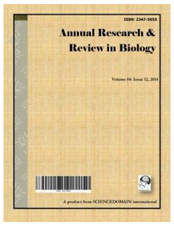

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Phytolith Transport in Texturally Differentiated Soils

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Author's contribution

The sole author designed, analyzed and interpreted and prepared the manuscript.

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ABSTRACT

Aims: Over the last decade phytolith analysis has been increasingly used in paleoenvironmental, archeological and paleopedological research. It resulted in standardization of phytolith sample collection, laboratory methods of phytolith extraction, counting and developing reference collections. In spite of all these advances some issues of phytolith translocation in soil profiles have not been comprehensively studied. This study describes an attempt to assess phytolith translocation along the profile of texturally differentiated soils.
Study Design: Comparative analysis of the two soil profiles.
Place and Duration of Study: Institute of Soil Science and Agrochemistry SB RAS, Core Facility "Cenozoic geochronology" (both in Novosibirsk, Russia), and the Center for Applied Isotope Studies, University of Georgia (Athens, USA), between June 2015 and May 2016.
Methodology: Catenary approach, accelerator mass spectrometry dating and phytolith analysis were used to register phytolith translocation down the profile of polygenetic Umbric-Cutanic Albeluvisols.
Results: The obtained results allowed to establish, depending on soil catenary position and the extent of profile texture differentiation, the following: 1) some facts of phytoliths' translocation, 2) varying rate of ^{14}C-phytoliths' rejuvenation, and 3) changes in soil phytolith profiles due to eluvial-illuvial processes. The most pronounced changes in phytolith profiles were found in soils at eluvial position of the catena, while smaller changes were observed in the soil at the transit positions.
Conclusion: The possible translocation of phytolith down soil profiles should be taken into consideration while performing phytolithic analysis of forest soils.

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Keywords: Phytoliths; phytolith translocation; phytolith AMS dating; Albeluvisols; second humus horizon; forest soils.

1. INTRODUCTION

Phytoliths are amorphous silicon mineral particles formed in plant cells and preserved in soil and sediments after dead phytomass has decayed. Specific morphology of phytoliths provides the possibility to identify plant taxa at the family level, sometimes even at the genus level, hence ensuring the use of phytolith analysis for paleoenvironment reconstruction [1-3].

During phytomass decomposition phytoliths get into soil and become part of its mineral component, determined by morphological studies as bioliths or biogenic opal. Phytolith profile formation takes place simultaneously with soil formation. Phytolith profile, together with other partial soil profiles (humus, carbonate, textural ones and others) records in its characteristics major environmental changes, soil forming factors or anthropogenic influence [4]. Changes in soil-forming conditions and phytocoenosis properties result in gradual shifts in a soil phytolith profile, but the general stratigraphic regularity remains recorded in its characteristics: the lower samples are older than those located closer to the surface. There has been some limited research focusing on the temporary dynamics of phytolith profiles under indigenous conditions. Due to their silicon nature most phytoliths are naturally resistant to weathering [5-7], but their soil profile distribution may change with time and, if so, it should be taken into consideration while reconstructing paleoenvironments.

Laboratory and field studies by O. Fishkis with coauthors [8,9] confirmed the possibility of phytoliths' translocation in sandy sediments and loamy soils, such as Haplic Cambisol and Stagnic Luvisol. Soil bioturbation is widely believed to cause phytoliths redeposition within soil profiles [10-13]. Researchers often acknowledge limitations of phytolith analysis in light-textured soils, as well as question its broad application for loamy and clay soils [4], arguing that using 5 µm phytoliths in silt fraction (0.001-0.05 mm) limits their ability to move within soil profile.

The main factor facilitating phytolith redistribution throughout soil profile is leaching water regime, which also determines specific nature of soils in the humid zone. Therefore one should assess potential relocation of phytoliths along soil profile while studying phytolith profiles in forest soils.

Umbric-Cutanic Albeluvisols are very common soils in the south of the forest zone (south taiga subzone) both in the European and Asian (Siberia) parts of Russia. These soils often have polygenetic structure of organic matter profiles due to preserving some relic signs resultant from the dark humus stage of soil formation during Middle Holocene (high humus content, different humus quality in modern and second humus horizons, occurrence of molecasts, etc.) [14-19]. In Middle Holocene these soils are believed to develop under warmer and more humid climate with humus accumulation as the main profile-forming process, but later subboreal climate change with decreasing temperatures shifted the border of taiga coniferous forests southward, thus favouring soil texture differentiation (podzolization, loessivage, etc.).

In these soils texture differentiation and preservation of properties resulting from the dark humus formation stage (decreased thickness of the secondary humus horizon, its colouration and spread, the intrapedic structures and other properties) are determined by their location in the relief. The polygenetic Umbric-Cutanic Albeluvisols, located along a catena, are heterogenic and relatively synchronic in origin, implying their coevolution. Such catenary chain of soils represents a very good model to study phytolith translocation due to shifts in soil formation types and the transformation of phytolith profiles in texturally differentiated soils.

The aim of the study was to explore variations in the composition of phytolith assemblages with depth in the profiles of the two genetically-related Umbric-Cutanic Albeluvisols with second humus horizons in an attempt to reveal phytolith translocation due to soil catenary position and soil texture differentiation and to assess the phytolith potential to be used for paleoreconstructions in case of phytolith translocation along soil profile.

2. MATERIALS AND METHODS

2.1 Study Area and Soil Characteristics

The studied soils were located at the eluvial and transit ecosystems of a catena ranging from the watershed down to the Iksa riverbed (56º54’50” Nl, 83º02’53” EL, Bakchar district, Tomsk region, Russia, Fig. 1). The area lies in the southern taiga zone with 550 mm of annual precipitation, average yearly temperature of -1.1°C, the annual sum of positive temperatures of 1650°C·day and 102 days a year of the frost-free period.

According to the WRB classification [20], the soils were classified as Umbric-Cutanic Albeluvisols. However, this classification does not indicate soil profile polygenetic structure that is reflected by the depth and thickness of the illuvial horizon and by the presence of the residual dark-humus horizon, so called second humus horizon, within the eluvial horizon and beneath it. The national classification of Russian soils [21] provides these soil peculiarities at the subtype titles. Thus the soil at the eluvial position was classified as soddy non-deeply podzolic soil with the second humus horizon (hereafter referred to as *ucA-1* soil), whereas the soil in the transit position was classified as soddy shallow podzolic soil with the second humus horizon (hereafter referred to as *ucA-2* soil).

The studied soils differed in thickness of their eluvial horizons and the extent of the second humus horizon preservation (its thickness, colour, texture and granulometric composition, Fig. 1, Table 1).

If the second humus horizon was explicit as a continuous dark-humus horizon below the eluvial one, it was denoted as horizon A’ (*ucA-2*). Incase the second humus horizon was represented by separate dark-grey patches within the light-grey eluvial horizon, it was denoted as horizon Eh (*ucA-1*).

The soil pits revealed eluvial and illuvial parts of soil profiles (below the second humus horizon), that displayed no explicit signs of windfall phenomena.

To identify soil genetic type some diagnostic chemical and physical soil properties were determined in laboratory in soil samples collected in 2016 from every 5-10 cm by continuous coring within soil genetic horizons, while for phytolith analysis samples were taken from the lower 1-2 cm of each 5 cm soil layer [4].

Granulometric composition of soil samples was determined by pipette method after preliminary treatment of soil with 4% $Na_4P_2O_7$ solution [22]. Soil organic matter carbon was determined by dichromate redox titration method [23]. Exchangeable cations content was measured according to [24]. Soil reaction and exchangeable acidity were measured according to [25].

The studied soils had heavy or light loamy particle size composition with sharply differentiated textural profiles with a slight difference in silt differentiation coefficients (Table 2).

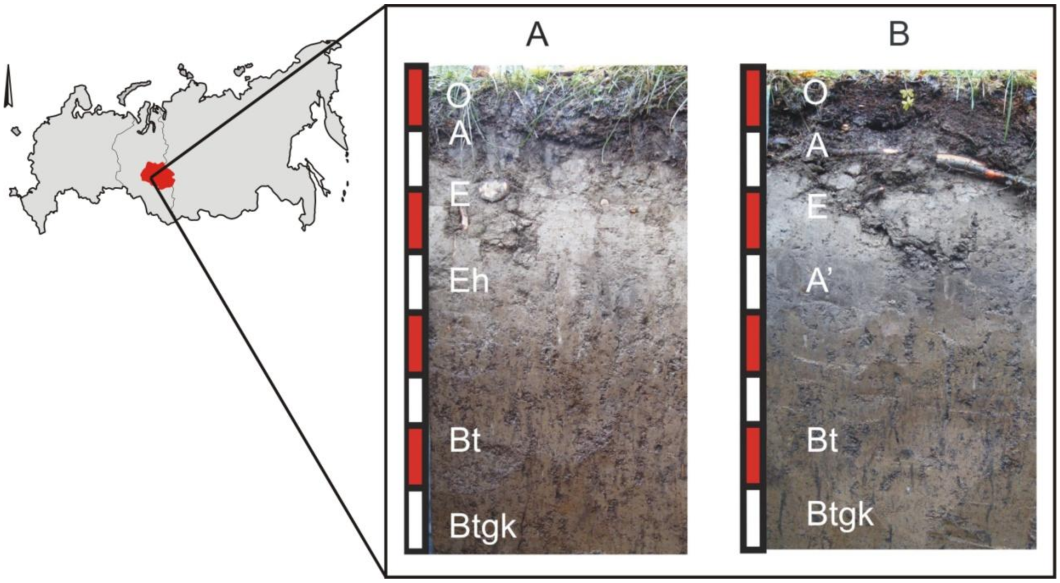

Fig. 1. Location site and morphology of the southern taiga Umbric-Cutanic Albeluvisols: A – *ucA-1*, B – *ucA-2*

Table 1. Some chemical properties of the soils

Horizon	n[a]	Depth, cm	TOC[c], %	pH		$CaCO_3$, %	Hr[d]	Exchangeable cations				V[f], %
				H_2O	KCl			Ca^{2+}	Mg^{2+}	Na^+	K^+	
							cmol kg^{-1}					
1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**
							Umbric-Cutanic Albeluvisol (ucA-1)					
A	1	7–13	1.9	4.1	3.1	0	22.4	3.1	0.9	0.5	1.3	21
E	2	13–30	0.5	4.4±0.3	3.2±0.1	0	7.4±0.7	3.1±0.0	0.9±0.0	0.4	0.8±0.5	43
Eh	1	30–40	0.5	5.1	3.4	0	6.3	10.5	3.9	0.5	0.6	71
Bt	4	40–70	0.3±0.1[b]	6.8±1.1	5.2±0.9	0.2±0.2	3.1±1.7	20.6±1.3	10±1.0	0.6	0.9±0.1	91
Btk	1	70–80	0.2	8.2	7.3	4.6	0.3	57.9	8.4	0.6	0.9	100
							Umbric-Cutanic Albeluvisol (ucA-2)					
A	1	6–13	0.6	4.3	3.2	0	19.8	7.4	1.8	0.6	0.5	39
E	2	13–24	0.5±0.1	5.0±0.1	3.5±0.1	0	8.1±1.4	6.1	0.9	0.4	0.3	58
A’	2	24–42	0.6±0.1	5.9±0.2	4.5±0.2	0	4.9±0.7	14.9±3.2	3.5±0.6	0.5±0.1	0.4±0.1	79
Bt	4	42–70	0.3±0.1	6.5±0.4	5.0±0.4	2.7±1.3	3.2±1.1	24.9±6.0	4.7±0.6	0.6	0.7±0.2	94
Btgk	1	70–80	0.2	7.9	7.1	5.8	0.4	35.9	4.8	0.6	0.8	99

[a] n = The number of soil samples collected at different depths from each horizon; [b] Physico-chemical data are reported as the mean ± standard deviation when n ≥ 2); [c] TOC – Total organic carbon; [d] Hr – Hydrolytic acidity; [f] V – Degree of saturation with bases

Table 2. Soil granulometric composition (particle size, mm) and soil texture differentiation in respect to silt [19]

Horizon	n	Depth, cm	Particle content (%) and size (mm)					Total, %	
			Sand	Silt			Clay	Physical clay, %	Physical sand, %
			0.25-0.05	0.05-0.01	0.01-0.005	0.005-0.001	<0.001	>0.01	<0.01
					Umbric-Cutanic Albeluvisol (*ucA-1*)				
A	1	7–13	10.6	44.2	16.2	14.8	14.1	54.9	45.1
E	2	13–30	6.3±0.9	28.4±2.5	15.9±0.5	10.7±0.7	11.6±1.7	31.2±1.4	44.8±0.9
Eh	1	30–40	3.0	41.0	15.0	15.4	25.6	44.0	56.0
Bt	4	40–70	2.7±0.8	46.5±2.3	7.6±0.6	16.2±1.4	50.5±2.0	55.5±2.0	68.8±1.6
Btk	1	70–80	4.2	33.7	06.0	09.5	46.7	37.8	62.2
S[a](El/Bt)=5.1									

Horizon	**n**	**Depth, cm**	**Particle content (%) and size (mm)**					**Total, %**	
			Sand	**Silt**			**Clay**	**Physical clay, %**	**Physical sand, %**
			0.25-0.05	**0.05-0.01**	**0.01-0.005**	**0.005-0.001**	**<0.001**	**>0.01**	**<0.01**
			Umbric-Cutanic Albeluvisol (*ucA-2*)						
A	1	6–13	7.3	40.3	16.7	17.6	18.0	47.7	52.3
E	2	13–24	3.8±0.2	47.1±0.6	17.2±1.2	18.3±0.8	13.2	51.0±0.4	49.0±0.4
A’	2	24–42	1.7±0.2	34.7±4.4	14.2±0.9	16.8±1.9	23.2±4.1	36.3±4.3	55.9±3.5
Bt	4	42–70	0.9±0.4	30.5±0.3	9.0±2.0	12.8±2.1	50.7±4.2	32.0±0.1	71.5±0.1
Btgk	1	70–80	2.4	32.2	7.1	9.1	49.2	34.6	65.4
S(El/Bt)=4.5									

[a]S – The extent of profile differentiation according to silt content

Table 3. Major phytolith types in the profiles of the Umbric-Cutanic Albeluvisols (%)

Horizon, cm	**Sample**	**Dicotyledonous**	**Ecological groups of grasses**				**Coniferous**	**Mosses**	**Corroded**	**Total (pcs.)**
			Forest	**Meadow**	**Forest**	**Semi-mire (Phragmites)**				
			Umbric-Cutanic Albeluvisol (*ucA-1*)							
A	306	85	11	3	0	0	0	0	0	100
7-13	305	58	11	27	0	0	3	0	8	886
	304	51	10	36	0	0	2	0	12	1155
E	303	49	8	41	0	0	1	0	24	1566
13-30	302	38	12	47	0	0	3	0	25	1978
	301	37	8	48	0	0	5	0	25	1714
Eh	300	52	24	21	<1	0	3	0	41	143
30-40	299	38	11	45	<1	0	5	0	43	380
			Umbric-Cutanic Albeluvisol (*ucA-2*)							
A 6-13	263	50	13	23	4	4	3	4	n.d.[1]	110
E 13-24	262	16	18	47	7	2	8	7	32	669
	261	41	10	43	1	2	2	1	5	1244
A’	260	33	13	49	1	4	2	1	51	1163
24-42	259	29	14	54	0	12	3	0	57	824
	258	37	13	46	0	0	2	0	18	1148
	257	50	22	24	0	0	2	0	n.d.	156

[1] n.d. stands for not determined

2.2 Phytolith Analysis

2.2.1 Phytolith sampling and extraction

For phytolith analyses soil samples were taken from the lower 1-2 cm of each 5 cm soil layer [4] within the humus-accumulating and eluvial part of the soil profile.

Soil samples for phytolith analysis were dried in laboratory at 60ºC and subjected to the standard extraction procedure [1]. Briefly, after treating soil with hot 30% hydrogen peroxide (H_2O_2) solution and then 10% hydrogen chloride solution, sand fraction was removed by wet sieving of *ca.* 50 g of sample and silt fraction was removed by flotation. Then heavy liquids (cadmium and potassium iodide solutions with 2.3. g/ml density) were added to the dried sample, and the obtained mixture was centrifuged at 1,500 rpm for 20 min. The phytolith from the supernatant were collected into a tube, washed several times with distilled water and studied by light microscopy at 300-400 magnification. Phytoliths were counted on the cover glass area of 24 mm by 24 mm.

2.2.2 Phytolith identification

Phytoliths were identified and classified into the common categories listed in Tables 2-3, mainly according to the International Code for Phytolith Nomenclature 1.0 [26]. The results of phytolith analysis were interpreted following the ecological classification of phytolith groups by Golyeva A.A. [4]. The phytolith assemblages consisted of universal dicotyledonous phytolith types, i.e. the types that do not allow indentifying phytolith origin to taxa below the class; ecological groups of phytolith assemblages derived from different phytocoenoses (taiga, meadow, steppe, dry steppe), as well as phytoliths produced by specific families (*Cyperaceae sp*., *Pinaceae sp.*) or even species (so called “signal forms” according to Golyeva [4], e.g. *Phragmites spp.*).

2.2.3 Radiocarbon dating

To assess phytolith translocation due to forest soil formation (textural differentiation, podzolic horizon development) we used radiocarbon dating of phytoliths (phytolith occluded carbon, PhyOC) by accelerator mass spectrometry (AMS dating) performed by the SB RAS Core Facility “Cenozoic Geochronolgy”, (Novosibirsk, Russia); and by the Center for Applied Isotope Studies, University of Georgia (Athens, USA).

Soil samples for radiocarbon analysis were collected at the upper and lower boundaries of E, Eh and A’ horizons 1 cm thick from each soil profile (Table 3). Sites with explicit signs of bioturbation were excluded from soil sampling. As some bioturbation may not be noticed morphologically, we used 5 cm increment to reduce the possibility of sampling the bioturbated soil.

Phytoliths were prepared for AMS dating according to the slightly modified method of Zuo et al. [27] by obtaining graphite that was analyzed for ^{14}C using CAIS 0.5 MeV accelerator [28]. Radiocarbon dates were calibrated with the help of OxCal v4.3 software [29] and calibration curve IntCal 13.

2.2.4 Age-depth model

To simulate the change of PhyOC radiocarbon age with soil depth we used the age-depth model using OxCal software [30], commonly employed to simulate peat growth. This model was used as the basis for our simulation as it allows constructing radiocarbon profile for undisturbed soil or sediment profile with accumulating input of organic carbon or organic matter containing materials.

3. RESULTS AND DISCUSSION

3.1 Soil Phytolith Profiles

Phytolith assemblage of *ucA-1* soil horizon A consisted of phytoliths of dicotyledonous plants with a diversity of gramineous phytoliths derived from meadow and forest grasses, with the meadow ones dominating; phytoliths from coniferous plants and mosses were also observed (Table 3). The phytolith assemblage of horizon El was rather similar to the one in horizon A, but had the increased meadow-specific phytolith content and increased disparity (4-6 fold) between meadow- and forest-specific gramineous phytoliths. Steppe-specific gramineous phytoliths (about 1%) were found in the lower part of eluvial horizon.

Thus the dark humus accumulation stage in soil formation seemed mostly resultant from the grassland stage, which is recorded in meadow-specific gramineous phytolith predomination over the forest-specific ones.

The phytolith assemblage of the *ucA-2* soil was found to be rather similar to the one of the *ucA-1*:

the presence of phytoliths derived from meadow and forest grasses with the meadow-specific ones dominating in the humus-accumulating and eluvial part of the soil profile (3.5-3.8 times, Table 3).

However, small amount of *Phragmites sp.* phytoliths, found in E and A' horizons as well as in the upper part of the A'/Bt horizon, resulted from periodically more humid conditions of the soil profile functioning. Phytoliths formed by coniferous trees were found throughout the humus-eluvial part of the profile, and the relative contribution of such phytoliths into the phytolith assemblage increased towards the topsoil.

Thus the presence of Phragmites-type bulliform cell and coniferous phytoliths confirmed the shift in phytocoenoses from the intrazonal mire-meadow or bogged forest ones to the zonal automorphous coniferous forest.

3.2 Phytholith Transport and PhyOC AMS-dating

The AMS-dating of the samples taken from the lower part of the second humus profiles resulted in relatively close age estimates of 5220-5321 B.C. (the median of 5270 B.C.) and 5710-5844 B.C. (the median of 5777 B.C.) (Table 4). According to the date-age model the carbon age in the second humus horizons of the studied soils at the depth of 30 cm is similar (*ca.* 5500 B.C., Fig. 2).

Samples from eluvial horizons of both studied soils gave PhyOC radiocarbon dates as 2151-1903 B.C. (with a median of 2026 B.C.) and 4042-3936 B.C. (with a median of 3989 B.C.), respectively. The difference between the dates reaches almost 2000 yrs.

The profile change of phytoliths carbon age in both soils confirms the general process of carbon rejuvenating. However, the rate of rejuvenating does not seem to be the same in different ecosystems of the catena. The comparison of radiocarbon and simulated age estimates in the eluvial ecosystem showed that the rate of phytolith carbon rejuvenation in the eluvial horizon at the depth of 10 cm was two times higher than in the transit ecosystem.

We established that the degradation of the second humus horizon was followed by the rejuvenation of phytolith carbon. The process of phytolith assemblage rejuvenation occurs by means of phytolith translocation, rather than the input of the younger soil carbon into phytoliths during their dissolution in the aggressive soil conditions. The increased percentage of the corroded phytoliths with soil depth (Table 3) corroborates the idea. Besides that, the increased relative concentration of phytoliths in E horizon of the soil at eluvial Catenary position at the depth of 25-30 cm as compared with the E and A' horizons at the same depths proves their translocation down the soil profile.

Some researchers, trying to explain the overrated PhyOC age of the phytomass from herbaria collections [31-33], suggested contamination of phytoliths with soil organic carbon. But if such process was widespread, and if the share of soil organic carbon was relatively quite large as compared to the carbon contained in phytoliths, then the carbon PhyOC age at different depth of a soil profile would have been close. But this contradicts our findings.

Weak morphological signs of the second humus horizon in the eluvial ecosystem, satisfactorily preserved signs in the transit ecosystem, as well as the similarity in phytolith profiles of both soils prove the potential of phytolith assemblages to benefit paleoecological studies in environments favouring soil texture differentiation and erasing the signs of the relic dark-humus soil formation.

Table 4. The results of AMS-dating of phytolith carbon from the eluvial and second humus horizons

Sample number	Horizon	Depth, cm	C, %	δ13CPDB, ‰	^{14}C, years BC	Calibrated age (years BC), 2σ	
							Median
			Umbric-Cutanic Albeluvisol (*ucA-1)*				
NSK-G1	E	9-10	1.14	-32.1	3663±53	2151-1903	2026
NSK-G3	Eh	29-30	1.19	-30.0	6301±26	5321-5220	5270
			Umbric-Cutanic Albeluvisol (*ucA-2)*				
NSK-G4	E	19-20	0.89	-28.6	5154±32	4042-3936	3989
NSK-G2	A'	34-35	1.43	-30.4	6883±32	5844-5710	5777

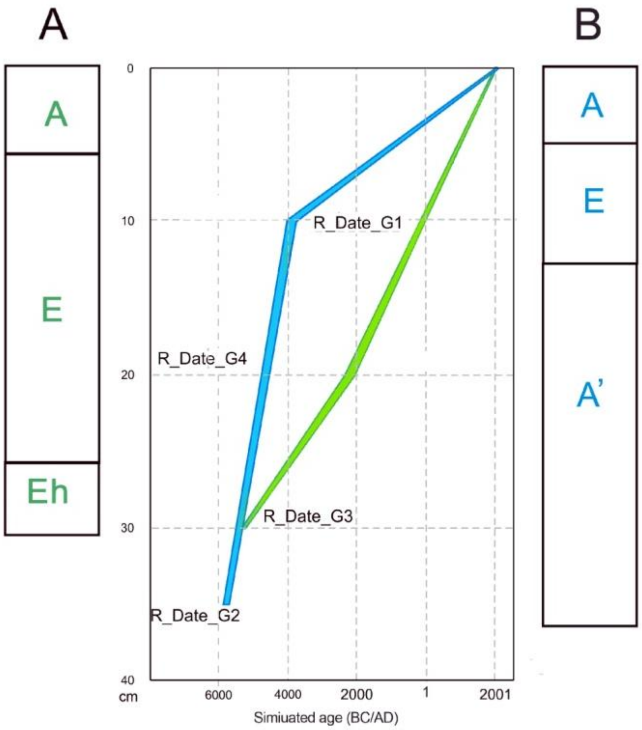

Fig. 2. Simulated carbon age of phytolith concentrates from eluvial and second humus horizons. See Fig. 1 and Table 4 for symbols

4. CONCLUSION

It was found that the process of changing with time is common for phytolith assemblages and phytolith occluded carbon age from the heavy loam forest soils, but the rate and the depth of new portions of phytoliths input, resulting in rejuvenating of phytolith organic carbon, depends on the extent of the texture differentiation of the soil profile.

Age-depth modeling of 14C phytolith and corroded phytoliths profile distribution made it possible to determine more modification in phytolith profiles and rejuvenation of phytolith carbon in soil of the eluvial ecosystem, and less pronounced in the transit ecosystem.

In spite of the phytolith carbon rejuvenation, the soil phytolith profiles, having quite similar characteristics (the ratio of different phytolith groups, phytolith profile distribution), seemed to be relatively weakly transformed with time.

The radiocarbon dates for phytolith occluded carbon of the second humus horizons in different ecosystems of the catena were rather close (*c.a.*5500 B.C.), therefore their phytolith content and assemblages should be considered synchronic with the dark-humus Middle Atlantic Holocene stage of soil formation.

Phytolith analysis of the forest soils is recommended to be conducted together with their morphological, genetic and catenary analyses in order to get a deeper insight into the phytolith profile development and assess its rate depending on soil location in respect to relief.

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COMPETING INTERESTS

Author has declared that no competing interests exist.

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