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The Effect of Tillage on Organic Carbon Stabilization in Microaggregates in Different Climatic Zones of European Russia

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Abstract: Tillage may affect the microstructural organization of soil, including the distribution of microaggregates with different mechanical strengths. We quantified the impact of tillage treatment on the amount and distribution of free organic matter, microaggregates (unstable and stable under low intensity sonification) and their components, in the upper horizons of zonal soils of the Center of the Russian Plain. Under plowing, the carbon content decreases, both in unstable and stable microaggregates. The loss of carbon in unstable microaggregates was ~24%, whereas in stable microaggregates, it was ~37%, relative to native soils. The carbon content of organic (LF_{oc}) and organo-clay ($Clay_{rd}$) fractions in unstable microaggregates ($C_{LF}{}^{oc}/C_{Clay}{}^{rd}$) was almost identical in the upper horizons of native soils: the ratio of these components is for Albeluvisols (1.1), Phaeozem (0.8) and Chernozems (1.0). Under plowing, these decrease to: Albeluvisols and Chernozems (0.6) and Phaeozem (0.5). The shares of carbon accumulated within the unstable and stable microaggregates ($C_{unstable}/C_{stable}$) are constant under equilibrium conditions and show a tendency to decrease from north to south on the order of: Albeluvisols and Phaeozem (2.2) > Chernozems (1.0). Under plowing, they increase to: Albeluvisols (3.0) and Phaeozem (3.2) > Chernozems (1.5).

Keywords: tillage; organic matter stabilization; sonification; density fractionation; microaggregates; light fractions; readily-dispersible clay; hardly-dispersible clay

1. Introduction

Soil organic matter (SOM) is one of the most important components of soil, which determines most of the soil quality [1], and reflects the balance between humification and mineralization processes in soil [2–5]. The amount of soil C storage is controlled primarily by two fundamental factors: input by net primary production (quantity and quality) and its decomposition rate. Some reports have pointed out that the chemical composition of organic matter is less important for C dynamics in soils than the location and physical protection of soil C [6–8]. Therefore, SOM dynamics ought to be investigated for their consequences; notably, its effect on the structural stability of soil [9]. The soil structure integrates all mechanisms of interactions among the physical, chemical and biological properties of soil. Soil organic carbon (SOC) can be protected in the soil matrix through physical- (chemical) stabilization processes, as well as by inherent 'chemical recalcitrance' [10]. The stabilization of organic matter (OM) in soil (protection of OM against microbial decomposition) can mainly be attributed to three general mechanisms: spatial inaccessibility, biochemical recalcitrance and organo-mineral association [7]. The importance of these mechanisms can differ for each soil horizon and depends on several factors, such as soil type and texture, mineralogical composition and land use [11].

Soil structure and soil organic matter are two of the most dynamic properties and are extremely sensitive to crop and soil management. Numerous reports have highlighted that changes in land use and management practices influence soil stabilization mechanisms, storage and the dynamics of organic C [7,8,12–17], which subsequently govern soil physical, chemical and microbial processes [18]. Generally, a great deal of SOM losses after land use changes are mainly due to reduced input rates of organic matter, increased decomposability of crop residues and native SOM and decreased physical protection of SOM [19–21].

Tillage can also result in a change in aggregate-size distribution and stability [17,18]. SOM is heterogeneous and consists of different functional and biological pools, varying chemically and dynamically [22,23]. SOM is present both in inter- and intra-aggregate spaces. Intra-aggregate organic matter is incorporated during the macro- and microaggregate formation processes and is physically stabilized within them [19,24,25]. Whether organic matter is located among aggregates, or within them, determines how easily it can be reached by microorganisms [24] and, therefore, its rate of mineralization [26,27]. Intra-aggregate organic matter is less susceptible to decomposition, relative to free OM in the soil matrix [28,29], due, in part, to the physical and chemical protection that aggregates provide. Physical disturbances, resulting from tillage, may destabilize aggregates and further their disruption, release physically-protected particulate organic matter (POM), increasing its susceptibility to decomposition [30].

Physical fractionation methods, which consider the accessibility of organic material to decomposition, and the character of organic material in situ may facilitate an improved understanding of C dynamics [31–34]. Physical fractionation of soil allows SOC separation into pools of different compositional fractions and reveals their localization in the soil matrix [2,3,33,35]. It should be emphasized that physical fractionation has been used for many years to separate OM that is more or less bound to minerals and is considered to be less destructive to soil organic matter than chemical fractionation, which may alter the chemistry of SOM [32,36].

The composition of the SOM components and the stability factors of microaggregates, considering their different mechanical strengths, were studied in several research projects [20,32,37–45]. However, data regarding the quantitative distribution of microaggregates with different mechanical strengths, in soil and SOC pools accumulated within them, as well as also their dynamics under land use, are yet scant.

In this study, our objectives were to: (1) separate microaggregates with different stabilities to sonification; (2) characterize their components (bulk and carbon content) and dynamics under different land uses; and (3) to estimate the balance between the humification and mineralization processes in soils under different land uses in zonal aspects.

2. Materials and Methods

2.1. Materials

Sample Collection

Our study comprised the climate changes across the north-south geographical gradient, from the southern taiga to the forest-steppes. In particular, the climatic gradient resulted in increasing mean annual air temperatures and mean annual evaporations, as well as decreasing mean annual precipitation (Figure 1; Table 1).

The soils we chose represented the zonal row of soils in the Center of the Russian Plain, and varied in pH, texture and C content. The applied methods included paired section cutting in native and arable soils, formed of the same rocks and making up analogous relief elements.

The native and arable Albeluvisols of the southern taiga subzone in European Russia are represented by the paired sections (5), which were cut within the Moscow Region and characterize soils with different texture. Two section pairs describe the low loamy Umbric Albeluvisols Abruptic (Zvenigorod Biostation and Chashnikovo Scientific-Experimental Ecological Centre, Moscow State

University) on light loams over a fluvioglacial sands. Medium loamy Umbric Albeluvisols Abruptic on loams are defined by three section pairs from Zelenograd Station (V.V. Dokuchaev Soil Science Institute) (Tables 1 and 2): (1) near village Semenovskoe and (2) near village Tishkovo, Malinki Biogeocenological Station (Severtsov Institute of Ecology and Evolution, Russian Academy of Sciences) and the surrounding area. Soils from the Phaeozems zone are represented by native and arable Greyic Phaeozems Albic (medium loamy) over a thick loess-like loam (Ivan'kovo Research Station of V.V. Dokuchaev Soil Science Institute, Tula region), and soils of the forest steppes zone are represented by native and arable Voronic Chernozems Pachic (medium loamy) on loesses (Strelestkaya steppe Reserve, Kursk region) and two section pairs of Chernozems (clay loamy) on eluvial-deluvial clays and on clay loess-like loams (Privolzhskaya forest steppes reserve, Volga Region): Poperechenskay steppe Reserve (Haplic Chernozems Pachic) and Ostrovtsovskay steppe Reserve (Voronic Chernozems Pachic), respectively. Tables 1 and 2 give a short description of the soil properties, soil management strategies and the cultivated plants. Soil profiles were excavated down to the parent material. The upper horizons from native and arable soils were analyzed. Soils samples were collected from different pits for each horizon and pooled.

Figure 1. Location of the investigation sites on a global scale [46].

Table 1. Location and main climate parameters of the studied areas, reference soil groups, pH, parent material, texture and C_{org} of the studied soils.

Zone	Location of the Studied Area				Climate		Parent Material	Soil Group	Soil Texture			pH_{H2O}	pH_{KCl}	C_{org} (%)
	Region	Point	Coordinate		Mean Annual Temperature	Mean Annual Precipitation/ Evaporation			Sand	Silt	Clay			
			N	E										
Taiga	Moscow	Zvenigorod Biostation	55°42′00″	36°43′35″	+3.2	620/550	Glacial deposits	Albeluvisols	54.5–55.5	40.2–40.9	4.3–4.6		4.1–5.2	1.2–2.2
		Chashnikovo Scientific-Experimental Ecological Centre	56°01′47″	37°10′09″	+3.3	600/550			21.0–22.9	71.0–71.4	6.1–7.6		4.0–5.2	1.5–2.4
		Zelenograd Research Station	54°48′18″	37°36′18″	+3.5	600/550			13.0–14.0	72.5–75.6	10.4–14.5		3.9–4.8	1.3–3.2
			55°14′10″	37°59′60″					15.0–18.0	68.7–70.4	13.3–14.6		4.0–5.1	1.2–2.3
		Malinki Biogeocenological Station	55°27′00″	37°13′00″	+3.5	550/550			19.0–20.0	64.9–66.8	13.2–16.1		5.1–5.4	2.0–3.5
	Tula	Ivan'kovo Research Station	54°28′45″	37°41′36″	+4.5	570/600	Loess sediments	Phaeozems	6.0–7.3	76.8–78.4	15.6–15.9		5.3–5.5	4.0
Semi-humid Steppe	Kursk	Strelestkaya steppe Reserve	51°34′00″	36°06′00″	+5.3	550/600		Chernozems	4.2–5.0	70.0–73.1	21.9–24.6	6.8–7.2		3.2–4.6
	Penza	Poperechenskay steppe Reserve	52°58′59″	44°19′20″	+5.2	521/550			2.0	68.3–71.2	26.8–29.7	6.3–7.0		5.0–7.4
		Ostrovtsovskay steppe Reserve	52°49′50″	44°23′27″	+5.3	440/460			5.4–6.0	64.4–66	29.0–29.6	6.8–7.2		4.9–6.3

Table 2. Sampling sites with information on land use, depth, vegetation and the soil management strategies.

Soil	Location	Land Use	Tillage Systems	Depth (cm)	Dominant Vegetation/Dominant Crops	Agricultural Amendment
Umbric Albeluvisols Abruptic	Zvenigorod Biostation	Forest Arable	absent CT *	4–12 0–25	*Betula pendula*, *Betula pubescens* barley crops	absent low
	Chashnikovo Scientific-Experimental Ecological Centre	Forest Arable	absent CT *	4–10 0–25	*Picea abies*, *Betula pendula*, *Betula pubescens* clover crops	absent low
	Malinki Biogeocenological Station	Forest Arable	absent CT *	5–13 0–25	*Quercus pubescens*, *Picea abies* barley crops	absent low
	Zelenograd Station (1)	Forest Arable	absent CT *	3–12 0–25	*Picea abies*, *Betula pendula*, *Betula pubescens* clover crops	absent low
	Zelenograd Station (2)	Forest Arable	absent CT	4–10 0–24	*Picea abies*, *Betula pubescens* clover crops	absent low
Greyic Phaeozems Albic	Ivan'kovo Research Station	Forest Arable	absent CT *	4–16	*Quercus pubescens*, *Betula pendula* barley crops	low low
Voronic Chernozems Pachic	Ostrovtsovskay steppe Reserve	Steppe Arable	absent CT *	5–33 0–28	*Stipa dasyphylla*, *Stipa pennata*, *Stipa zalesskii* wheat crops	absent low
Haplic Chernozems Pachic	Poperechenskay steppe Reserve	Steppe Arable	absent CT *	8–51 0–30	*Stipa dasyphylla*, *Stipa pennata*, *Stipa pulcherrima*, *Stipa zalesskii* wheat crops	absent low
Voronic Chernozems Pachic	Strelestkaya steppe Reserve	Steppe Arable	absent CT *	5–30 0–30	*Stipa pennata*, *Stipa dasyphylla*, *Stipa pulcherrima* barley crops	absent low

* CT, conventional tillage using tractor + leveler for plowing and harrowing with one crop per year.

2.2. Methods

Before use, each sample was air-dried and passed through a 1-mm sieve. The residue was gently ground with the help of a rubber-tipped pistil and also passed through a 1-mm sieve. All sieved parts were pooled and homogenized. The rubber tip was used instead of porcelain in order to avoid superfluous disruption of aggregates and the crushing of the primary minerals.

Different methods are currently applied to study the carbon distribution in aggregates of various sizes [47]. The results obtained depend on the method selected [48]. We used a modified variant of granulo-densimetric fractionation [3,49]. This scheme allows subdividing soil microaggregates into two groups according to their stability to sonification and the properties of organic and organo-mineral components. According to the microaggregate stability concept [38,39], the soil components identified over short-term (5–15 min) ultrasonic processing are constituents of coarse (unstable) microaggregates of 50–250 μm in size. Other soil components are constituents of fine (stable) microaggregates, 1–50 μm in size.

A probe-type ultrasonic vibrator was used for physical dispersion of soils. Briefly, after sonication (71 J·mL^{-1}) of the soil sample (10 g + 50 mL deionized water) for 1 min., the aqueous suspension of soils was centrifuged to yield clay-sized particles (<1 μm), according to Stokes' law [50,51]. The procedure was repeated 15 times, and the aqueous-clay suspension was collected and dried (80 °C) (Figure 2). The repeated procedure of successive fractional clay separation using low intensity sonification (71 J·mL^{-1}) of the aqueous suspension of soil (1 min) allows, first, to destroy microaggregates gradually, as the intensity increases, and secondly, the removal of each fresh clay portion from the initial soil sample, allows not to expose the already-separated clay to superfluous ultrasonic agitation. The choice of a 1-μm dimension was determined by the greater homogeneity of OM in the clay-sized fraction (<1 μm) versus a 2-μm dimension [52,53].

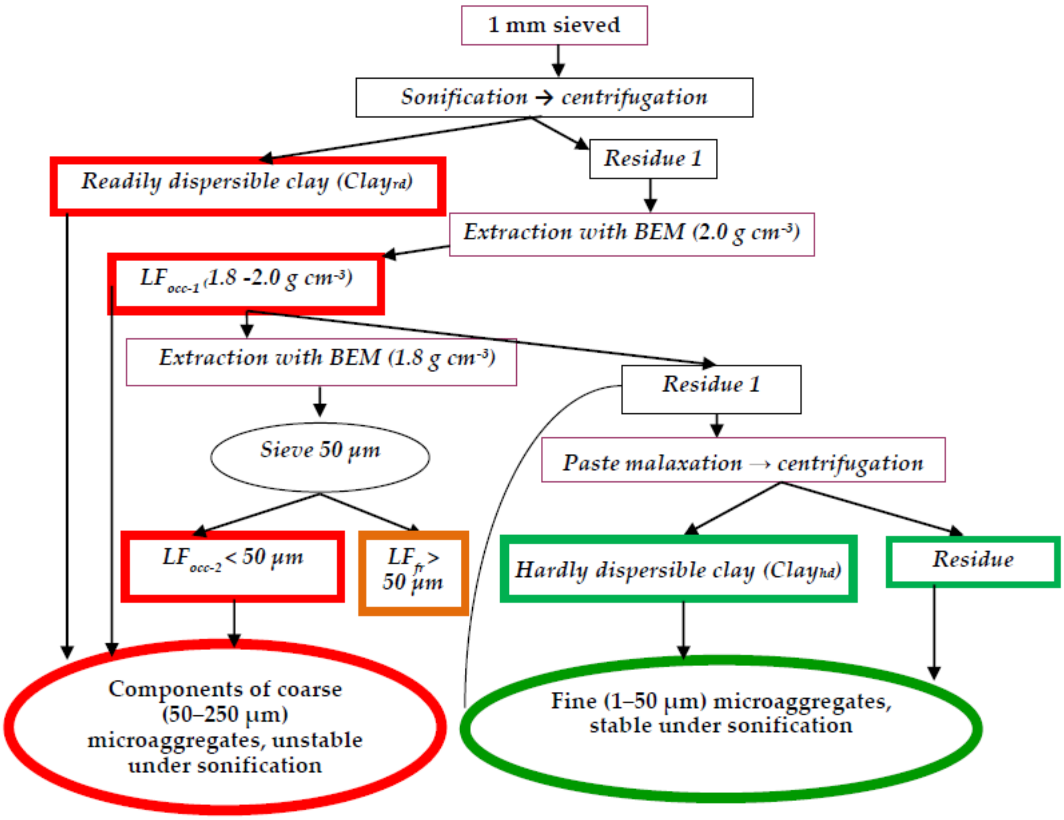

Figure 2. Soil fractionation scheme (simplified procedure).

After removal of the major clay fraction (readily dispersible clay ($Clay_{rd}$)) from the soil sample, the light fractions (densities of 2.0 and 1.8 $g \cdot cm^{-3}$) were isolated, according to a simplified scheme, by extracting with a bromoform-ethanol mixture (BEM) (Figure 2). Fractions with a density lower than 1.8 $g \cdot cm^{-3}$ were divided into two subfractions: light fractions containing particles larger than 50 μm—non-aggregated organic matter localized in the inter-aggregate space (LF_{fr}) and finer particles (<50 μm) localized within microaggregates (LF_{oc}). Isolation of free OM, after the removal of the clay (simplified procedure), was shown previously to have no major effect on the quantity and quality of OM, as compared to OM isolated prior to clay removal (according to the comprehensive scheme) [3,48]. All separations were performed in two replicates.

Hardly-dispersible clay ($Clay_{hd}$) (particles < 1 μm) was separated from the residue fraction using paste malaxation.

Hence, the applied methods allow the identification of five SOM pools: non-occluded (free) organic matter (LF_{fr}), occluded organic matter ($LF_{oc} = LF_{oc\text{-}1} + LF_{oc\text{-}2}$), readily- dispersible clay ($Clay_{rd}$), hardly-dispersible clay ($Clay_{hd}$) and residue (Res) (Figure 2).

Coarse (50–250 μm) microaggregates, unstable under sonification, consist of variably-humified organic residues (LF_{oc}) and clay particles ($Clay_{rd}$). The connection between the components is loose, and they are readily disconnected under sonification.

Fine (1–50 μm) microaggregates, stable under sonification, consist of clay particles ($Clay_{hd}$) and organic Res.

The carbon content in the soil and fractions was determined in duplicate using Tyurin's method. Statistical data processing was carried out using Microsoft Excel 2010 (Microsoft Corporation, Redmond, WA, USA) and OriginPro 8 (OriginLab Corporation, Northampton, MA, USA). The selected level of significance was $p < 0.01$.

3. Results and Discussion

3.1. Share of Microaggregates in Native and Arable Soils

On average, the share of coarse microaggregates in the studied zonal soils varies from 13%–33% of the microaggregated bulk soil and increases in the order of Albeluvisols < Phaeozem < Chernozems (Table 3).

Table 3. Share of microaggregates in the upper horizons of native and arable soils (mean ± standard error) and the Student criterion between pairs of native/arable soils.

Soil	Land Use	Unstable (Coarse) Microaggregates (50–250 μm)		Stable (Fine) Microaggregates (1–50 μm)		Microaggregates Total	
		% of Soil Bulk	Student Criterion	% of Soil Bulk	Student Criterion	% of Soil Bulk	Student Criterion
Albeluvisols	Forest Arable	13.38 ± 1.95 13.34 ± 2.56	0.02 < t = 2.10	85.48 ± 2.26 86.16 ± 2.72	0.08 < t = 2.10	98.86 ± 0.73 99.49 ± 0.59	1.33 < t = 2.10
Phaeozems	Forest Arable	20.04 ± 0.32 19.22 ± 0.50	1.35 < t = 4.30	79.34 ± 1.06 80.70 ± 2.08	0.93 < t = 4.30	99.38 ± 0.32 99.92 ± 0.77	0.63< t = 4.30
Chernozems	Steppe	33.49 ± 5.31	0.97 < t = 2.23	66.15 ± 5.51	1.06 < t = 2.23	99.64 ± 0.36	1.46 < t = 2.23

t, Student criterion are given for $p = 0.95$.

The comparison of the average contents of coarse and fine microaggregates in studied soils using a t-test did not reveal any significant statistical differences (Table 3).

The abundance of coarse microaggregates is dependent on the particle size composition of soil: an increase of fine particles is accompanied by growth of unstable (coarse) microaggregates. The abundance of coarse microaggregates increases when going from low loamy to clay loamy soils. The share of this group of microaggregates is 2.1-times higher in medium-loamy soils, relatively to low-loamy (Albeluvisols) and 1.3-times higher in clay-loamy, relative to medium-loamy (Chernozems).

This is confirmed by the close link between the share of sand-sized microaggregates and the content of clay particles in these soils. The coefficient of correlation for sand-sized microaggregates is R^2 = 0.98, at $p < 0.01$ (Figure 3; Table 4).

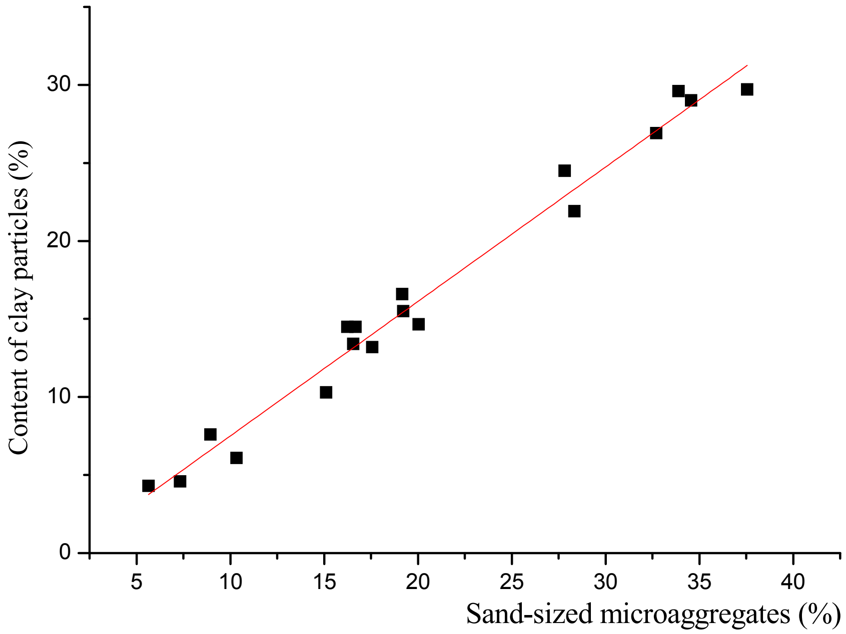

Figure 3. Correlation between sand-sized microaggregates and the content of clay particles ($Clay_{rd}$) in the studied soils.

Table 4. The linear equation coefficients with standard errors (SE), the Pearson coefficient (*r*), the root mean square error (RMSE) and the coefficient of determination (R^2) built for the dependence of the content of clay particles (%) on the sand-sized microaggregates content (SSMA,%).

Number of Samples (*N*)	*m*	SE_m	*b*	SE_b	*r*	R^2	RMSE
18	0.86	0.031	−1.11	0.69	0.9899	0.9786	1.25

Clay = *m*·SSMA + *b*. Significance: $p < 0.01$.

3.2. Carbon Concentration in Organic and Organo-Clay Fractions of Native and Arable Soils

According to the analysis data, the C concentration in the studied fractions, the maximum values (38%–47% C in fraction, on average) were obtained for the free OM localized in the inter-aggregate space (LF_{fr}) (Table 5).

These findings are in accordance with previous data [31,54]. The C concentration of LF_{oc} is 1.3–2.4-times lower than that of the free OM. The carbon concentration varies from 3.9%–13.5% in organo-clay fractions of coarse ($Clay_{rd}$) and from 5.7%–12.1% of fine ($Clay_{hd}$) microaggregates. However, only the differences between the native and arable Albeluvisols for LF_{oc} and Res and between the native and arable Chernozems for $Clay_{hd}$ and Res were statistically significant. We observed a close link between the content and carbon concentration of clay particles ($Clay_{rd}$) in unstable microaggregates: the coefficient of correlation is R^2 = 0.79, at $p < 0.01$ (Figure 4; Table 6).

Table 5. C concentration in organic and clay fractions extracted from the upper horizons of the studied native and arable soils.

Soil	Land Use	LF_{fr}		Unstable (Coarse) Microaggregates (50–250 μm)				Stable (Fine) Microaggregates (1–50 μm)			
				LF_{oc}		$Clay_{rd}$		$Clay_{hd}$		Res	
		% C in fraction (Mean ± Standard Error)	Student Criterion	% C in Fraction (Mean ± Standard Error)	Student Criterion	% C in Fraction (Mean ± Standard Error)	Student Criterion	% C in Fraction (Mean ± Standard Error)	Student Criterion	% C in Fraction (Mean ± Standard Error)	Student Criterion
Albeluvisols	Forest Arable	40.69 ± 1.74 38.08 ± 2.68	1.66 < t = 2.12	23.11 ± 2.24 23.22 ± 3.86	**2.28** > t = 2.01	8.93 ± 1.80 6.65 ± 1.87	1.32 > t = 2.10	8.80 ± 1.74 7.07 ± 1.62	1.43 < t = 2.23	0.65 ± 0.22 0.30 ± 0.10	**2.90** > t = 2.23
Phaeozems	Forest Arable	39.75 ± 1.47 47.15 ± 3.63	**3.71** < t = **4.30**	18.59 ± 0.52 20.97 ± 1.90	1.01 < t = 4.30	6.05 ± 0.29 7.00 ± 0.39	**0.25** > t = **4.30**	n.d. the same	n.d.	n.d. the same	n.d.
Chernozems	Steppe Arable	38.23 ± 3.98 42.01 ± 2.71	1.54 < t = 2.23	24.07 ± 5.86 21.81 ± 1.46	2.03 < t = 2.23	5.86 ± 0.65 5.75 ± 0.51	0.27 > t = 2.23	10.95 ± 1.33 7.18 ± 1.02	**4.4** > t = 2.45	3.87 ± 0.17 3.06 ± 0.22	**5.72** > t = 2.45

Note: The critical values of the Student t criterion are given for p = 0.95; bold type for t–criterion characterizes the significant differences, on average; n.d., not determined.

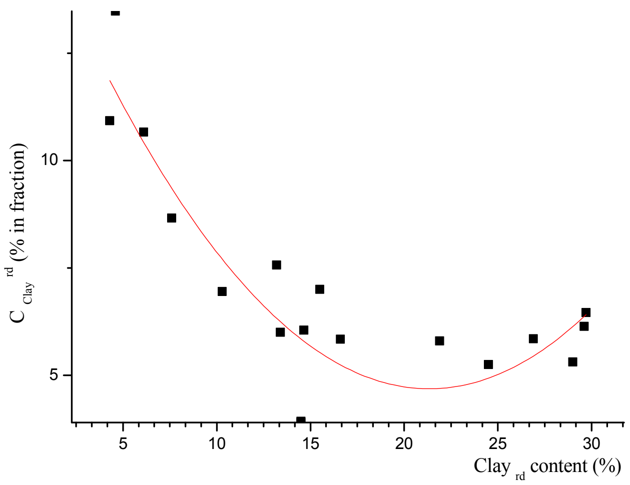

Figure 4. Correlation between the $Clay_{rd}$ content and carbon concentration (${C_{Clay}}^{rd}$) of unstable (coarse) microaggregates.

Table 6. Coefficients of the polynomial statistical model y = B0 + B1 x + B2 x^2.

	B	Value	Standard Error
Intercept	B0	15.94	1.18
x	B1	−1.05	0.15
x^2	B2	0.02	0.004

x = $Clay_{rd}$ (%); y = ${C_{Clay}}^{rd}$ (% in fraction). Significance: $p < 0.01$.

The lowest C concentration is characteristic for the Res fraction.

3.3. Variations in Organic Matter Content in Soils under a Change of Land Use

Analysis of the data revealed a significant difference between native and arable soils in terms of the content of both soil total carbon and carbon of all studied organic and organo-clay fractions (Table 7).

Plowing causes a significant decrease of free OM. On average, the content and share of non-occluded (free) organic matter (LF_{fr}) in arable Albeluvisols are more than nine and 4.3-times lower than that of native soils, respectively. In arable Phaeozem, the content and share of free organic matter are 7.5- and 4.5-times lower than that of native soils, respectively, and in arable Chernozems are 3.3 and 2.5-times lower than that of native soils, respectively (Table 7). The differences were statistically significant in all studied soils, except Phaeozems (Table 8). Therefore, the LF_{fr} accumulation level is 3–9-times lower in the arable soils than in the native soils. This is due to the greater accessibility of free OM (LF_{fr}) for microorganisms relative to the physically-defended OM (LF_{oc}) and, also, to the difference in the amount of plant residues incorporated into the soil matrix. The drop of the free organic matter, as a result of plowing in various types of soils, has been reported [2,3,12,23,33,55].

The major part of the carbon in the studied arable soils (>85%–97% of soil total C) is involved in the formation of microaggregates. The carbon content, stabilized in coarse (unstable under sonification) and fine (stable under sonification) microaggregates is described in Table 7. As a result of plowing, the C content decreases, both in coarse and fine microaggregates, and it affects the ratio of OM stabilized within them.

Microaggregates are formed by both organic and organo-clay compounds [39,56,57]. The organic phase of coarse microaggregates (unstable under sonification) includes a dispersed mixture of residues with different degrees of mineralization, which forms a nucleus, and organo-clay particles forming a shell [19,31,38,45]. The encrustation of SOM in the center of the microaggregates is the basic pathway to SOC sequestration [31,41]. The components of unstable microaggregates are relatively loosely bound together, as microaggregates disintegrate, from the impacts of sonification, into separate components (organo-clay particles ($Clay_{rd}$) and discrete OM particles (LF_{oc})). On average, the discrete OM (LF_{oc}) and organo-clay particles in studied native soils bind about 50% of the soil total carbon of unstable microaggregates. The carbon content of organic and organo-clay fractions in unstable (coarse) microaggregates is almost the same in the upper horizons of native soils: the ratio ${C_{LF}}^{oc}/{C_{Clay}}^{rd}$ is for Albeluvisols (1.1 ± 0.3), Phaeozem (0.8 ± 0.1) and Chernozems (1.0 ± 0.0) (Table 9).

As a result of plowing, the carbon content in organic fractions (LF_{oc}), which form unstable microaggregates, decreases more significantly relatively to that in organo-clay fractions ($Clay_{rd}$) [57]. The carbon content in aggregated organic fractions (LF_{oc}) in native soils is twice (in Albeluvisols) and one and a half times (in Phaeozem and Chernozems) as high as that of arable soils. In arable soils, the share of discrete OM particles (LF_{oc}) in the total carbon of this group of microaggregates decreases to 34%–39%, while the share of organo-clay particles ($Clay_{rd}$) increases to 61%–66% of the total C in unstable microaggregates (Table 7). The differences were statistically significant (Table 8). These changes in the carbon contents of the components affect the ratio ${C_{LF}}^{oc}/{C_{Clay}}^{rd}$, which became: Albeluvisols and Chernozems (0.6 ± 0.1) and Phaeozem (0.5 ± 0.1). The differences were statistically significant (Table 9).

On average, the share of free organic matter varies quite widely (from 2% to 8%–10% of soil total C) in the humus horizons of studied soils from the forest and steppe ecosystems and shows a tendency to decrease as one moves from north to south, which indicates the more intensive mineralization of free OM in steppes (Chernozems) as compared to the south taiga (Albeluvisols and Phaeozem) because of the high biological activity and favorable climatic conditions for the former type of soil (increasing mean annual air temperatures and mean annual evaporations, as well as decreasing mean annual precipitations from taiga to steppes) (Tables 1 and 10) [58–60].

On average, the share of aggregated organic matter (LF_{oc}) varies from 26%–33% of soil total C in the upper horizons of studied native soils (Table 10). In arable soils, the share of discrete OM (LF_{oc}) particles in the soil total carbon decreases (by 10%–14% from initial values, relative to native soils), and the share of organo-clay particles ($Clay_{rd}$) increases (by 1.4–1.6-times), correspondingly (Table 10). On average, the organo-clay complexes (both in unstable and stable microaggregates) contain 38%–54% of soil total carbon. This part of the organic matter is stabilized in soil owing to its inclusion into microaggregates and the organo-mineral interactions. On average, the share of organo-clay fraction ($Clay_{rd}$) in the total carbon of unstable microaggregates is 1.5–4.7-times higher than that in stable ones ($Clay_{hd}$) (Table 10).

The ratio between carbon in the unstable and stable microaggregates ($C_{unstable}/C_{stable}$) in the studied native soils shows a tendency to decrease from north to south and follows the order of Albeluvisols (2.2 ± 0.4) and Phaeozem (2.2 ± 0.7) > Chernozems (1.0 ± 0.1) (Table 9). It is notable that the calculations were made for samples taken from native soils; hence, the data given below characterize the soils in an equilibrium state. In arable soils, this ratio increases to: Albeluvisols (3.0 ± 0.4) and Phaeozem (3.2 ± 0.2) > Chernozems (1.5 ± 0.2). The differences were statistically significant in all studied soils, except Phaeozems (Table 9). The constancy of this ratio ($C_{unstable}/C_{stable} = 1$) for native Chernozems with different textures apparently points to their equilibrium state and indicates the closed type of carbon cycle, typical for natural steppe cenoses. Taking that into account, in the most balanced soils (native Chernozems), which are characterized by a closed carbon cycle, the ratio of carbon in the unstable and stable microaggregates is 1.0, so we can assume that the procedure of successive fractionalization (per minute, summary time: 15 min) using low intensity sonification (60–70 $J \cdot mL^{-1}$) of the aqueous suspension of soils allows us to divide the soil by unstable and stable microaggregates adequately, in order to assess the balance between the processes of humification and mineralization, assuming that unstable microaggregates represent mostly active soil organic matter and stable, passive OM. Therefore, we propose the ratio between carbon within the unstable and stable microaggregates ($C_{unstable}/C_{stable}$) for the characterization of the equilibrium status of soil organic matter in different ecosystems.

Table 7. C content (% of soil bulk) (mean ± standard error) in organic and organo-clay fractions extracted from the upper horizons of the studied native and arable soils.

Soil	Land Use	C_{total} (%)	LF_{fr} (% of Soil Bulk)	Unstable (Coarse) Microaggregates (50–250 µm)				Stable (Fine) Microaggregates (1–50 µm)			
				LF_{oc}		$Clay_{rd}$		$Clay_{hd}$		Res	
				% of Soil Bulk	% of C_{total} Microaggregates	% per Soil Bulk	% of C_{total} Microaggregates	% of Soil Bulk	% of C_{total} Microaggregates	% of Soil Bulk	% of C_{total} Microaggregates
Albeluvisols	Forest	2.72 ± 0.36	0.28 ± 0.02	0.88 ± 0.12	53.44 ± 4.26	0.76 ± 0.09	46.56 ± 4.26	0.25 ± 0.07	32.82 ± 9.55	0.46 ± 0.22	67.18 ± 9.55
	Arable	1.43 ± 0.21	0.03 ± 0.01	0.41 ± 0.05	38.86 ± 2.60	0.65 ± 0.11	61.14 ± 2.60	0.14 ± 0.03	37.34 ± 8.23	0.25 ± 0.07	62.66 ± 8.23
Phaeozems	Forest	2.79 ± 0.08	0.23 ± 0.04	0.78 ± 0.06	44.89 ± 2.34	0.96 ± 0.02	55.11 ± 2.34	0.82 ± 0.08			
	Arable	2.20 ± 0.03	0.04 ± 0.01	0.55 ± 0.10	33.72 ± 6.76	1.09 ± 0.13	66.28 ± 6.76	0.52 ± 0.04			
Chernozems	Steppe	6.08 ± 1.01	0.13 ± 0.07	1.58 ± 0.27	49.99 ± 1.20	1.58 ± 0.24	50.01 ± 1.20	1.18 ± 0.28	37.05 ± 5.81	1.97 ± 0.11	62.95 ± 5.81
	Arable	4.35 ± 0.75	0.04 ± 0.01	0.99 ± 0.24	38.14 ± 4.59	1.56 ± 0.19	61.86 ± 4.59	0.42 ± 0.14	20.00 ± 6.29	1.64 ± 0.11	80.00 ± 6.29

Table 8. Student criterion determining the degree of difference between the average values of parameters between pairs of native/arable soils.

Soil	C_{total} (%)	LF_{fr} (% of Soil Bulk)	Unstable (Coarse) Microaggregates (50–250 µm)				Stable (Fine) Microaggregates (1–50 µm)			
			LF_{oc}		$Clay_{rd}$		$Clay_{hd}$		Res	
			% of Soil Bulk	% C_{total} Microaggregates	% of Soil Bulk	% C_{total} Microaggregates	% of Soil Bulk	% C_{total} Microaggregates	% of Soil Bulk	% C_{total} Microaggregates
Albeluvisols	**6.00 > *t* = 2.1**	**4.86 > *t* = 2.1**	**6.88 > *t* = 2.1**	**5.73 > *t* = 2.1**	1.53 < *t* = 2.1	**5.73 > *t* = 2.1**	**3.0 > *t* = 2.23**	0.70 < *t* = 2.23	**2.29 > *t* = 2.23**	0.70 < *t* = 2.23
Phaeozems	**7.29 > *t* = 4.3**	**6.72 > *t* = 4.3**	3.31 < *t* = 4.3	**5.15 > *t* = 4.3**	1.92 < *t* = 4.3	**5.15 > *t* = 4.3**	**6.71 > *t* = 4.3**			
Chernozems	**2.94 > *t* = 2.23**	**2.52 > *t* = 2.23**	**3.28 > *t* = 2.23**	**4.90 > *t* = 2.23**	0.03 < *t* = 2.23	**4.90 > *t* = 2.23**	**4.73 > *t* = 2.45**	**3.90 > *t* = 2.45**	**4.12 > *t* = 2.45**	**3.90 > *t* = 2.45**

Note: The critical values of the Student *t* criterion are given for $p = 0.95$; bold type for *t* characterizes the significant differences, on average.

Table 9. The ratios C_{LFoc}/C_{Clayrd} and $C_{unstable}/C_{stable}$ (mean ± standard error and Student criterion, determining the degree of difference between the average values of parameters between pairs of native/arable soils) in the studied soils.

Soil	Land Use	$C_{LF}{}^{oc}/C_{Clay}{}^{rd}$		$C_{unstable}/C_{stable}$	
		Mean ± Standard Error	Student Criterion	Mean ± Standard Error	Student Criterion
Albeluvisols	Forest Arable	1.1 ± 0.3 0.6 ± 0.1	**5.0** > *t* = 2.10	2.2 ± 0.4 3.0 ± 0.4	**3.1** > *t* = 2.10
Phaeozems	Forest Arable	0.8 ± 0.1 0.5 ± 0.1	**5.0** > *t* = 4.30	2.2 ± 0.7 3.2 ± 0.2	2.8 < *t* = 4.30
Chernozems	Steppe Arable	1.0 ± 0.0 0.6 ± 0.1	**5.3** > *t* = 2.23	1.0 ± 0.1 1.5 ± 0.2	**3.7** > *t* = 2.23

Note: The critical values of the Student *t* criterion are given for $p = 0.95$; bold authentic values of *t* characterize the significant differences, on average.

Table 10. Share of C content in organic and organo-clay fractions in the upper horizons of native and arable soils (mean ± standard error) and the student criterion between pairs of native/arable soils.

Soil	Land Use	LF_{fr}		Unstable (Coarse) Microaggregates (50–250 μm)				Stable (Fine) Microaggregates (1–50 μm)			
				LF_{oc}		$Clay_{rd}$		$Clay_{hd}$		Res	
		% of Total Soil C (Mean ± Standard Error)	Student Criterion	% of Total Soil C (Mean ± Standard Error)	Student Criterion	% of Total Soil C (Mean ± Standard Error)	Student Criterion	% of Total Soil C (Mean ± Standard Error)	Student Criterion	% of Total Soil C (Mean ± Standard Error)	Student Criterion
Albeluvisols	Forest Arable	9.89 ± 2.96 2.28 ± 1.12	**5.39** > *t* = **2.10**	32.75 ± 3.74 28.47 ± 1.83	2.02 < *t* = 2.10	28.41 ± 2.99 44.87 ± 2.47	2.02 < *t* = 2.10	9.43 ± 2.42 9.44 ± 2.05	0.01 < *t* = 2.23	19.85 ± 3.91 16.06 ± 3.15	1.48 < *t* = 2.23
Phaeozems	Forest Arable	8.26 ± 1.64 1.83 ± 1.04	3.21 < *t* = 4.30	27.99 ± 1.30 25.10 ± 4.93	1.44 < *t* = 4.30	34.36 ± 1.65 49.40 ± 5.26	1.44 < *t* = 4.30	n.d.	n.d.	n.d.	n.d.
Chernozems	Steppe Arable	2.06 ± 1.09 0.84 ± 0.32	2.09 < *t* = 2.23	26.03 ± 0.47 22.39 ± 2.33	**3.00** > *t* = **2.23**	26.07 ± 1.34 36.50 ± 3.71	**3.00** > *t* = **2.23**	17.07 ± 2.58 8.34 ± 2.72	**4.56** > *t* = **2.45**	29.05 ± 3.00 33.25 ± 2.40	**3.00** > *t* = **2.45**

Note: Bold authentic values of *t* characterize the significant differences, on average. n.d., not determined.

4. Conclusions

The variations in organic matter content related to the change of land use depend on OM localization and soil stabilization mechanisms. More than 90% of soil organic matter is stabilized within microaggregates. Under plowing, carbon content decreased, both in unstable (coarse) and stable (fine) microaggregates. This is commonly related to the mineralization of the organic constituents and is caused by the cardinal disturbance of an essential condition of the sustainable renewal of the organic matter content: the continuous input of an adequate amount of fresh organic matter.

The accumulation level of free OM, localized in the inter-aggregate space, in the studied native soils shows a tendency to decrease from north to south, across the north-south geographical gradient, decreasing in order as: Albeluvisols > Phaeozem > Chernozems. Plowing causes a significant decrease of free OM. The carbon losses of the occluded OM (LF_{oc}) within the unstable microaggregates were much lower. Readily dispersible clay ($Clay_{rd}$), another component of unstable microaggregates, demonstrates the lowest decrease in carbon content, due to the protection of organic matter from mineralization owing to both inclusion into microaggregates and organo-mineral interactions.

The carbon content of organic and organo-clay fractions in unstable (coarse) microaggregates ($C_{LF}{}^{oc}/C_{Clay}{}^{rd}$) is almost identical in the humus layers of native soils: Albeluvisols (1.1), Phaeozem (0.8) and Chernozems (1.0). Under plowing, the ratio $C_{LF}{}^{oc}/C_{Clay}{}^{rd}$ became: Albeluvisols and Chernozems (0.6) and Phaeozem (0.5).

The shares of carbon accumulated within the unstable (coarse) and stable (fine) microaggregates ($C_{unstable}/C_{stable}$) of native soils were constant and showed a tendency to decrease from north to south, in the order of Albeluvisols (2.2) and Phaeozem (2.2) > Chernozems (1.0). Under plowing, the ratio ($C_{unstable}/C_{stable}$) increased to: Albeluvisols (3.0) and Phaeozem (3.2) > Chernozems (1.5).

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