

The Mineral Composition of Clay Subfractions in the Rhizosphere of Norway Maple (*Acer platanoids* L.) and in the Enclosing Soil

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Abstract—Fractionation of the clay fraction was carried out and the mineral composition of clay and its subfractions in the soil of the rhizosphere of the Norway maple (*Acer platanoides* L.) and in the bulk soil from the AE_{Loa} horizon of podzolic soil was studied. It was found that the clay fraction is dominated by a 1–0.2 μm subfraction (56% in the rhizosphere of maple and 60% in the bulk soil) and contains 20% of 0.2–0.06 μm subfraction in the rhizosphere and 27% of this fraction in the bulk soil. The content of 0.06–0.02 and <0.02 μm subfractions did not exceed 10%. A significant increase was revealed in the proportion of 0.06–0.02 μm subfraction and a decrease in the proportion of <0.02 μm subfraction in the rhizosphere compared with the bulk soil. The size fractions of clay both in the rhizosphere and in the bulk soil are differentiated by their mineral composition. The 1–0.2 μm fraction is dominated by kaolinite; the proportion of micas increased and the proportion of kaolinite decreased in the 0.2–0.02 μm fraction, while in the <0.02 μm fraction the proportion of labile minerals significantly increases. There was no significant difference in the mineral composition of the clay fraction and the 1–0.2 μm subfraction. It was found that the 0.2–0.02 μm subfraction in the rhizosphere contains significantly more labile minerals compared to the bulk one, which are a result of intensively occurring mica transformation processes into labile structures in the rhizosphere.

Keywords: clay minerals, podzolic soils, X-ray diffractometry, soil organic matter, rhizospheric processes

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INTRODUCTION

The silt fraction of the soil is a polydisperse system, including particles of different sizes—from 1 μm on the Kachinsky scale (from 2 μm on the Atterberg scale) to several nanometers. Subfractions of silt, like larger granulometric fractions of soil, are differentiated by mineral composition. It is known that with a decrease in particle size, the total content of Si, Ca, and Na decreases, while the content of Al, Fe, Mg, and K increases. For Mg, K, Ca, and Na, this pattern is also observed for soil-forming rocks (except carbonate rocks) and is associated, first of all, with the differentiation of particles by mineral composition (Rode, 2008). In podzolic soils, with a decrease in the particle size in the clay fraction, the proportion of minerals with a labile structure increases, mainly in the form of mixed-layer structures, and the degree of crystallization of minerals decreases (Sokolova, 1985; Simonov, 1992). Similar patterns of distribution of layered aluminosilicates across different particle size fractions were found in Cambisols (Hubert et al., 2012). In the fraction of 2–0.2 μm isolated from horizon S₂, the content of minerals with a labile crystal lattice is small, and the finest subfraction <0.05 μm is represented

mainly by mixed-layer structures: ordered kaolinite–illite (71%), disordered mica–smectite (11%), and disordered illite–smectite–chlorite (18%). For the A horizons of Alfisols, it was shown that the mineral composition of the 2–0.1 μm fraction is generally similar to the mineral composition of particles <2 μm in size, but differs significantly from the mineral composition of the 0.1–0.025 μm fraction (Zhang et al., 2008). The authors showed that trioctahedral micas were contained mainly in large fractions (up to 0.45 μm), while dioctahedral structures predominated in finer fractions. The distribution of minerals by size fractions can be either inherited from the parent rock or a consequence of weathering, as a result of which mineral particles are crushed and become part of finer fractions (Sokolova et al., 2005; April, Keller, 1990).

In podzolic soils, clay minerals dissolve and transform with the formation of intermediate transformation products, represented mainly by mixed-layer layered aluminosilicates. The direction and depth of such transformations depend on the acid–base and oxidation–reduction conditions in the soil, on the ionic strength and chemical composition of the soil solution, and on the conditions for the outflow of dissolu-

tion products and transformation changes in minerals. The most intensive processes of transformation of clay minerals in the soil occur in the rhizosphere, the volume of soil adjacent to the roots, which is characterized by specific properties (Sokolova, 2015) and in which continuous interaction of its biotic and abiotic components occurs. Weathering of minerals in the rhizosphere is one of the main sources of plant nutrients (Calvaruso et al., 2009). This process is considered a key reason for the resilience of forest ecosystems (Uroz et al., 2009). Knowledge of how individual species and their communities influence mineral weathering is important for predicting the long-term development and productivity of ecosystems (Bormann et al., 1998). Despite the key role of rhizospheric processes in the functioning of terrestrial ecosystems (including forests), the differences in the mineral composition of the rhizosphere from the soil of the nonrhizosphere space and the reasons for their formation have not been sufficiently studied, especially for the rhizosphere of broad-leaved tree species. Taking into account the differentiation of mineral composition by silt subfractions and the different resistance of clay minerals to weathering, in order to form a more complete and objective understanding of the processes of transformation of clay minerals in the rhizosphere, it is necessary to study their composition not only in the silt fraction ($<1\ \mu\text{m}$), but also in its subfractions.

The purpose of this study is to conduct a comparative analysis of the content and mineral composition of 1–0.2, 0.2–0.06, 0.06–0.02, and $<0.02\ \mu\text{m}$ granulometric subfractions of silt in the rhizosphere of Norway maple and in the enclosing podzolic soil.

MATERIALS AND METHODS

The objects of study were samples of the AE_{Lo} horizon from the rhizosphere of Norway maple (*Acer platanoides* L.) (hereinafter, “maple”) and from the enclosing podzolic soil, selected on the territory of the Central Forest Reserve (Nelidovsky raion, Tver oblast). In one of the areas with a high proportion (30–40%) of maple in the tree layer with trees aged from 15 to 30 years, a sampling site of about $50\ \text{m}^2$ in size was selected², with sampling carried out in five replicates. The separation of the clay fraction was carried out using the sedimentation method (Aydinyan, 1960) without preliminary chemical treatment with double kneading of the soil. The sludge suspensions were coagulated with a 1 M CaCl_2 solution and had excess chloride ions washed off by dialysis. The separation of sludge subfractions (1–0.2, 0.2–0.06, 0.06–0.02, and $<0.02\ \mu\text{m}$) was carried out by ultrasonic treatment and centrifugation after preliminary treatment of the sludge fraction with a 10% H_2O_2 solution and according to Mera–Jackson (Laird, Dawdy, 1994). The mineral composition of the clay fraction and its subfractions was determined by X-ray diffrac-

tometry. X-ray diffraction patterns from oriented preparations were obtained on a DRON-3 diffractometer using Cu-K_α radiation filtered by Ni and a voltage and current in the tube of 35 kV and 20 mA, respectively. The content of individual groups of clay minerals was calculated using the modified Kornblum method (Sokolova et al., 2005). Comparison of means was performed using the Wilcoxon test. The Kruskal–Wallis test was used for multiple comparisons of means. When testing statistical hypotheses, a 5% significance level was chosen.

RESULTS

Content of Silt Subfractions in the Silt Fraction

The proportion of the clay fraction isolated from the soil samples in the maple rhizosphere was $6.5 \pm 1.1\%$, which is significantly less than in the enclosing soil ($7.3 \pm 0.6\%$).

The clay fraction is more than half represented by the 1–0.2 μm subfraction (56% in the maple rhizosphere and 60% in the enclosing soil) and approximately a third by the 0.2–0.06 μm subfraction (20% in the maple rhizosphere and 27% in the enclosing soil). The content of two finer subfractions does not exceed 10% on average (Fig. 1). A significant increase in the 0.06–0.02 μm subfraction was revealed in the rhizosphere compared to the enclosing soil. The content of the subfraction $<0.02\ \mu\text{m}$ in the rhizosphere is significantly less compared to the enclosing soil. For the remaining fractions, no significant differences were found between the rhizosphere and the enclosing soil.

Mineral Composition of the Silt Fraction and Silt Subfractions

X-ray diffraction patterns of air-dried preparations of samples of the clay fraction as a whole and of subfractions of clay from the rhizosphere and enclosing soil are similar to each other. Figure 2 shows the X-ray patterns of the clay fraction and clay subfractions of the enclosing soil; they are similar to the X-ray patterns of the rhizosphere and vary uniformly with particle size and treatment (ethylene glycol saturation and calcination). The X-ray diffraction patterns of air-dry samples of the clay fraction as a whole and of the 1–0.06 μm subfractions (Fig. 2a, curves 1–3) are visually indistinguishable: all of them contain a symmetrical intense peak with a maximum at $2\theta = 12.4^\circ$ ($d/n\ 0.72\ \text{nm}$) and an asymmetrical peak in the range of $2\theta = 9.0$ – 5.5° (d/n in the region of 1.0–1.6 nm) in the air-dry state, shifting toward larger angles 2θ after calcination (Fig. 2c) and becoming bimodal upon saturation with ethylene glycol (Fig. 2b). The change in the position of the peaks on the X-ray patterns after the above treatments indicates the presence of minerals with a labile crystal lattice (vermiculite) and vermiculite and/or smectite layers in the composition of mixed-layer

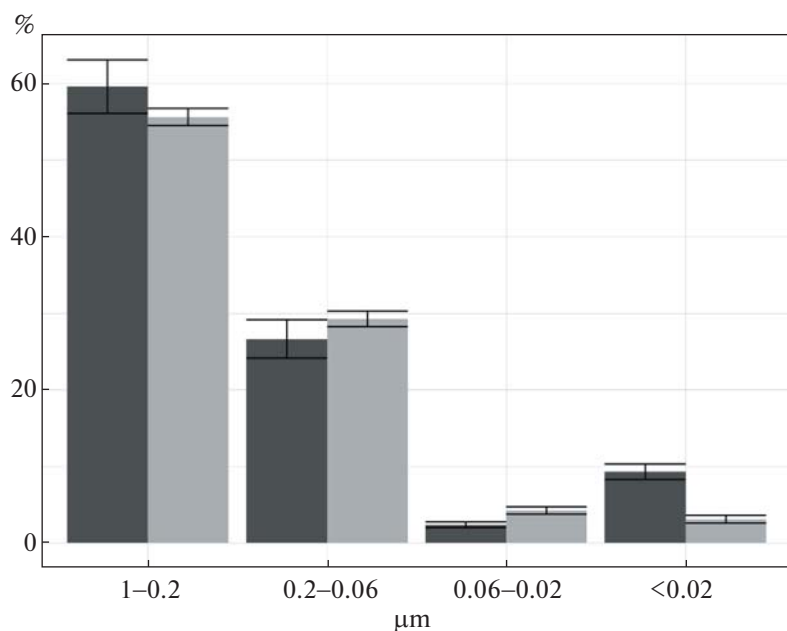


Fig. 1. Average content of clay fraction and clay subfractions in the composition of the enclosing soil (dark gray) and maple rhizosphere (light gray) (error bars show standard deviations).

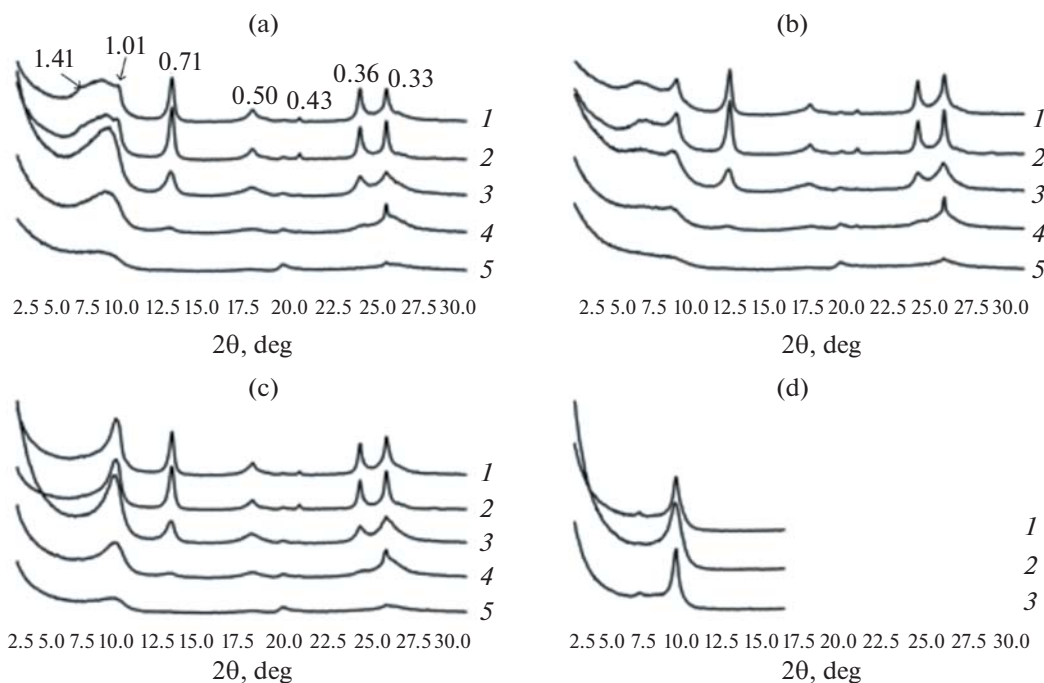


Fig. 2. X-ray diffraction patterns of the clay fraction (I) and subfractions of the enclosing soil clay (2–5: subfractions 1–0.2, 0.2–0.06, 0.06–0.02, and <0.02 μm , respectively). (a) Samples in air-dry condition; (b–d) saturated with ethylene glycol and calcined at 350 and 550°C, respectively. Numbers on curves—d/n, nm.

illite–vermiculites (smectites). The presence of a low-intensity peak with a maximum at $2\theta = 6.3^\circ$ (d/n 1.4 nm) on the radiographs of on preparations calcined at 550°C (Fig. 2d) indicates an insignificant content of chlorite group minerals in the samples.

With decreasing particle size, the intensity of the peaks in the X-ray diffraction patterns caused by the presence of illite ($2\theta = 9.0^\circ$) and kaolinite ($2\theta = 12.4^\circ$) in the samples decreases, while their asymmetry and width at half-height increase. This pattern is observed

Table 1. Content of the main groups of clay minerals in the silt fraction of the AELOa horizon (rhizosphere and enclosing soil) and in silt subfractions

Fraction, μm	Enclosing soil			Rhizosphere		
	mica	kaol. + chl.	lab. min.	mica	kaol. + chl.	lab. min.
<1	27 ± 3	55 ± 2	18 ± 3	27 ± 3	52 ± 7	21 ± 4
1–0.2	30 ± 2	54 ± 7	15 ± 7	29 ± 4	57 ± 7	14 ± 3
0.2–0.06	$56 \pm 4^*$	34 ± 1	11 ± 3	$51 \pm 2^*$	36 ± 2	13 ± 3
0.06–0.02	$68 \pm 4^*$	12 ± 3	$20 \pm 2^*$	$59 \pm 3^*$	15 ± 2	$25 \pm 2^*$
<0.02	71 ± 3	5 ± 3	24 ± 1	71 ± 10	6 ± 1	22 ± 11

kaol.—Kaolinite; chl.—chlorite; lab. min.—minerals with a labile crystal lattice; arithmetic mean $\pm$ standard deviation; * significant differences between the rhizosphere and the enclosing soil are marked with a sign.

both for the subfractions of silt in the enclosing soil (Fig. 2) and for the subfractions of silt in the rhizosphere soil (X-ray diffraction patterns are not provided). Most noticeably, with a decrease in the size of the subfractions, the intensity of reflection from kaolinite ($2\theta = 12.4^\circ$) decreases (Fig. 2a). Based on the change in the X-ray patterns of the sludge subfractions after saturation of the preparations with ethylene glycol and calcination, it can be concluded that with a decrease in the size of the subfractions, the content of mixed-layer minerals in their composition increases, which include vermiculite or smectite layers (Figs. 2b, 2c).

The composition of the silt fraction as a whole is dominated by kaolinite together with chlorite (59–70%) (Table 1). The weak peak at $2\theta = 6.3^\circ$ (d/n 1.4 nm) in the X-ray diffraction patterns of samples calcined at 550°C indicates the predominance of kaolinite over chlorite (Fig. 2d).

The share of minerals of the mica and illite groups in the composition of silt as a whole accounts for 19 to 32%. The content of minerals with a labile crystal lattice (vermiculite and mixed-layer illite–vermiculite or illite–smectite) ranges from 13 to 27% (Table 1). Quartz and feldspars are present in small quantities in the silt fraction (Fig. 2). No significant differences in the content of illite, kaolinite and minerals with a labile crystal lattice in the clay fraction as a whole were found between the rhizosphere and the enclosing soil (Table 1).

In the largest subfraction of silt (1–0.2 μm), the proportion of labile minerals in the rhizosphere was significantly lower compared to the silt fraction as a whole. In the 0.2–0.06 μm fraction, the proportion of mica group minerals increases significantly compared to larger fractions, while the proportion of kaolinite decreases, while the proportion of micas in the enclosing soil is significantly greater compared to the rhizosphere. The proportion of minerals with a labile crystal lattice does not change compared to the 1–0.2 μm fraction, but is significantly less compared to the clay fraction (Table 1).

In the 0.06–0.02 μm fraction, the relative contents of the three main groups of clay minerals change significantly compared to larger ones: the proportions of labile minerals and mica group minerals increase, while the proportion of kaolinite decreases. The differences between the rhizosphere and the enclosing soil for this fraction are expressed to the greatest extent: the proportion of micas in the rhizosphere is significantly less, and the proportion of minerals with a labile crystal lattice is significantly greater (Table 1).

In the composition of the finest fraction <0.02 μm , the proportion of kaolinite decreases compared to the 0.06–0.02 μm fraction both in the rhizosphere and in the enclosing soil, and the proportion of other minerals does not change (Table 1).

DISCUSSION

Content of Silt Subfractions in the Enclosing Soil and in the Rhizosphere

The content of the clay fraction in the enclosing soil and in the rhizosphere soil was determined without preliminary removal of organic matter from the soil, which is a structure and promotes the formation of microaggregates. Therefore, the rhizosphere soil, which contains more organic matter compared to the enclosing soil (Sokolova et al., 2019), contains significantly fewer particles <1 μm in size than the enclosing soil does. The revealed significant predominance of the silt subfraction with a particle size of 1–0.2 μm is consistent with the data obtained for podzolic soils by other researchers. The accumulation of the precolloidal (1–0.2 μm) fraction in the eluvial and overlying organomineral horizons of podzolic soils developed on loamy rocks is explained by the predominant destruction and removal down the profile of finely dispersed fractions as a result of the podzol formation process (Sokolova et al., 2005; Rode, 2008).

A significant increase in the content between the rhizosphere and the enclosing soil was revealed only for the 0.06–0.02 μm subfraction. These differences can be explained by the mechanical fragmentation of the largest particles of silt under the influence of the

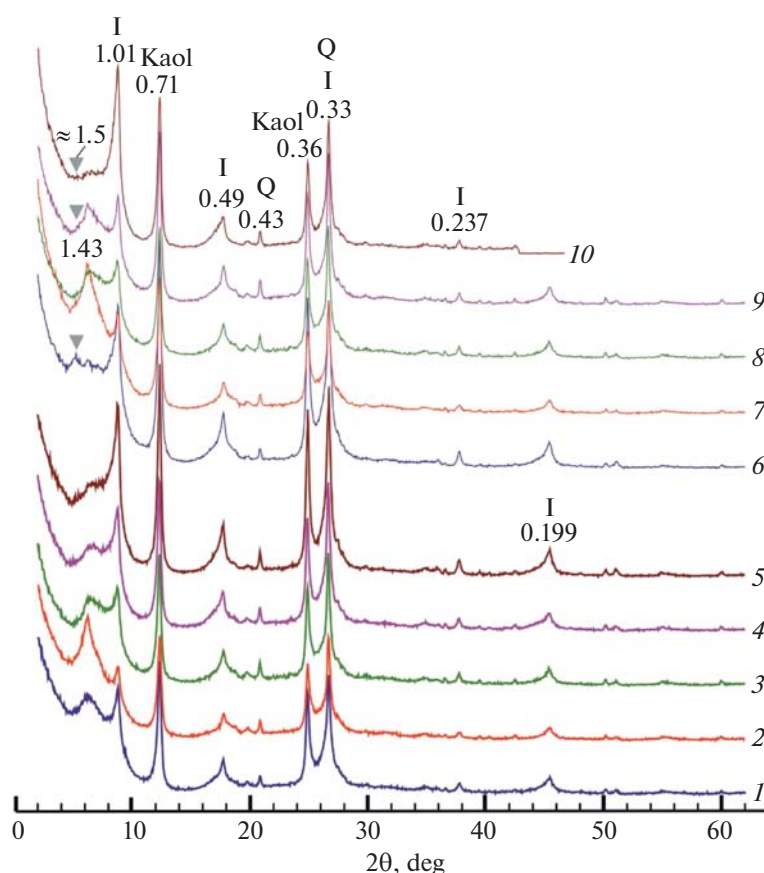


Fig. 3. X-ray diffraction patterns of ethylene glycol-saturated clay fractions of the enclosing soil (replicates 1–5) and rhizosphere soil (6–10). I—illite, Kaol—kaolinite, Q—quartz. Numbers on curves—d/n, nm.

functioning of roots and the microbial community to the size of colloidal particles (April, Keller, 1990) and transformational changes in minerals, accompanied by a decrease in the size of crystallites (Sokolova et al., 2005). It can be supposed that the identified significant decrease in the content of the subfraction $<0.02 \mu\text{m}$ in the rhizosphere compared to the enclosing soil is due to both the dissolution of the finest particles in the rhizosphere and their removal to the underlying horizons.

Mineral Composition of the Silt Fraction and Silt Subfractions

The mineral composition of the clay fraction in the AELOa horizon is typical for podzolic soils of the Central Siberian Polytechnical Reserve (Kiryushin et al., 2002; Sokolova et al., 2019) and indicates active processes of intrasoil weathering, leading to the relative accumulation of the most stable minerals of the kaolinite group in the clay fraction.

As the particle size decreases, the proportion of kaolinite group minerals in the silt fraction decreases. Weathering of clay minerals, including kaolinite, is accompanied by a decrease in particle size, but, since

kaolinite is one of the clay minerals most resistant to weathering and dissolves more slowly than other clay minerals (Sokolova et al., 2005), with a decrease in the size of silt subfractions, its share also decreases. Other factors that influence the weathering of minerals are the properties of dissolved and sorbed organic matter. Sorbed organic matter can have a multidirectional effect on the resistance of minerals to dissolution. Thus, the sorption of humic acid on kaolinite leads to a shift in the dehydroxylation effect towards lower temperatures, which may indicate partial destruction of the crystal lattice (Danilin et al., 2023). On the other hand, sorption of fulvic acids at $\text{pH} > 4.5$ can reduce the solubility of kaolinite due to the formation of strong polynuclear surface complexes (Ochs, 1996). Low-molecular organic acids intensify the dissolution of minerals (Sokolova, 2013, 2015), but since, all other things being equal, the kaolinite crystal lattice is most resistant to the effects of low-molecular organic acids and the specific surface area of kaolinite is smaller than that of minerals with a labile crystal lattice, these acids should not significantly affect the destruction of kaolinite. The influence of rhizospheric processes on the content of kaolinite in the clay fraction was not revealed in any of the size fractions considered. Thus,

the decrease in kaolinite content with decreasing particle size is due to the high resistance of kaolinite to weathering.

Micas, as one of the main sources of potassium for plants and soil microorganisms, are most intensively transformed into labile structures in the rhizosphere (Hinsinger et al., 1992; Calvaruso et al., 2006), as a result of which a significant decrease in their share in the composition of clay minerals with a parallel increase in the share of labile minerals is possible in the rhizosphere. However, this process is not irreversible: for finely dispersed fractions of soils, the possibility of the functioning of the illite–labile–minerals buffer system has been shown, in which the predominance of one or another group of minerals is determined by the dynamics of potassium activity in the soil solution (Sokolova et al., 2015). The influence of rhizospheric processes on the proportion of micas and illites is not expressed in the 1–0.2 μm fraction, but, in the 0.2–0.06 and 0.06–0.02 μm fractions, a significant decrease in the proportion of micas in the rhizosphere is observed compared to the enclosing soil. The method used in the work for calculating the relative content of clay minerals does not allow for a quantitative assessment of the content of mixed-layer minerals, which are both inherited from the parent rock and can form in soils as an intermediate product of transformation changes in micas. However, on radiographs such transformational changes can be seen at a qualitative level. Figure 3 shows X-ray diffraction patterns for five replicates of the clay fraction of the enclosing soil (curves 1–5) and five replicates of the rhizosphere soil (curves 6–10). In the X-ray diffraction patterns of the clay fractions, in three out of five replicates for ethylene glycol-saturated rhizosphere samples, differences are observed in the low-angle region compared to the X-ray diffraction patterns of the clay fraction of the enclosing soil in the form of a shift in diffuse scattering toward small angles in the range of $2\theta = 5\text{--}7^\circ$ and the appearance of an independent reflection with a maximum approximately corresponding to d/n 1.5 nm in one of the replicates (Fig. 3a, curves 1, 4–5). The differences revealed in the X-ray images indicate an increase in the number of mixed-layer structures containing vermiculite or smectite layers and a more advanced stage of vermiculitization of micas in the rhizosphere.

Labile minerals in the composition of the silt fraction and silt subfractions are represented mainly by vermiculite and mixed-layer illite–vermiculite and/or illite–smectite. In general, there is a relative accumulation of these minerals in the composition of fractions $<0.06 \mu\text{m}$. This fact can be explained by a decrease in the proportion of kaolinite group minerals with a decrease in the size of silt subfractions, the crushing of crystallites by roots and hyphae (April, Keller, 1990; Finlay, 2008) and the transformation of micas into mixed-layer structures and vermiculite, which is accompanied by a decrease in the size of crystallites, as

evidenced by a decrease in the intensity of the corresponding peaks and an increase in their half-width at half-height on X-ray diffraction patterns of subfractions $<0.06 \mu\text{m}$ (Fig. 2, curves 4, 5).

CONCLUSIONS

The rhizosphere soil of Norway maple in the AE_{Loa} horizon of podzolic soil contains fewer particles $<1 \mu\text{m}$ compared to the enclosing soil. The formation of microaggregates in the rhizosphere is facilitated by the increased content of organic matter compared to the nonrhizosphere space.

It was established that the 1–0.2 μm subfraction predominates in the clay fraction (56% in the maple rhizosphere and 60% in the enclosing soil); the content of the 0.2–0.06 μm subfraction was 20% in the maple rhizosphere and 27% in the enclosing soil. The content of other subfractions (0.06–0.02 μm and $<0.02 \mu\text{m}$) does not exceed 10%. A significant increase in the proportion of the 0.06–0.02 μm subfraction and a decrease in the proportion of the $<0.02 \mu\text{m}$ subfraction were found in the rhizosphere compared to the enclosing soil.

The size fractions of silt in both the rhizosphere and the enclosing soil are differentiated by mineral composition. In the composition of the fraction 1–0.2 μm , kaolinite predominates; in the composition of the fractions 0.2–0.02 μm , the proportion of micas increases and the proportion of kaolinite decreases; and, in the composition of the fraction $<0.02 \mu\text{m}$, the proportion of labile minerals increases significantly.

No significant difference was found in the mineral composition of silt as a whole and the 1–0.2 μm subfraction in the rhizosphere soil and in the enclosing soil; however, differences in the X-ray diffraction patterns of the silt fraction of samples saturated with ethylene glycol in the low-angle region indicate more intensive processes of transformation of micas into labile structures in the rhizosphere. The influence of the rhizosphere is most pronounced for the 0.2–0.02 μm subfraction, which is confirmed by a significant increase in the content of minerals with a labile crystal lattice in the rhizosphere compared to the enclosing soil.

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ETHICS APPROVAL AND CONSENT TO PARTICIPATE

This work does not contain any studies involving human and animal subjects.

CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

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