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Calcium carbonate accumulations in Technosols of Moscow city

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Abstract

Purpose Complex phenomena of the precipitation and accumulation of calcium carbonate in urban soils and sediments have been studied. They are linked to the interactions between the lithosphere, the biosphere, and the atmosphere. Calcium and its compounds are among the main elements determining soil's morphological, physical, and chemical characteristics. This study was aimed at summarizing information about calcium carbonate concentrations in soils and finding evidence for the formation of carbonate micronodules by pedogenetic processes in Urbic Technosols under automorphic conditions.

Materials and methods Moscow is located within the southern taiga belt, with a humid temperate climate and a percolative water regime. The city's technogenic deposits contain significant amounts of carbonates due to inclusions of construction waste and additions of airborne dust. Contemporary humus-accumulative horizons of Urbic Technosols were found to contain carbonate accumulations (rounded or isomorphic nodules). Samples from such horizons were studied using a polarizing microscope (analysis of thin sections) and a scanning electron microscope with an energy-dispersive X-ray analyzer (element mapping to detect carbonate accumulations). Laboratory analyses of main chemical and physical soil properties were performed.

Results and discussion The analyzed samples contained both primary (i.e., inherited from parent materials) and secondary (i.e., newly formed in soils) calcium carbonates in various forms. Inclusions of solidified mortar (carbonate building solutions) contained dolomite. Although the studied carbonates underwent recrystallization processes, they remained in soils for a long time. Fibrous Mg silicate films deposited at the surface of recrystallization areas were observed. The formation of carbonate micronodules at depths of 15–40 cm in slightly and moderately calcareous humus horizons was investigated. Newly formed calcite was distinguished by a homogeneous composition, a microsparitic size of crystals, and a compact packing within the groundmass. Newly formed calcite was observed within microzones that were free from carbonate inclusions with dissolution features.

Conclusions The dissolution of primary carbonates in soils under humid climate conditions triggers pedogenetic processes of the carbonate heritage redistribution and the mineral matrix transformation. Recrystallized carbonates are the main forms of secondary carbonate accumulations in automorphic Technosols. The presence of compact carbonate nodules composed of microsparitic calcite within the bulk of silicate material may be indicative of carbonate neoformation as a result of interactions between the dissolved CO_2 from root respiration and the soil absorbing complex saturated by Ca from soil solution. This is confirmed by a number of studies on the carbonate isotopic composition in urban soils.

Keywords Soil morphology · SUITMAs · Calcite micronodules · Electron microscopy · Urbic Technosols

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1 Introduction

The precipitation and the accumulation of calcium carbonate in soils and regoliths are very complex phenomena. They are linked to the interaction between the lithosphere, the biosphere, and the atmosphere (Durand et al. 2014). Calcium (Ca) plays a significant role in plant nutrition, while the most important soil-chemical functions of Ca are associated with its participation in cation exchange reactions and the formation of the soil solution composition. Soil absorbing complex, saturated by exchangeable bases, contributes to the formation and accumulation of stable forms of humic substances. Ca acts as a coagulator in processes of soil structuring and creating favorable physical conditions for living organisms. The diversity of forms of calcium carbonate accumulations makes it possible to use them as indicators of soil formation conditions and processes (Kovda 2008).

It is known that urban soils formed on man-made deposits contain significant amounts of carbonates due to inclusions of construction debris (Davidson et al. 2006; Adderley et al. 2014; Howard and Orlicki 2016; Greinert and Kostecki 2019). From the first years of studying the soils of Moscow, it has been noted that not only individual structural elements of soils react with a 10% HCl solution, but a continuous effervescence of fine earth is frequently observed (Stroganova et al. 1998). This makes it possible to suppose that an active dissolution and redistribution of carbonate inclusions occurs in urban soils.

Studies on carbonate segregations in the soils of Moscow are very important, because carbonation phenomena can change the direction of soil-forming processes typical for the natural zone of southern taiga, where the city is located. The presence of carbonates contributes to soil structuring and the formation of a strong microporous matrix and hinders the migration of highly dispersed components, i.e., makes it difficult to mobilize dispersed particles, which are carriers of scattered heavy metals (Dobrovol'skii 2001). Soil studies in different cities around the world show a significant contribution of inorganic carbon to the total carbon stocks in urban soils. Urban soils accumulate significant amounts of inorganic carbon in forms of carbonates and bicarbonates, both in upper and lower horizons (Lorenz and Lal 2015). Studies on ancient cultural layers (including those in Moscow city) describe various newly formed accumulations of carbonates (Alexandrovskaya and Aleksandrovskiy 2000; Alexandrovskiy 2007; Davidson et al. 2006; Kazdym 2006; Macphail and Goldberg 2014; Itkin et al. 2016). Apparently, anthropogenic calcite formation—anthropogenic calcretisation processes—develop not only in deep cultural layers but also in surface layers of urban soils (Manning et al. 2013; Itkin et al. 2016). Up to $39.4 \pm 8.8\%$ of the carbonate C is captured from the atmosphere through hydroxylation of dissolved CO₂ in high pH solutions within soils (Washbourne et al. 2012).

2 Studies on modern soils of Moscow

Table 1 contains the available data on the forms of secondary carbonates in modern soils of Moscow. In most cases, soil micromorphological studies reveal features of dissolution and recrystallization of carbonate debris inclusions.

Spherical carbonate nodules (spherulites) can be formed in the replacement of accumulations of domestic animal excrements (e.g., in upper horizons of lawn soils). Such pedofeatures have been reported in association with a high activity of *Escherichia coli* at the archeological excavation site of a cesspool of the nineteenth century within the Moscow center (Alexandrovsky et al. 1998; Prokof'eva et al. 2001).

The greatest abundance and diversity of forms of secondary carbonates have been found in hydromorphic and sealed soils, where moisture stagnation is possible.

Table 1 Forms of calcium pedofeatures in Urbic Technosols of Moscow

References	Conditions of formation	Shapes of Ca-carbonate concentrations	Formation processes
Tselischeva and Stroganova (1996)	Automorphic uncovered soils	Calcite nodules (0.1–0.5 mm)	Dissolution of inclusions, redistribution of dissolved material
Prokof'eva et al. (2001)	Sealed soils (Urbic Ekranic Technosols)	Areas with carbonate micrite impregnations of fine earth (K-fabric)	Dissolution of inclusions, redistribution of dissolved material
	Automorphic uncovered soils	Microfragments of limestone, concrete, cement, plaster, and their recrystallization	Dissolution of inclusions, redistribution of dissolved material
	Biota influence	Pale yellow spherulites (0.01–0.1 mm) with the characteristic cross-shaped extinction optical effect	Vital activity of Escherichia coli
Prokof eva et al. (2010)	Semi-hydromorphic soils	Areas with carbonate micrite impregnations of fine earth (K-fabric); carbonate-clay and carbonate covers and crusts; reddish yellow and light red fibrous hemispherical concretions of complex Fe–P–Ca–C–O composition (0.01–0.5 mm), forming crusts on the pore walls	Dissolution of inclusions, redistribution and recrystallization of dissolved material (micrite);
Prokof'eva et al. (2017)	Low-thickness soils, above the asphalt surface (Leptic Urbic Technosols)	Dissolution of inclusions, redistribution and recrystallization of dissolved material (micrite)	

Waterlogged areas in cities are usually sealed. The surface is raised due to the accumulation of anthropogenic sediments within depressions. As a result of the river flow regulation, floodplains of urban rivers develop mostly due to alluvial sedimentogenesis. However, flooding and accompanying hydrogenic accumulation processes (typical for formation of floodplain and other hydromorphic soils) have not been completely stopped, as their influences on groundwater table are still observed within areas of former ravines and river valleys. Not only preserved natural soils of parks but also Technosols are subjected to hydromorphism. In submerged horizons, carbonate crystallization features are represented by continuous carbonate impregnations (microcrystals—micrite) from a depth of 20-30 cm in natural soils of floodplains and from a depth of about 1 m in waterlogged Technosols. Clay-carbonate films have been found within the horizons that had combined features of alluvial soil formation and urban pedosedimentogenesis (Prokof'eva et al. 2010).

Newly formed rounded microconcretions are mentioned in the study on automorphic soils of the Rozhdestvenskiy Monastery in the Moscow center (Tselischeva and Stroganova 1996), which is also the very first publication on micromorphology of Technosols in Moscow. We have also seen such micronodules in Urbic Technosols from different parts of the city. There is a question about the origin of these micronodules, i.e., whether they are true pedofeatures (really soil neoformations) similar to those in natural soils (Kovda and Mermut 2014). Based on their sizes and general morphology, we can also suggest that they have similarities with the following: rounded micro-inclusions of technogenic materials (Konstantinov et al. 2018), aggregates of salt crystals resulting from atmospheric depositions (Prokof'eva et al. 2015), and carbonate concentrations from earthworm secretions (Zamanian et al. 2016).

The purpose of this study was to find evidence for the pedogenetic origin of carbonate micronodules under automorphic conditions in Urbic Technosols.

Table 2 Locations and properties of the studied soil horizons

Habitat, soil, horizon, depth	pH _{water}	C org, %	CaCO ₃ , % in fine earth	\sum Exchangeable cations (Na, K, Ca, Mg), cmol (+)/kg	Ca ²⁺ ,% from the amount of exchangeable cations	Bulk density, g/cm ³	USDA texture
Nodules were detected under the electron microscope							
The lawn near the highway BS10 Urbic Technosol (Novic) buried humus horizon under the substrate imported for reclamation 10–20 cm	8.1	2.04	1.5	20.58	78	1.2	Sandy loam
Lawn inside the block of building TU6 Urban Technosol Stratified contemporary humus horizon. The lower part 10-30 cm	7.5	0.34	0.7	9.56	75	1.4	Loamy sand
Lawn inside the block of building MSU5 Urbic Technosol Stratified contemporary humus horizon. The lower part 10-40cm	8.1	0.69	3.2	13.12	80	1.3	Sandy loam
No nodules were detected under the elec	etron micr	oscope					
The lawn near the highway MSU1 Leptic Urbic Technosol humus horizon over crumbling asphalt cover 2–10 cm	8.0	1.30	2.7	2.34	26	1.0	Loam
The lawn in the botanical garden AG1 Urbic Technosol (Hortic) Stratified contemporary humus horizon. The lower part 17–46 cm	7.5	2.10	2.3	14.16	86	1.13	Sandy loam
Lawn inside the block of building S15 Urbic Technosol (Novic) The buried humus horizon under man-made substrate 39-66 cm	8.2	0.39	1.2	19.66	77	n.d.*	Silt loam
The lawn near the highway BS10 Urbic Technosol (Novic) A young humus horizon forming on a reclamation substrate 0–10 cm	7.6	5.63	1.7	25.26	75	1.2	Sandy loam

*n.d. Not determined

3 Materials and methods

The study had been conducted in the city of Moscow (Russia), which is located in the southern taiga zone, with a moderate and moderately continental climate and a percolative water regime. The average annual temperature is + 5.8 °C, and the average annual precipitation is 700 mm (Revich 2006). There is a seasonal freezing of soils. Both air and soil temperatures have increased since the middle of the twentieth century (Lokoshchenko and Korneva 2015).

Most of the natural soils and soil parent materials of the Moscow Region are free from carbonates. Within urban areas, soils are formed mainly on technogenic parent materials, which include large amounts of carbonate-containing construction debris (limestone gravel, mortar, plaster, etc.). Other sources of calcium carbonate in Moscow soils include animal bones, mollusk shells, and atmospheric solid fallouts, with up to 10% CaCO3 found in road dust, which is deposited at a rate of 30 g/m² per year (Prokof'eva et al. 2015).

The studied Urbic Technosols are typical city soils found in urban environments around the world (including Moscow). Their profiles are known to contain abundant debris and wastes in various forms, from microfragments (diameter \leq 2 mm) to large inclusions. The material of such soils is calcareous, their pH level is higher than that of seminatural soils of city suburbs, and there is a tendency for accumulation of organic carbon and nutrients due to a continuous aggradation of humus horizons (Stroganova et al. 1998; Lehmann and Stahr 2007; Prokofyeva et al. 2011; Hulisz et al. 2018). The most typical humus horizon of cities is named "Urbic" by Stroganova and co-authors (Stroganova et al. 1998). Urbic horizons, which initially form on the surface, often become buried under new materials as a result of construction works and/or slowly buildup due to lawn construction and airborne

Fig. 1 Carbonate micronodules in thin sections (marked with arrows): a rounded micronodule 0.8 mm inside a dense sandy loam-silty structural unit (XPL); b rounded micronodule 0.8 mm inside a dense sandy loam-silty structural unit (PPL); c Isomorphic micronodule 0.2 mm without films on the surface inside the clay-silty material (PPL); d rounded nodule 0.3 mm coated ferrous-humus film (PPL); e isomorphic prolate micronodule 0.6 mm with a fragmentary thin humus film on the surface (PPL); f a 0.6 mm isomorphic prolate micronodule with a fragmentary thin humus film on the surface (XPL)



dust accumulation (Prokofyeva et al. 2011; Prokof'eva et al. 2001, 2017).

The present study was focused on carbonate accumulations in contemporary automorphic humus-accumulative horizons of Urbic Technosols in Moscow. In total, samples were taken from 7 humus horizons of 6 soil pits (Table 2). The MSU5 and AG1 pits were characterized by the presence of thick humus horizons, which had resulted from a long-term deposition (70–150 years old). The TU6 and MSU1 pits had thin humus horizons, which developed on a gradually accumulated substrate (over 50 and 20 years old, respectively). The TU6 profile was also characterized by features of repeated compost additions. The S15 profile had a similar humus horizon buried under construction debris. In the BS10 profile, the upper part of the humus horizon (40 years old) was removed and replaced with a layer (10 cm) of compost (approximately 10 years ago during a lawn construction).

The samples for the present study were taken from the humus horizons, where carbonate accumulations (nodules) of rounded or isomorphic shapes and microscopic sizes have been identified in our previous meso- and micromorphological studies on those soils (Prokof'eva 2016).

In the present study, morphological properties of such carbonate micro-accumulations within the humus horizons were studied at different levels of organization, i.e., at different magnification scales. We used a binocular magnifier (magnifications from $\times 4$ to $\times 56$) to identify areas of carbonate accumulations within undisturbed samples, which were subsequently sub-sampled for investigations under a JEOL 6610 LV scanning electron microscope with an INCA XACT energydispersive X-ray spectrometer. Element mapping was used to detect carbonate accumulations, i.e., to differentiate microcrystalline calcite from silicate grains of similar shapes. Element maps show the distribution of chemical elements at the sample surface within the scan area, which makes it possible to identify areas of high concentration of components such as calcium and magnesium characteristic of carbonate accumulations. The visual representation of the chemical composition significantly speeds up the search process and improves its quality. We also examined thin sections under LOMO POLAM I-213 (working magnifications $\times 25$, $\times 100$, $\times 250$, and $\times 400$) and NIKON E200 POL (working magnifications $\times 40$, $\times 100$, and $\times 400$) polarizing microscopes.

Chemical and physical properties of soils were analyzed using conventional techniques. Concentrations of cations and anions of water-soluble compounds were determined in water extracts from the studied soils. Concentrations of exchangeable cations were measured in 0.1 N NH₄Cl-ethanol extract after leaching of water-soluble compounds (Vorob'eva 2006). K and Na were determined by the flame photometry, Ca and Mg by the atomic absorption spectrometry, and anions by the ion chromatography method. The pH was determined potentiometrically in an aqueous 1:2.5 suspension. It was assumed that all soil horizons with a pH level greater than 7 had the base saturation of nearly 100% (Vorob'eva 2006).

Fig. 2 The composition of the carbonate micronodule



The content of carbonates was determined volumetrically using a calcimeter, in three replications. The organic carbon content was determined by the bichromatic oxidation with a photometric termination.

The soil texture was characterized in the field in compliance with the FAO Guidelines for Soil Description (2006). The bulk density values were determined in undisturbed core samples taken from the walls of soil pits by metal cylinders of a known volume (32.5 cm³), in three replications (IUSS Working group WRB 2015).

4 Results

Most of the studied humus horizons of the Urbic Technosols contained inclusions of construction debris discernible at both macro- and micro-scales and had similar chemical characteristics, i.e., a slightly alkaline reaction (pH 7.5–8.2) and a high content of exchangeable Ca^{2+} , with a total concentration of exchangeable cations of 10–20 cmol (+)/kg. The content of CaCO₃ in fine earth was generally not very high—only 1–3%.

One of the studied soils—Leptic Urbic Technosol (pit MSU1)—differed from the others by its chemical properties (with approximately equal concentrations of exchangeable Ca^{2+} , Mg^{2+} , K^+ , and Na^+ and a very low cation exchange capacity), because it was formed as a result of airborne dust deposition over a demolished road surface.

The texture of the studied humus horizons varied from sandy loam to loam. The content of organic carbon ranged quite widely, i.e., from low (0.39%) to very high (5.63%), which could be explained by different factors affecting the formation of humus horizons, e.g., the frequency and thoroughness of removing fallen leaves, the presence or absence of a continuous grass turf, and compost additions to the soil surface (Table 2). For the same reasons, the bulk densities of the studied horizons were also different.

In thin sections newly formed carbonate accumulations were observed. They were represented by excretion forms, i.e., micronodules (0.1–1.0 mm) of rounded (Fig. 1a–d) and isomorphic (Fig. 1c–f) shapes, consisting of crystals of 4–10 μ m in size. Most of those micronodules were dense (Fig. 1a–c). Some micronodules had signs of destruction on the periphery of a dense core (Fig. 1e, f). Some of them were covered with humus and ferruginous-humus films (Fig. 1d).

As a result of element mapping, calcium carbonate micronodules were found in samples from humus horizons of three pits: BS10 (depth 10–20 cm), MSU5 (10–40 cm), and TU6 (10–30 cm). Despite that those carbonate micronodules looked similar to fine particles of the ground-mass (Fig. 2), they were clearly differentiated by X-ray signals of red and green colors, respectively, on the element maps. It was found that micronodules were dense and homogeneous in composition and consisted of calcite crystals of >4 μ m in

sizes (Fig. 2). In the center of micronodules, there were zones with slightly enlarged crystals. On the periphery of the dense micronodules, there were clusters and individual crystals of calcium carbonate, which were protruding into the surrounding soil material of a silty texture.

Accumulations of carbonate building materials, which prevailed in the studied soil horizons, mostly consisted of dolomite (Figs. 3 and 4). Ca and Mg were marked by X-ray signals of red and green colors, respectively, on the element maps both in the fragment of a pure carbonate mortar and in a fragment of a silicate concrete (Fig. 4). Their recrystallization products (Figs. 5 and 6) varied in composition and often consisted of calcite (Fig. 5). Carbonate crystals within fragments of solidified mortar had both small (micrite) and large (sparite) sizes (Fig. 3). Crystal sizes of $4-10 \mu m$ (microsparite) within recrystallization zones are previously reported in natural soils (Kovda 2008). Figure 6 shows Mg silicate fibrous coatings forming tracery retting or dense crusts



Fig. 3 Carbonate micro-inclusions in the material of urban soils: a microfragment of mortar with sparitic crystals of $50-100 \ \mu m$; b carbonate mortar where silicate grains are bonded with microcrystalline carbonate cement

Fig. 4 Structure and composition (elemental mapping) of microfragments of building mortar



on the surface of carbonate crystals within the buried humus horizon (pH = 8.2) of pit S15.

5 Discussion

Results of studies on carbonate accumulations in Moscow city soils (predominately, Urbic Technosols) have been summarized in this paper. These include both primary carbonate inclusions, the source of which is construction debris and airborne dust deposition, and secondary (newly formed) accumulations of calcium carbonate in various forms. The driving forces of transformation of soil carbonates include the humid climate, which promotes dissolution and re-deposition processes, and living organisms. The forms of carbonate accumulations associated with different moisture conditions include impregnations and crusts within horizons subjected to flooding and nodules and recrystallization pedofeatures within automorphic horizons (Tselischeva and Stroganova 1996; Kazdym 2006; Prokofyeva et al. 2011; Prokof'eva et al. 2001, 2017).

Sources of primary carbonates are diverse. Mostly, these are man-made inclusions. Observations show that they have a significant stability in a humid climate, as they are stored both at the macro- and micro-scales in soil horizons of different ages (Alexandrovskaya and Aleksandrovskiy 2000; Kazdym 2006; Davidson et al. 2006; Prokof'eva et al. 2001; Adderley et al. 2014; Konstantinov et al. 2018; Greinert and Kostecki 2019).

Our research has confirmed the results of previous studies. Anthropogenic carbonate inclusions (fragments of solidified mortar, plaster, limestone, etc.) were found in all studied horizons at all levels (macro-, meso-, micro-, and submicro-) of soil organization. Data from X-ray analysis showed that inclusions of carbonate mortar often contain both dolomite and calcite, in contrast to carbonate pedofeatures. Traces of dissolution and recrystallization processes were observed in carbonate inclusions. However, it should be noted that they remained in the soil for a long time, despite their occurrence close to the soil surface and the percolative water regime. The long-term preservation of carbonate inclusions in soils could be associated with the observed phenomenon of deposition of newly formed fibrous Mg silicates within the recrystallization zone on the surface of carbonate inclusions (see Fig. 6), which has previously been observed in soils of dry subtropical climate (Mees 2014). The dissolution of carbonate inclusions with a significant Mg content creates conditions (probably, locally) for the dissolution of silicates and the formation of fibrous Mg silicates (palygorskite) (Mees 2014).

The case of carbonate micronodules formation at depths of 15–40 cm within slightly and moderately calcareous humus horizons under automorphic conditions was analyzed. Our search for such micronodules involved mesomorphological, micromorphological (thin section), and submicromorphological



Fig. 5 Recrystallization area of carbonate inclusions (a) and its composition (b)

(scanning electron microscopy) methods. As a result, for the first time, we found newly formed carbonate micronodules in three humus horizons (see Table 2), which had Ca contents of more than 75% of the sum of exchange bases, a weakly alkaline reaction ($pH \ge 7.5$), a moderately compact consistency, and a sandy loam and loam texture. According to the subdivision of soil morphological transformation by Hulisz with co-authors (Hulisz et al. 2018), soils with calcite micronodules can be referred to as urban soils of long-term deposition.

For the first time, we discovered indications of a newly formed character of carbonate micronodules, which included a homogeneous calcite composition, a microsparite (fine and medium silt) size of crystals, and a compact packing within a silicate groundmass. An absence of dissolving carbonate inclusions nearby also indicated that such pedofeatures did not result from recrystallization. It can be assumed that they resulted from an interaction of carbonic acid of the soil solution with Ca contained in the soil absorbing complex. The atmospheric carbon is accumulated by green plants, which emit carbon dioxide (CO_2) during respiration and decomposition. CO_2 accumulates directly under the main mass of roots and dissociates in soil solution under conditions of insufficient aeration. This is confirmed by a number of carbonate isotope composition studies on urban and natural soils, which show that a significant proportion of inorganic carbon in soils is originated from photosynthesis of modern vegetation (Renforth et al. 2009; Washbourne et al. 2012; Manning et al. 2013). Thus, the mechanism of formation of carbonate micronodules in city soils is similar to that in natural humus-rich soils saturated with bases.

6 Conclusions

Our studies have shown that accumulations of primary carbonates (inclusions of solidified mortar and other construction debris) have undergone dissolution under the humid climate conditions of Moscow, which has triggered soil-forming processes associated with the redistribution of the carbonate heritage and the transformation of the mineral matrix under conditions of high pH values of the soil solution. The



Fig. 6 The peripheral surface of the mortar inclusion (recrystallization area): \mathbf{a} , \mathbf{b} Carbonate crystals are covered with films of newly formed fibrous silicates Mg (marked with arrows), \mathbf{c} the dense cover of fibrous silicates, and \mathbf{d} the composition of the silicate crust (area of spectrum 4)

recrystallization of dissolved primary carbonates was shown to be the main process of secondary carbonate formation in such automorphic urban soils.

The presence of carbonate nodules composed of microsparite-sized calcite crystals in the bulk silicate material may be indicating not only the dissolution and redistribution processes of carbonate heritage material but also the possibility for carbonate neoformation by the interaction between dissolved CO_2 originated from root respiration and soil absorbing complex saturated by Ca.

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