

The Study of Physical and Chemical Characteristics of Organo-Clay Complexes of the Chronosequence of Albic Retisols Using Dynamic Light Scattering and Phase Analysis Light Scattering

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Abstract—Dynamic light scattering (DLS) and phase analysis light scattering (PALS) have been used to study the physical and chemical characteristics of organo-clay complexes of soddy-podzolic soils (Albic Retisols (Loamic, Cutanic, Differentic, Ochric)) of forest sites of the Central Forest Reserve. The study of the chronosequence of abandoned agricultural soils afforested for 100 years showed that the value of the average diameter of the organo-clay complexes slightly decreased compared to that at the site with minimum duration of afforestation (5–7 years), but remained still higher than in the forest soils. Multiple linear regression statistical models were developed to predict the average diameter of clay particles. The best model ($r = 0.83$), where all parameters were significant ($P < 0.05$), included the content of clay and carbon concentration in clay. The chronosequence of postagrogenic soddy-podzolic soils demonstrated the proximity of the colloidal system stability, but in a peptization state, as evidenced by the values of zeta potential and average diameter of the clay particles.

Keywords: afforestation, aggregate stability, organo-clay complexes, average diameter of clay particles, zeta-potential

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INTRODUCTION

The adsorption to minerals is one of the natural mechanisms of soil organic matter (OM) protection from the biodegradation [23, 24]. Primary organo-mineral complexes—“primary structure of soil”—is a result of OM association with primary mineral particles. This kind of structural units can be isolated after a complete dispersion of soils [14]. The interaction of OM with mineral particles is a fundamental process in the upper horizons of soils. The OM stabilized by clay particles is defined as the OM adsorbed to the surface of clay minerals $< 1 \mu\text{m}$ in size and / or localized inside the $< 1 \mu\text{m}$ clay microaggregates [12, 37].

Soil particles exist throughout the soil matrix in a variety of sizes, shapes, and degrees of aggregation. The breakdown of soil into smaller aggregates and particles can be accomplished using ultrasonic equipment. The breakdown occurs when sufficient mechanical stresses are applied to overcome the attractive forces within the aggregates [30]. Ultrasonic dispersion of aggregated soils is usually conducted using high-intensity ultrasonic instruments [17, 18, 38]. It is important to note that ultrasonic dispersion of soil in water is a widely applied method of aggregate disruption and is

admitted as the most “soft” method of separation of mineral, organic, and organomineral particles—the components of soil microaggregates—without the danger of the destruction of primary particles [20, 30, 31].

The number, size, and stability of the aggregates in the soil vary under the impact of both natural and anthropogenic factors [7, 11, 15, 35].

Dynamic light scattering (DLS) is one of the modern research methods that can expand our understanding of the colloidal properties of soil particles of different sizes, as well as identification of the factors that have the greatest impact on them [8, 21, 25, 28, 29]. The measurement of diffusion coefficients of aqueous-clay suspensions by dynamic light scattering involves the correlation of fluctuations in scattering intensity arising from the Brownian motion of the scatterers obtained at different delay times [19]. This technique enables a rapid measurement of particle sizes in a solution within the nano- and micrometer ranges. This is accomplished by a measurement of the diffusion coefficient, D , which is related to the hydrodynamic diameter of the scattering particle by the Stokes–Einstein equation:

$$D = \frac{k_B T}{3\pi\eta d}, \quad (1)$$

where k_B is the Boltzmann constant (1.38054×10^{-16} ergs/deg), T is the temperature, η is the diluent viscosity of the liquid in which the particle is moving, and d is the equivalent spherical diameter. This equation assumes that the particles are moving independently of one another [26].

Zeta potential (ζ) is the electrokinetic potential arising at the interface of slipping of phases (clay–water system) during their relative movement in electric field [33]. Zeta potential reflects the influence of clay mineralogy, size, charge, and electrolyte composition. It can be considered an indicator of the electrical properties of clay–water–electrolyte systems [32]. The electrokinetic potential determines the effective charge of the particle moving in solution under the influence of electrostatic forces.

The value of zeta potential of the system determines the tolerance of the dispersion system to aggregation. Particles with zeta potential greater than the energy of thermal movement electrostatic repulsion would prevent their aggregation and, therefore, will stabilize the dispersion system preventing coagulation.

There are a few studies on evaluation of soil particles ability to coagulate, primarily due to the high degree of heterogeneity of the soil composition and the high cost of equipment. The dynamics of these properties in the process of land withdrawal from agricultural use is poorly studied, although, some data obtained on the chronosequence of soils of the Czech Republic suggest that the stability of soil colloids is gradually being restored over several decades [8]. The experiments were mainly carried out either with refined clay minerals, or with artificial (model) systems [10, 16, 27, 28, 36]. In this regard, the study of the aggregate stability of soil clay particles in water using modern analytical methods is of great interest. We propose minimizing the problem of soil heterogeneity by isolating soil fractions that are more homogeneous in terms of properties, and the most homogeneous of which, undoubtedly, is the clay fraction [1].

Our objectives were to isolate organo-mineral complexes of soddy-podzolic soils from the chronosequence of abandoned lands, to characterize their size and aggregate stability using dynamic light scattering and phase analysis light scattering, and to study the dynamics of these parameters in soils after the withdrawal from agricultural use and overgrowing by natural zonal vegetation.

OBJECT AND METHODS

The objects of the study were the humus-accumulative horizons of soddy-podzolic soils (Albic Retisols (Loamic, Cutanic, Differentic, Ochric) (WRB 2014)) of the chronosequence of abandoned sites, including the main stages of the secondary succession development: from overgrowing with grassy vegetation to zonal spruce forests of the Central Forest State Natu-

ral Biospheric Reserve. All abandoned sites are located on comparable soddy pale-podzolic sandy loamy soils underlain by moraine loam within the 300-m transect in comparable geomorphic and lithological conditions (diagnosed by the color and bulk density of soil). The abandoned sites are as follows: (1) recently (5–7 yr) abandoned site overgrown with meadow herbs; (2) abandoned site overgrown with birch and the inclusion of aspen undergrowth (*Betula pendula* + *Pópulus trémula*) (10–15 yr); (3) abandoned site overgrown with birch (*Betula pendula*) (20–25 yr); (4) birch with an admixture of aspen (*Betula pendula* + *Pópulus trémula*) (50–60 yr); (5) bilberry–herbaceous spruce forest (*Picea abies*–*Vaccinium myrtillus*–*Hylocomium splendens* + *Pleurozium schreberi*) (>100–120 yr); (6) wood sorrel–bilberry spruce forest (*Picea abies*–*Vaccinium myrtillus* + *Oxalis acetosella*) (>100–120 yr); and (7) wood sorrel–fern spruce forest (*Picea abies*–*Oxalis acetosella* + *Dryopteris dilatata*) (>100–120 yr).

The reserve is located in Tver oblast, in the upper reaches of the Mezha River to the north of Nelidovo, 54°54.17" N, 37°33.45" E.

The Central Forest State Natural Biospheric Reserve serves as a standard for southern taiga forests in the central part of European Russia. This is the area of moderately continental climate. The mean annual air temperature is + 3.6°C. The mean annual precipitation is 700 mm, and the potential evaporation is 1120 mm. The average value of the Selyaninov hydrothermal coefficient is 1.6. The soils are soddy-pale-podzolic, sandy loamy, on contrasting two-layered deposits.

Basic soil characteristics. The total C (TC) and N (TN) contents of clay particles were quantified in an aqueous suspension of clay particles using the combustion catalytic oxidation method on a TOC Analyzer (Shimadzu, Japan). Soil pH in H₂O was measured in accordance with GOST 26483-85 and in 1 N KCl, in accordance with GOST 26483.

Clay particles (organo-clay complexes). Organo-clay complexes were isolated after the preliminary removal of the free OM localized in the interaggregate space of soil. The free OM was isolated by extraction with a bromoform-ethanol mixture (BEM) (density <1.8 g cm⁻³). After the removal of the free OM, the residue of soil sample was washed off the BEM with ethanol (60 mL) and dried (60°C). Soil aggregates were broken down by sonication. A probe-type ultrasonic vibrator (LUZD-0,5K-02-00000 PS (Criamid, Russia)) was used for physical disruption. Sonication (69.7 J mL⁻¹) of the soil sample (10 g + 50 mL deionized water) was carried out for 1 min followed by centrifugation and repeated 15 times. The aqueous–clay suspension (<1 µm) was collected and dried (60°C) [6, 34]. All separation procedures were performed in triplicate.

The repeated procedure of successive fractional organo-clay separation by low-intensity sonication of the aqueous suspension makes it possible to destroy aggregates gradually, as the intensity increases; the

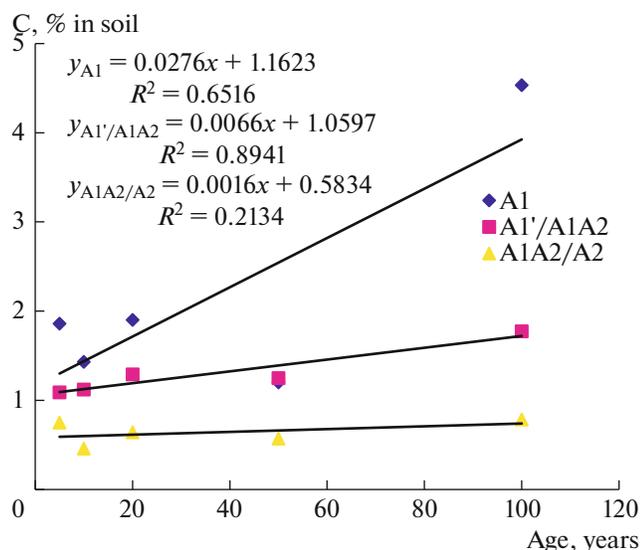


Fig. 1. The dependence of the carbon accumulation level in the studied soils on the cenosis age (interpretation of horizons is in Table 2), $P < 0.05$.

removal of each fresh portion of clay particles from the initial sample prevents superfluous ultrasonic agitation of the already separated clay particles.

Phase analysis light scattering (PALS). The electrokinetic potential (zeta potential) of the aqueous suspensions of clay particles collected after sonification was measured using a NanoBrook Omni (Brookhaven Instruments Corporation, USA) analyzer by the phase analysis light scattering technique (dilution 1 : 20). Three series of five consecutive measurements were performed for each of the organo-clay complexes studied, and the average zeta potential value was calculated.

The values of zeta potential were calculated using a computer with software based on the Helmholtz–Smoluchowski equation:

$$\mu_E = \frac{2\varepsilon\zeta}{3\eta}, \quad (2)$$

where ζ is the electrokinetic potential, μ_E is the electrophoretic mobility, ε is the dielectric constant, and η is the dynamic viscosity of the dispersion medium.

Dynamic light scattering (DLS). The average diameter of clay particles in samples of aqueous–clay suspensions (dilution 1 : 20) were analyzed using a NanoBrook Omni (Brookhaven Instruments Corporation, USA) analyzer by the dynamic light scattering technique. Three series of five consecutive measurements were performed for each of the organo-clay complexes studied, and, further, the average diameter value was calculated.

Statistical analysis. 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 $p < 0.05$.

RESULTS AND DISCUSSION

Basic Chemical Properties

In general, the studied soils can be classified as very acidic in terms of potential acidity (pH_{KCl}). An analysis of the distribution of potential acidity in the soil profiles at different stages of succession shows that it is characterized by differentiation along the profile typical of the studied soil subtype; with an increase in the age of the abandoned site, the differentiation of the acidity distribution along the soil profile becomes more pronounced (Table 1). The absence of a noticeable change in the pH_{KCl} value (horizon A1) with increasing age of the abandoned site was revealed. It should be noted that spruce forests of different species composition are characterized by the highest pH values with a maximum in the soil of the bilberry–herbaceous spruce forest.

When analyzing the total carbon (C_{total}), all genetic horizons were combined into three groups: upper (A1 horizon), intermediate ((A1' or A1A2 horizons; A1'/A1A2), and lower (A1A2 or A2 horizons; A1A2/A2) horizons.

Figure 1 demonstrates a tendency for an increase in the carbon accumulation in the soil with increasing age of cenosis; note that the angle of inclination for the upper humus-accumulative horizon is greater than that for the underlying horizons (coefficient of determination (R^2) 0.65 vs. 0.89, respectively); for the lower horizon, this effect is statistically insignificant ($R^2 = 0.21$).

The average values of C_{total} (A1 horizon) for spruce forests of different species composition are more than 2.9 times higher compared to those for the soils of the chronosequence of the abandoned sites. The obtained analytical data are consistent with literature data on the increase in the total carbon during overgrowing of texture-differentiated soils, including soddy-podzolic soils, withdrawn from agricultural use [2–5, 8, 9, 13, 22].

Characteristics of Organo-Clay Complexes

The C concentration value (C_{Clay}) in the chronosequence decreased with increasing content of clay particles and the age of the abandoned sites (Table 2). This is consistent with previous data [1, 4, 8, 9].

In the upper horizons, there is a tendency for an increase in the clay content with increasing age of the abandoned site. This is due to the lack of regular plowing of soils after their withdrawal from the active agricultural use. This leads to the compaction of the upper horizons, and their composition includes the mass of the underlying horizon with a higher clay content. This is reflected in the increase in the clay content and density of the upper horizons [8, 9].

Table 1. Some chemical and physical characteristics of the studied soils

Cenosis	Age (years)	Horizon	Depth (cm)	pH		C _{total}	N _{total}	C/N
				H ₂ O	KCl			
Abandoned site	5–7	A1	3–12	4.80	3.73	1.86	0.16	11.6
		A1A2	12–29	4.91	3.80	1.09	0.10	10.9
		A2	29–35	5.00	3.92	0.75	0.06	12.5
	10–15	A1	3–15	4.50	3.71	1.43	0.10	14.3
		A1A2	15–28	4.62	3.78	1.12	0.09	12.4
		A2	28–43	4.71	3.86	0.46	0.04	11.5
	20–25	A1	4–20	4.79	3.66	1.20	0.10	12.0
		A1A2	20–26	4.91	3.80	1.25	0.11	11.4
		A2	26–40	4.83	3.92	0.57	0.05	11.4
	50–60	A1	3–16	4.70	3.70	1.90	0.14	13.6
		A1A2	16–30	4.79	3.81	1.29	0.10	12.9
		A2	30–44	4.90	3.95	0.64	0.06	10.7
SFBH	100–120	A1	4–10	5.40	4.07	3.57	0.20	17.8
		A1'	10–16	4.80	3.64	1.74	0.12	14.5
		A1A2	16–26	5.20	4.11	1.13	0.08	14.1
SFWSB		A1	4–10	5.20	3.95	4.76	0.29	16.4
		A1A2	10–20	4.80	3.75	0.85	0.07	12.1
		A2	20–30	4.90	3.95	0.31	0.03	10.3
SFWSW		A1	3–7	4.93	3.71	5.81	0.36	16.1
		A1'	7–17	5.00	3.63	2.73	0.38	7.2
		A1A2	17–32	5.17	3.93	0.92	0.07	13.1

Hereinafter, SFBH is the bilberry–herbaceous spruce forest; SFWSB is the wood sorrel bilberry–spruce forest; and SFWSW is the wood sorrel–fern spruce forest.

Zeta Potential

Zeta potential values were negative in the investigated pH range and independent of pH ($R^2 < 0.1$). Electrokinetic potential of colloidal systems in humus-accumulative horizons of studied chronosequence varied from -25.73 to -13.70 mV and had a tendency to increase with increasing age of the abandoned site (Table 2). Soils of spruce forests of different species composition were characterized by higher values of zeta potential in comparison with the soils of the abandoned sites. The only exception was observed for the wood sorrel–fern spruce forest. The maximum zeta potential value was observed for the wood sorrel–bilberry spruce forest (-10.15 mV).

This indicates that in the upper horizons of soils withdrawn from agricultural use and overgrown with natural forest vegetation, a renewal of the electrokinetic features of the colloidal system typical for this

soil type can be observed. This renewal is expressed in an increase in their peptizing properties, which is indicated by an increase in zeta potential values with increasing age of forest cenoses.

The obtained analytical data are in accordance with published data [8]. Previously, it was shown that afforestation of former agricultural lands leads to less negative values of zeta potential of organo-clay complexes in comparison with their arable analogues [8].

Thus, the value of zeta potential increases as the age of afforestation increases and reaches its maximum in the soils of spruce forests of different species composition. The lowest values of zeta potential observed for the abandoned site of the youngest age, in our opinion, are due to the prolonged aftereffect of the positive influence of their agricultural past. Previously, it was revealed that intensive tillage reduces the colloidal system stability, which can be recovered during further afforestation [8].

Table 2. Characteristics of the clay particles in soils of the studied chronosequence

Cenosis, age	Depth (cm)	pH	<1 μm		Diameter (D)	Polydispersity (Poly)	Zeta potential (ζ)
			content (%)	C_{Clay} (% in fraction)	nm		mV
Abandoned site 5–7 yr	3–12	5.71	2.58 ± 0.26	12.36 ± 0.12	599.29 ± 15.00	0.30	-23.05 ± 2.09
	12–29	5.49	3.41 ± 0.18	12.54 ± 0.10	591.36 ± 12.00	0.31	-20.02 ± 0.96
	29–35	5.34	3.18 ± 0.16	10.14 ± 0.08	292.45 ± 11.00	0.17	-19.14 ± 1.30
Abandoned site 10–15 yr	3–15	5.72	3.78 ± 0.15	10.09 ± 0.42	470.73 ± 3.00	0.21	-25.73 ± 0.82
	15–28	5.68	4.32 ± 0.31	12.15 ± 0.10	547.66 ± 6.00	0.32	-20.98 ± 0.72
	28–43	5.51	2.90 ± 0.16	9.56 ± 0.14	457.38 ± 9.00	0.26	-27.58 ± 0.52
Abandoned site 20–25 yr	4–20	5.68	7.52 ± 0.22	6.62 ± 0.08	430.05 ± 17.00	0.22	-13.70 ± 1.61
	20–26	5.49	8.23 ± 0.19	8.60 ± 0.11	492.08 ± 6.00	0.29	-19.36 ± 1.53
	26–40	5.58	6.35 ± 0.11	6.11 ± 0.12	383.71 ± 8.00	0.25	-22.92 ± 0.70
Abandoned site 50–60 yr	3–16	5.65	3.69 ± 0.27	10.88 ± 0.07	474.97 ± 4.00	0.26	-18.62 ± 0.77
	16–30	5.69	5.33 ± 0.11	10.91 ± 0.46	602.68 ± 14.00	0.30	-14.21 ± 2.10
	30–44	5.67	5.27 ± 0.14	7.31 ± 0.07	367.82 ± 5.00	0.22	-14.00 ± 5.05
Forest 100–120 yr, SFBH	4–10	5.54	7.99 ± 0.11	10.88 ± 0.24	452.75 ± 5.00	0.23	-12.51 ± 1.43
	10–16	5.84	10.00 ± 0.32	10.71 ± 0.34	283.36 ± 12.00	0.04	-17.67 ± 1.72
	16–26	5.58	5.54 ± 0.29	12.21 ± 0.41	401.96 ± 2.00	0.21	-21.10 ± 1.18
Forest 100–120 yr, SFWSB	4–10	5.64	7.39 ± 0.17	10.11 ± 0.11	411.39 ± 6.00	0.19	-10.15 ± 2.94
	10–20	5.76	5.71 ± 0.22	7.28 ± 0.24	358.07 ± 11.00	0.14	-16.61 ± 1.24
	20–30	5.74	1.92 ± 0.13	7.88 ± 0.39	313.93 ± 7.00	0.28	-13.38 ± 0.87
Forest 100–120 yr, SFWSW	3–7	5.68	5.97 ± 0.23	17.69 ± 0.02	527.91 ± 11.00	0.19	-23.20 ± 0.79
	7–17	6.05	6.12 ± 0.19	13.91 ± 0.04	509.75 ± 12.00	0.25	-20.25 ± 0.66
	17–32	5.94	5.49 ± 0.14	9.34 ± 0.12	420.61 ± 1.00	0.22	-15.96 ± 1.31

The Average Diameter of Organo-Clay Complexes

The average diameter of organo-clay complexes in aqueous suspensions in the two upper horizons of the studied soils varied from 358 to 603 nm. In the lower horizons (A1A2/A2), the average diameter was smaller: from 292 to 457 nm. The only exception was noted for a bilberry–herbaceous spruce forest, where the minimum average diameter (283 nm) was determined in the A1' horizon. The value of the average diameter is largely determined by the carbon concentration in clay particles. This is in accordance with previously obtained data indicating the formation of larger clay ultramicroaggregates with an increase in the carbon concentration [8].

Afforestation of the former agricultural soils favors the reduction of the average diameter of the organo-clay complexes in the upper horizons with increasing age of the abandoned sites forming the following sequence: wood sorrel–bilberry spruce forest (411.39 nm) < abandoned site 50–60 yr (474.97 nm) < bilberry-herbaceous spruce forest (452.75 nm) < abandoned site 10–15 yr (470.73 nm) < abandoned site 5–7 yr (599.29 nm).

The abandoned site of 20–25 yr in age and the wood sorrel–fern spruce forest do not fit into this sequence, which may be due to other soil properties not investigated in this experiment, for example, to significant differences in their texture.

A polynomial relationship between the average diameter of clay particles (D) and the clay content was revealed. The determination coefficient (R^2) in the upper horizons (A1 ($R^2 = 0.65$) > A1'/A1A2 ($R^2 = 0.57$)) was significantly higher than that for the underlying horizons (A1A2/A2 ($R^2 = 0.15$)) (Fig. 2). Thus, for the upper horizons of the studied chronosequence of the abandoned sites, the average diameter of the clay particles decreases with an increase in the clay content increases.

Taken into consideration the importance of the organo-clay complexes in soil aggregation, the multiple linear regression statistical models to predict the average diameter of clay particles were calculated. The following parameters were used in the model: clay content, carbon concentration in clay (C_{Clay}), zeta potential (ζ), polydispersity (Poly), and pH of the aqueous suspension of the organo-clay complexes (Table 3).

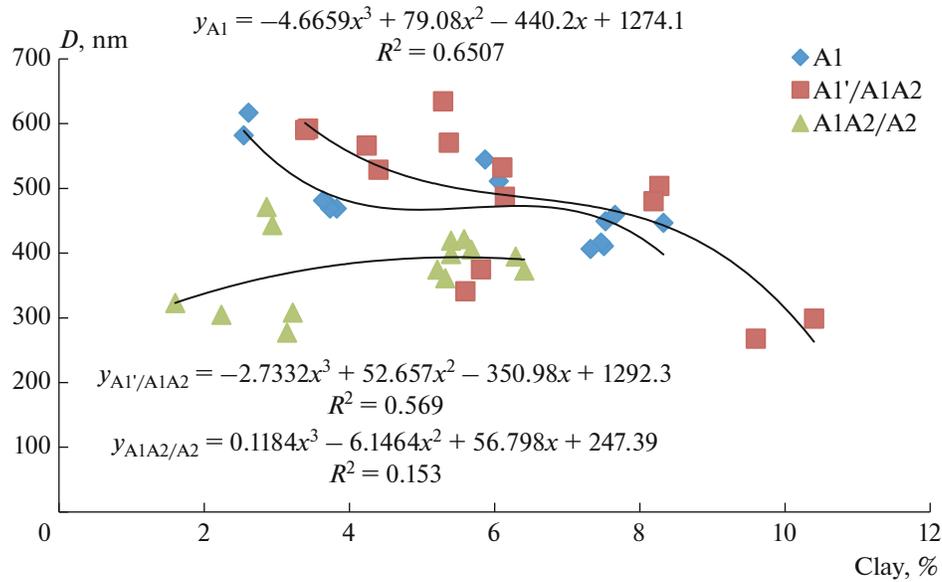


Fig. 2. The dependence of the clay particle size (D) on the clay content ($P < 0.05$).

At the initial stage, for models describing the average diameter of clay particles $< 1 \mu\text{m}$ in size, all parameters for the entire data set were used. It turned out that, despite the satisfactory result ($R_{adj}^2 = 0.72$), only three of the five parameters were significant.

Assuming that the average diameter of the organo-clay complexes in the upper humus-accumulative horizon should be different from that of the underlying horizons, we examined them separately. It turned out

that such a separation of the entire data set makes sense: despite a lower determination coefficient ($R_{adj}^2 = 0.63$, $n = 14$, $\text{RMSE} = 38.34 \text{ nm}$ ($P < 0.05$)) with a smaller error, all model parameters (clay content and carbon concentration in clay) were significant (Table 4, Fig. 3).

This is consistent with the noted trend of increasing clay content due to compaction of the upper horizons

Table 3. The dependence of the clay particle diameter (y) on the clay content (Clay), carbon concentration in clay (C_{Clay}), zeta potential (ζ), polydispersity (Poly), and pH of the aqueous suspensions of organo-clay complexes; coefficients of the multiple linear regression statistical model $y = A_0 + A_1x_1 + A_2x_2 + A_3x_3 + A_4x_4 + A_5x_5$, $P < 0.05$. The entire data set (all studied horizons, $n = 42$) was used for calculations

x	A	Value	Standard Error	t -value	Prob $> t $
	A_0	-379.58	306.33	-1.24	0.22
x_1 (Clay)	A_1	11.87	4.67	2.54	0.01
x_2 (C_{Clay})	A_2	15.99	3.17	5.05	0.00
x_3 (pH)	A_3	50.66	52.61	0.96	0.34
x_4 (Poly)	A_4	1182.10	143.79	8.22	0.00
x_5 (ζ)	A_5	-1.99	1.83	-1.08	0.29

Table 4. The dependence of the clay particle diameter (y) in samples ($n = 14$) of the upper humus-accumulative horizon on the clay content (Clay) and the carbon concentration in clay particles (C_{Clay}); coefficients of the multiple linear regression statistical model $y = A_0 + A_1x_1 + A_2x_2$, $P < 0.05$

x	A	Value	Standard error	t -value	Prob $> t $
	A_0	480.58	53.67	8.95	2.20E-06
x_2 (Clay)	A_1	-17.78	5.17	-3.44	0.01
x_3 (C_{clay})	A_2	8.84	3.40	2.60	0.02

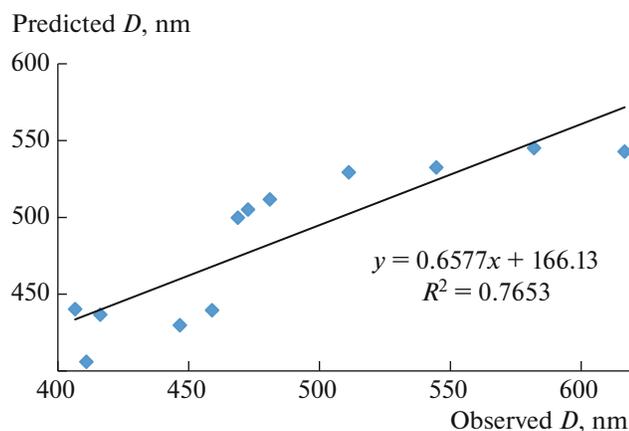


Fig. 3. The prediction of D using multiple linear regression statistical model ($D = f(\text{Clay}, C_{\text{clay}})$, $n = 14$, $R_{adj}^2 = 0.77$, $P < 0.05$).

and the partial inclusion of the soil mass from the underlying horizons with smaller clay particles.

CONCLUSIONS

The organic-mineral complexes of soddy-pale-podzolic sandy loam soils of the studied chronosequence of the abandoned sites were isolated and investigated using dynamic light scattering (DLS) and phase analysis light scattering (PALS). A tendency toward a decrease in the carbon concentration in the organo-mineral complexes with an increase in the clay content was observed.

The postagrogenic soddy-pale-podzolic soils of the studied chronosequence demonstrate a similar stability degree of the colloidal system in a state of peptization. This is confirmed by an increase in the zeta potential and a decrease in the effective diameter with increasing age of the abandoned sites from 5–7 years (as close as possible to arable soils) to 50–60 years.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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