	4.8 ± 0.5^{a}	-25.9 ± 0.1^{a}	55 ± 3^{b}	45 ± 3^{a}
	NIR	10.4 ± 0.5^{b}	3.1 ± 0.2^{b}	-26.2 ± 0.1^{b}	60 ± 2^{a}	40 ± 2^{b}
Slope position (n = 54)	UP	11.1 ± 1.1^{b}	3.2 ± 0.4^{b}	-25.3 ± 0.2^{a}	42 ± 3^{c}	58 ± 3^{a}
	MID	11.9 ± 0.7^{a}	4.3 ± 0.6^{a}	-26.7 ± 0.1^{c}	71 ± 2^{a}	29 ± 2^{c}
	DOWN	12.2 ± 0.8^{a}	4.4 ± 0.6^{a}	-26.2 ± 0.1^{b}	59 ± 2^{b}	41 ± 2^{b}
Soil layer (n = 54)	0–0.05	11.3 ± 0.7^{b}	5.0 ± 0.7^{a}	-26.2 ± 0.2^{c}	61 ± 3^{a}	39 ± 3^{b}
	0.05–0.15	11.2 ± 0.7^{b}	3.0 ± 0.3^{b}	-26.1 ± 0.1^{b}	58 ± 3^{ab}	42 ± 3^{ab}
	0.15–0.30	12.6 ± 1.2^{a}	3.8 ± 0.5^{ab}	-25.9 ± 0.2^{a}	53 ± 3^{c}	47 ± 3^{a}
Soil size fraction (n = 54)	A	18.5 ± 0.8^{a}	7.7 ± 0.6^{a}	-26.7 ± 0.9^{c}	70 ± 3^{a}	30 ± 3^{c}
	B	10.81 ± 0.3^{b}	3.1 ± 0.2^{b}	-26.4 ± 0.1^{b}	64 ± 3^{b}	36 ± 3^{b}
	C	6 ± 0.2^{c}	1.2 ± 0.1^{c}	-25.1 ± 0.1^{a}	38 ± 2^{c}	62 ± 2^{a}

SOC_{fresh}/SOC – ratio of fresh and total organic carbon; SOC_{sed}/SOC – ratio of sedimentary and total organic carbon; IRR – irrigated; NIR – non-irrigated; UP – upslope; MID – midslope; DOWN – downslope; values are means (n = 3) with standard error; values followed by the same letter according to each factor are not significantly different ($P < 0.05$)

fine fraction C is dominated by ancient and stable SOC. In the coarser fractions A and B, the fresh/sedimentary SOC ratio is approximately 2, while in the finest C fraction it is approximately 1. As for the C/N ratio, a very low value, 3.6 to 4.4, is visible in the C fraction, for the soil sample in the upslope position. C/N ratio gives the information of SOC turnover rate: higher C/N ratio is related with more fresh SOC (microbial decomposition is not finished yet; with low density) and lower to more stable SOC

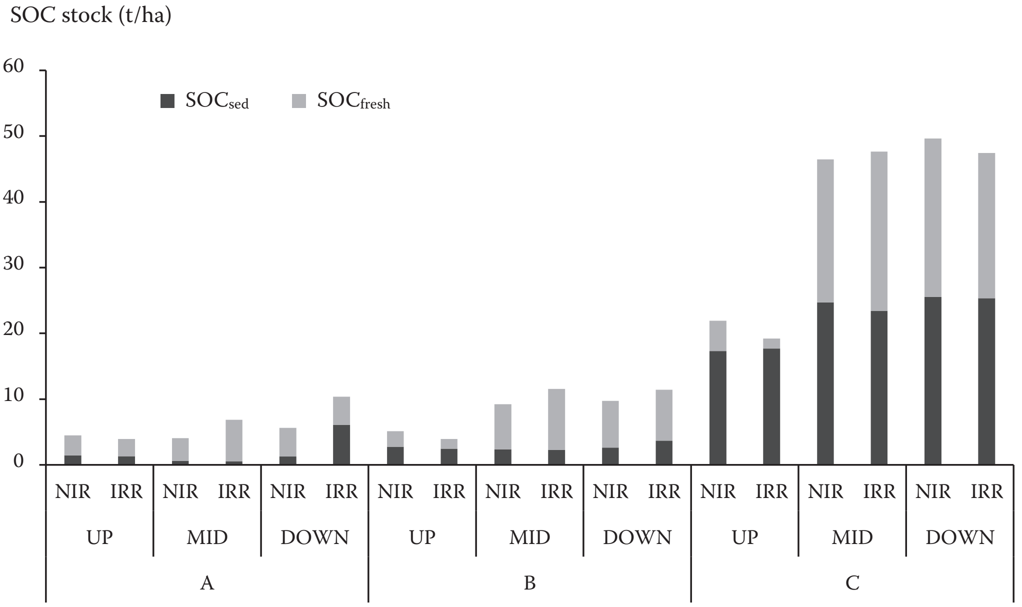

Figure 2. Distribution of fresh and sedimentary soil organic carbon (SOC) stock in the layer 0–0.3 m by size fraction (A > 0.0002 m, 0.00005 m < B < 0.0002 m, C < 0.00005 m), slope position and water treatment

UP – upslope; MID – midslope; DOWN – downslope; NIR – non-irrigated; IRR – irrigated; SOC_{fresh} – fresh SOC; SOC_{sed} – sedimentary SOC (already decomposed; with high density) (Wagai et al. 2008). The low C/N ratio was measured in the marl sample and represents the SOC from the parent material (sedimentary SOC).

Considering the total soil bulk, the results of the calculated sed. and fresh SOC stocks in the 0–0.3 m layer are presented according to slope position, water treatment and size fraction (Figure 2). The finest C fraction is the most abundant in the total soil bulk, accounting for 86 to 92%, while the A fraction is the least abundant (1 to 4%). The highest proportion of fresh SOC stock is present in the coarser A and B fractions (ratio of fresh SOC/SOC in both fractions 0.68), midslope and downslope (fresh SOC/SOC ratio 0.57 and 0.52) and almost equally in both water treatments (0.50 in NIR and 0.49 in IRR soils). On the other hand, the largest proportion of sed. SOC stock is found in the finest C fraction (sed. SOC/SOC ratio is 0.58), upslope position (sed. SOC/SOC ratio is 0.73) and almost equally in both water treatments (sed. SOC/SOC ratio is 0.50 in NIR and 0.51 in IRR soils).

Focusing on individual fractions, irrigation contributes to a higher SOC stock in the coarse A fraction: SOC stock in the IRR soils is 21.14 and 14.17 t/ha in the NIR soils, while fresh/sedimimentary OC ratio in IRR is 1.67 in IRR and 3.33 in NIR soil. It is assumed that irrigation contributes to a higher SOC input (mostly fresh and less stable), probably due to better conditions for vegetation growth, root development and higher microbial biomass, and furthermore to a more active mineralisation process of fresh SOC due to better conditions for microbial activity. Kochsiek et al. (2009) reported that irrigated management regimes not only lead to greater litter-SOC inputs but also to higher decomposition rates. Increased mineralization of carbon compounds through an increase of soil biological activity leads to a decrease in SOC (Chaplot & Cooper 2015), which could explain the decrease in SOC in the surface soil layer of IRR soils upslope.

It should be taken into account that in this orchard, large amounts of basic rock were mixed with fresh SOC during mechanical rehabilitation before planting. Therefore, the high proportion of sed. SOC in the marl is also very important and beside carbonates, could potentially prevent the negative effects of irrigation on structural stability (Muršec et al. 2018).

Our conclusions are: (1) In the studied soil, carbonates significantly contributed to the stability of structural aggregates and playing a protective role of organic matter in relation to soil aggregates. This mechanism is enhanced by irrigation, which increases the effect of slope. (2) The method of physical fractionation combined with the use of natural carbon isotopes has enabled to detect the existence of ancient organic matter of sedimentary origin from the parent material. This organic source is expressed in various ways in the topographic profile of the apple orchard. (3) Sedimentary SOC may contribute to soil structural stability. (4) Irrigation contributes to the higher SOC stock in the coarse A fraction and possibly to the higher decomposition rates of fresh SOC there.

Physical fractionation gives the information about the main sources and basic chemical characteristics of SOC, and delta C^{13} signature is a useful tool for determining the origin of SOC (fresh and sedimentary SOC). In our study, it is important to highlight the role of the marl parent material in participation of sedimentary SOC, which is a driven parameter for structural stability.

Acknowledgement. We thank to English lecturer K.M. Hempkin for her valuable help.

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Received: July 13, 2022
Accepted: January 20, 2023