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Calcium Carbonate Formations in Edaphic Components of Ecosystems

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Abstract

Calcium carbonate is an important component of the edaphic components of ecosystems (soils, river and lake sediments, suspensions). Calcite, aragonite, and vaterite are the three its polymorphs, in order of decreasing thermodynamic stability. Aragonite and vaterite, as less thermodynamically stable and more soluble polymorphs than calcite, are found in natural setting much rare (except for their biogenic formations) and are much less studied. Although they are important precursors in many carbonate-forming systems. We found examples of vaterite microforms in the river ice suspensions, river and lake bottom sediments of the different natural zones - the Amur and the Simmi Rivers (the Amur River basin, Khabarovsk Region, Russia), the Jordan River (Jordan). Vaterite was originally thought to have a very short lifespan; however it may have greater longevity. In the presence of low molecular amino acids ex and in situ vaterite is stabilized as toroidal suprastructures. These microforms of CaCO₃ are characterized with internal diameter of 8–10 µm and cross-sectional radius of about 1 µm. The age of peat stratum, in which the toroidal vaterite microforms were found, is more than 6000 years. Another polymorphic modification of CaCO₃, aragonite, is stabilized in the bottom sediments of drainless lakes of the arid zone with extremely high salinity, e.g. the famous ooids of the Grand Salt Lake (USA). However, outside the "habitat" - the salt water of the lake - aragonite ooids are destroyed.

Keywords: calcium carbonate, calcite, aragonite, vaterite, river, lake, bottom sediments.

1. Introduction

Calcium carbonates are an important component of the terrestrial carbon pool. The global importance inorganic carbon is reflected by the fact that it links the long-term geological cycle with the fast bionic cycle (Zamanian et al., 2016) and this linkage is ongoing in the edaphic components of ecosystems (soils, river and lake sediments, suspensions). The main attention of scientists was and is being paid to geochemical confinement, processes of formation, content and morphology of calcite, the most thermodynamically stable polymorphic modification of calcium carbonate (Lal et al., 2016).

Climate, i.e. precipitation and temperature, is suggested as the main controlling factor for formation and localization of calcite in soils (Borchardt, Lienkaemper, 1999; Eswaran et al., 2000).

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Accumulation of calcite near the soil surface is common for arid and semi-arid zones at mean annual precipitation (MAP) <500 mm (Landi et al., 2003; Retallack, 2005). Calcite can accumulate in soils in a wide range of temperatures from very hot conditions in hot deserts (Amit et al., 2011; Thomas, 2011) to cold climatic zones such as tundra (Courty et al., 1994; Pustovoytov, 1998) and Antarctic Dry Valleys (Gibson et al., 1983; Bishop et al., 2014). The amount and seasonal distribution of MAP controls the depth of carbonate leaching and accumulation (Egli, Fitze, 2001). The balance between MAP (total amount and seasonality) and evapotranspiration (driven by temperature and wind speed) determines the rates and the amounts of carbonates as well as the depth of carbonates accumulation. Hence carbonates are formed during soil drying when evapotranspiration exceeds precipitation (Birkeland, 1999; Gile et al., 1966; Hough et al., 2014; Rawlins et al., 2011).

The calcite formation in the river and lake waters and, accordingly, in the bottom sediments is also a characteristic process of arid and semi-arid zones (Strakhov et al., 1954). Due to the prevalence of evaporation over precipitation, surface waters (rivers and lakes) are oversaturated by Ca^{2+} and CO_3^{2-} that leads to the formation and precipitation of $CaCO_3$ in bottom sediments. In the humid zone contrary, $CaCO_3$, as a relatively highly soluble compound, is in a state of undersaturation in the surface waters. Therefore, it does not accumulate either in alluvium or in bottom sediments.

Two other polymorphic modifications of calcium carbonate, aragonite and vaterite, as less thermodynamically stable and more soluble than calcite (Friedman, Schultz, 1994; Lee Kabalah-Amitai et al., 2013), are found in natural setting much rare (except for their biogenic formations) and are much less studied. Aragonite only occasionally occurs in the soil profile (Wing, 2016; Quigley, Dreimanis, 1966). In lake sediments, aragonite is more common (Tompa et al., 2014).

Vaterite is thought to be metastable under all known conditions and has a higher solubility than the other CaCO₃ polymorphs, calcite and aragonite (Plummer, Busenberg, 1982; Ogino et al., 1987). Although vaterite was precipitated inorganically in the laboratory, it was originally thought not to occur as a naturally forming mineral (Ievins et al., 1955). Natural occurrences of vaterite are largely associated with organic tissue, gallstones, urinary calculi and microbial biscuits (Prien, Frondel, 1947; Hall, Taylor, 1971; Lowenstam, Abbott, 1975; Rodgers, 1983; Giralt et al., 2001). However, rare occurrences have been reported in a contact metamorphic aureol in Ireland (McConnell, 1960), in zones of thermal metamorphism in Israel (Kolodny, Gross, 1974), and in carbonate concretions (Fong, 1981). Vaterite has also been found in drilling mud (Friedman et al., 1993; Friedman, Schultz, 1994).

Rowlands and Webster (1971) reported the first occurrence of vaterite precipitating spontaneously from natural waters in Holkham Lake, UK. However, a reexamination of this site did not detect vaterite (Lucas, Andrews, 1996; Friedman, 1997). The next time vaterite was identified in precipitates forming at a supraglacial sulfur spring, in Borup Fiord Pass, northern Ellesmere Island, Canadian High Arctic (Grasby, 2003). The unusual conditions of the site, including an extremely cold climate, supersaturated alkaline waters, and the presence of gypsum, mimics conditions used to grow vaterite in laboratory experiments. Found vaterite is characterized by the spherical habit (size of vaterite particles was from 0.5 up to 10 μ m), δ^{13} C values are indicative of an inorganic origin.

Previously it was supposed that vaterite is stable $CaCO_3$ phase at low temperatures <10°C at 1 atm (Albright, 1971). Kralj et al. (1990) indicate that pure vaterite could only precipitate under a narrow range of initial conditions and within a pH range from 9.3 to 9.9. However, mixed calcite/vaterite was precipitated at slightly lower pH (8.5) in experiments by Vecht and Ireland (2000). Experiments by Flörke W. and Flörke O. (1961) also show that vaterite growth is favored by reaction of alkaline solutions with crystalline gypsum. Vaterite is highly unstable when exposed to water; it can recrystallize to calcite within 20 to 25 h at room temperature (Silk, 1970). At higher temperatures (~ 60°C), vaterite will transform to aragonite in 60 min (Ogino et al., 1987). As a result of these rapid reaction rates at low temperatures, vaterite was originally thought to have a very short lifespan. However, recent dating of natural occurrences suggests that vaterite may have greater longevity up to 20–45 years in drilling mud (Friedman et al., 1993; Friedman, Schultz, 1994).

The task of this work is to study the manifestations of metastable modifications of calcium carbonate (aragonite and vaterite) in river suspensions and bottom sediments of rivers and lakes of different natural zones.

2. Materials and Methods

Study area

River (the Jordan River, Jordan, site 1) and lake (the Grand Salt Lake, USA, site 2) bottom sediments of semi-arid and humid zones (the Simmi River, Far East, Russia, sites 3 and 4) were chosen as objects of study. Additionally, ice cores were studied (the Amur River, Khabarovsk, Far East, Russia, site 5).

Sample 1 – bottom sediments of the Jordan River were sampled near Al-Maghtas ruins (300 m upstream, 31°50'13" N 35°32'47" E) in May 2014. The land is generally arid. The average monthly temperature (Amman) ranges between 8 °C and 26 °C. Winter rains enable moderately strong weathering of silicate minerals and especially enable the dissolution of limestones and other calcareous parent rocks.

Sample 2 – bottom sediments, edge of the Great Salt Lake (40°43'04" N 112°13'58" W), were sampled in 2017 by V. Kislovsky (Technion–Israel Institute of Technology, Haifa, Israel). Lake locates in the semi-arid intermountain West of the USA (Rey et al., 2016). It is the largest salt water lake in the Western Hemisphere. The lake's size fluctuates substantially due to its shallowness (from 2,460 up to 8,500 km²) (Arnow, Stephens, 1990; Gillies et al., 2015). As it is endorheic (has no outlet besides evaporation), it has very high salinity (far saltier than seawater). The salinity of the lake's main basin, Gilbert Bay, is highly variable and depends on the lake's level; it ranges from 5 to 27 %. For comparison, the average salinity of the world ocean is 3.5 % and 33.7 % in the Dead Sea. The ionic composition is similar to seawater, much more so than the Dead Sea's water; compared to the ocean, Great Salt Lake's waters are depleted in calcium. Aragonite ooids are interpreted to have been actively forming throughout most of the Holocene in the lake in the shallow nearshore in hypersaline water (Eardley, 1938; Carozzi, 1962; Kahle, 1974; Halley, 1977; Pedone, Norgauer, 2002; Rey et al., 2016). The δ^{18} O composition of the carbonate minerals is consistent with the formation of highly evaporated saline water (Langbein, 1961; Arnow, Stephens, 1990).

Sample 3, 4 – bottom sediments of the Simmi River, the main tributary of the Bolon Lake (the Amur River basin, Khabarovsk Region, Russia). Bolon is floodplain lake, a residual element of the ancient channel of the Amur River (Avaryaskin, 1970). Sediments were sampled at the end of May 2018 during the period of abnormally low water level. Site 3 ($49^{\circ}42'23'' \times 136^{\circ}16'53'' E$) is located in the downstream on the right bank of the Ersuyn channel, which connects the Kiltasin and the Bolon Lakes. The Kiltasin Lake has a water mirror in the period of high and medium water levels; with a low level water occupies only the channel. As a result, accumulated in the Kiltasin Lake sediments are removed through the Ersuyn channel. Therefore, the bottom sediments can be considered transitional from lake to river (lake-river sediments). Site 4 ($49^{\circ}30'21'' \times 136^{\circ}01'34'' E$) is located in the upstream of the Simmi River. The climate of the region is cool, moderately humid, transitional from the northern monsoon to the continental. In the cold season continental polar air masses dominated. The average January temperature is -28 °C. The annual temperatures range is 48-50 °C. Winters are not snowy; soils freeze up to 160 cm. Summer is warm and humid. July – September accounts for 50-55 % of the annual precipitation (400-500 mm) (Nikonov, Anisimova, 1974).

Sample 5 – ice core drilled in a zone of moderately hummocky ice, Middle Amur, 350 m from the edge of the right bank (48°36'18" N 135°02'24" E), ice thickness 1.33 m (Kharitonova et al., 2017). Sampling was carried out in March 2014 (with a minimum water level and maximum ice thickness). By color, transparency of ice, presence of inclusions the layers (0–40, 40–43, 43–52, 52–70, 70–117 and 117–133 cm) were distinguished in the core.

Methodology

The major methods were granulometric and gross chemical analyses and scanning electron microscopy (SEM). The SEM analysis was carried out using VEGA 3 LMH (TESCAN, Czech Republic). For the analysis, the samples, after grinding and sieving through a 2-mm sieve, were prepared via pouring, Pt-spraying and magnification of up to 20,000. A backscattered electron detector (BSE detector) was used for the analysis of phases with a high atomic number. When images are acquired using a BSE detector, phases with a high average atomic number are reflected

in contrast more vividly than those with a lower atomic number. The X-max 80 energy-dispersive spectrometer (Oxford Instruments, UK) was used to analyze the elemental composition of the most representative regions. The capture area of the microanalysis was about 1 μ m in diameter. If a smaller object was scanned, the result was distorted due to the influence of the surrounding matrix or the carbon table of the device. The granulometric composition (without decomposition of carbonates) determined with the laser diffraction method on a particle size analyzer SALD-2300 (SHIMADZU, Japan) (Rawle, 2017; Wolform, 2011). The contents of selected chemical elements were determined via the X-ray fluorescence method (XRF) (Pioneer S4, Bruker AXS, Germany), using the silicate technique. The SEM and XRF analyses were carried out in the Analytical Centre at the Institute for Tectonics and Geophysics, Khabarovsk, Far-eastern Branch of the Russian Academy of Sciences.

3. Results and Discussion

An analysis of the metastable polymorphic modifications of calcium carbonate (aragonite and vaterite) in the selected series of samples will be preceded by consideration of some their physical and chemical properties – the gross chemical composition and particle size distribution (PSD). According to the Table 1, the change in the calcium content and, accordingly, carbonates in sediments does not contradict the climatic zonality of their distribution: a high calcium content in the sediments of the semi-arid zone and much less in those of the humid zone. Highly dispersed bottom sediments of the Jordan River are characterized by extremely high calcium content (more than 40 % CaO from the total composition). Lake-river sediments of the Simmy River (downstream) have the close granulometric composition (the predominance of silt). But since the basin of the Simmy River is located in the humid zone, and in addition, the processes of physical weathering prevail in the territory (as a result of deep freezing of soils), which is confirmed by the PSD data of the upstream river bottom sediments, the calcium content is almost two orders of magnitude lower.

No	Gross chemical composition, %								Content of granulometric fractions*, %					
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	Sr**	<2	2-	20-	50-	200-	
										20	50	200	2000	
Jordan River														
1	29.7	5.7	7.0	45.1	6.1	1.3	1.4	2831	12.3	63.1	15.8	9.9	0	
Grand Salt Lake														
2	55.2	2.9	19.7	16.4	2.6	0.6	1.4	854	_	_	_	_	-	
Simmi River (downstream)														
3	70.0	16.4	5.1	0.7	1.4	2.4	2.7	192	7.0	48.0	27.1	17.9	0	
Simmi River (upstream)														
4	76.4	12.7	2.5	0.7	0.8	3.2	2.8	170	0	4.1	3.3	56.6	36.1	
	Amur River (ice inclusions***)													
5	_	_	—	—	_	—	_	_	0	0	0	0.1	99.9	

Table 1. Some physical and chemical properties of sediments

* – size fractions in μm; ** – content in mg/kg; *** – 43–52 cm ice layer; dash – no data.

The absolute predominance of coarse sand (200–2000 μ m) in the Amur ice (43–52 cm layer) is associated with the formation of the layer at the end of the freeze-up and participation in its composition of bottom ice (Kharitonova et al., 2017). Calcium carbonate was detected only in this layer (Figure 1). It could be assumed that calcium carbonate may have entered into the layer from bottom sediments with rising bottom ice. However, our previous studies of the Amur River bottom sediments do not confirm this assumption (Chizhikova et al., 2004, 2011; Kharitonova et al., 2014). Most likely, CaCO₃ is formed in the layer during freezing of river water, since it contains the highest concentrations of bicarbonate ion HCO₃⁻ in at the end of the freeze-up (up to 60–80 mg / l).

Calcium carbonate is represented by particles up to $100-200 \ \mu m$ in size. Most of them are poorly (Figure 1*a*, *c*) and moderately crystallized (Figure 1*b*, arrow). But there are also spherical shaped particles of CaCO₃ (Figure 1*d*). Similar formations (Figure 1*e*, *f*) were found in precipitates

forming at a supraglacial sulfur spring, in Borup Fiord Pass, northern Ellesmere Island, Canadian High Arctic (Grasby, 2003). Vaterite found at the site was characterized by 2 to 10 μ m rounded to spherical shaped particles (comprising smaller 0.5 to 2 μ m spheres) as both individuals and in chainlike structures. Unfortunately, the content of CaCO₃ in our sample did not allow us to identify μ -CaCO₃ by means of power XRD. However, the morphology of the particles and similar conditions for their formation allow us sufficient confidence to assume that is vaterite.

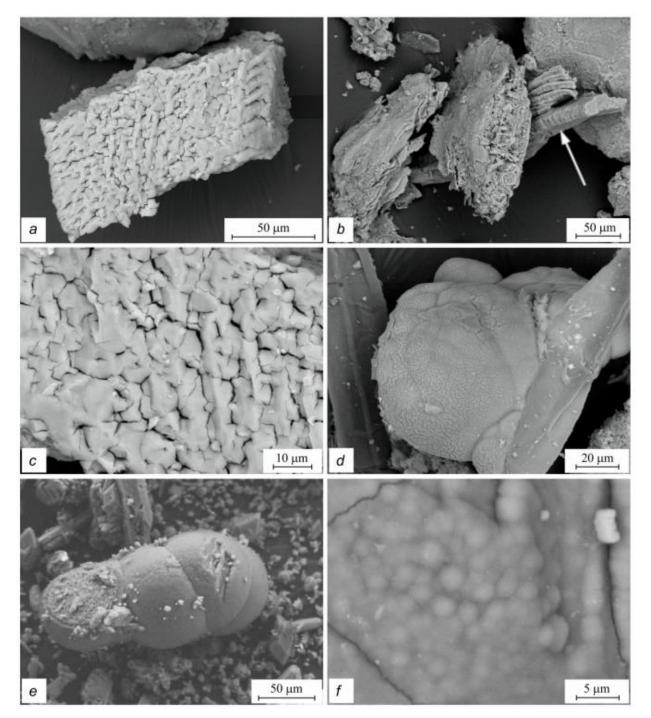


Fig. 1. SEM images of CaCO₃ inclusions in the Amur ice (a - d) and vaterite particles from supraglacial spring, Canadian High Arctic (e, f, Grasby, 2003): a, c – poorly and b – moderately crystallized calcite; d – spherical calcium carbonate structures; e – chainlike structures of spheres, f – close-up of (e) showing micron-sized spheres that comprise the larger spherical structures

Since vaterite was not diagnosed in the bottom sediments of the Middle Amur, but was found in ice cores, we tried to assess the possibility of its formation in the sediments of the Lower Amur. The Simmy River (the Amur River basin) was chosen for this. It is almost a degree to the north, it is much colder there, winters are lightly snowy and soils freeze up to 160 cm. As SEM and EDS analyzes showed, bottom sediments of the Simmi River are characterized by a relatively high content of sphene CaTiSiO₅ (Figure 2*a*, *b*). However, CaCO₃ was diagnosed only in the lake-river sediments (site 3, downstream of the river) in the form of rare cauliflower-shaped agglomerates up to 5 μ m in size (Figure 2*c*, arrow). In addition, unusual toroid-shaped microforms of CaCO₃, with internal diameter of 8–10 μ m and cross-sectional radius of about 1 μ m, were determined (Figure 2*d*, *e*). The similar toroid-shaped microforms of CaCO₃ (Figure 2*f*) we discovered in a peat stratum of the Amur Region (Far East, Russia). The peat samples were kindly provided by M.A. Klimin and A.Yu. Peskov (Far East Branch of RAS, Khabarovsk, Russia).

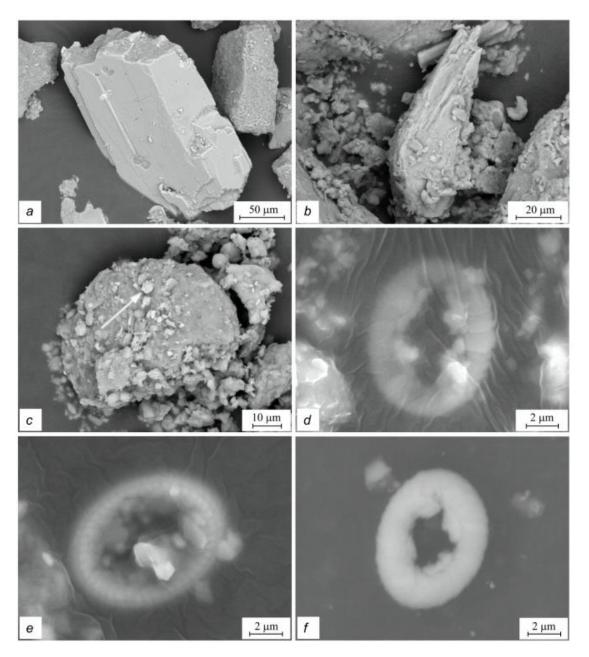


Fig. 2. SEM images of bottom sediments, the Simmi River: a, b – sphene CaTiSiO₅ grains in varying degrees of weathering; c – particles of calcium carbonate on the surface of the microaggregate (arrow); d, e – toroid-shaped microforms of CaCO₃; f – the similar microforms of CaCO₃ from a peat stratum

In contrary, the bottom sediments of the Jordan River are characterized by an extremely high content of CaCO₃ (over 40 % of CaO). It is represented by both lithogenic calcite (Figure 3*a*), and neoformations of poorly crystallized rhombohedral calcite (Figure 3*b*, *c*), and its needle-like crystals (Figure 3*d*). Porous microaggregates up to 200 μ m in size (Figure 3*e*) from micritic particles <4 μ m are also encountered. As in bottom sediment of the Simmi River toroid-shaped microforms of CaCO₃ were found (Figure 3*f*).

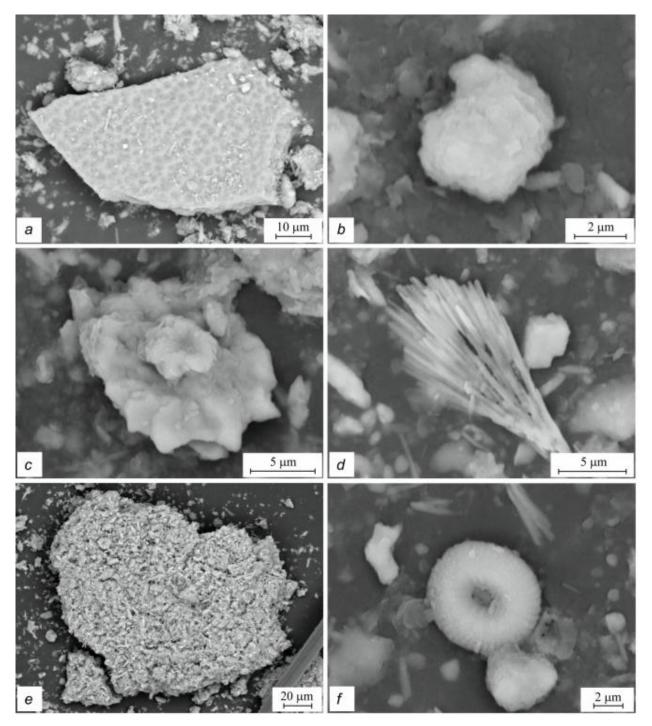


Fig. 3. SEM images of CaCO₃ formations from bottom sediments, the Jordan River: a – weathered primary calcite, b, c – poorly crystallized rhombohedral microcalcite, d – needle-like microcalcite; e – calcite microaggregate; f – toroid-shaped microform of CaCO₃

We were unable to establish the polymorphic modification of toroidal microforms due to the difficulty of their extraction from natural samples for TEM and Micro-Raman analysis. Jiang W. with collaborators (Jiang et al., 2017) solved this problem in the laboratory. They showed that toroidal, hierarchically organized architectures for vaterite can be controlled simply by adding chiral acidic amino acids (Asp and Glu). Chiral, vaterite toroidal suprastructure having a 'right-handed' spiraling morphology is induced by L-enantiomers of Asp and Glu (Figure 4a-c), whereas 'left-handed' morphology is induced by D-enantiomers, and sequentially switching between amino-acid enantiomers causes a switch in chirality. It should be noted the high stability of these suprastructures. Thus, the age of peat stratum, in which similar toroidal formations (Figure 2f) were found, is more than 6000 years. The ¹⁴C dating is according Bazarova et al. (2018).

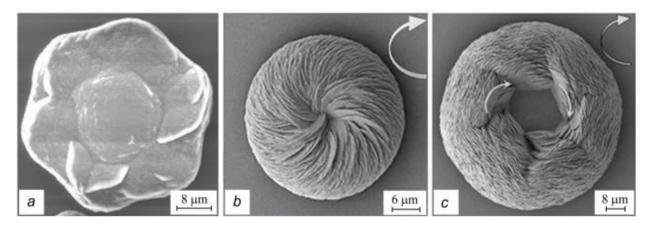


Fig. 4. SEM images of $CaCO_3$: *a* – hexagonal vaterite crystal; *b* and *c* – vaterite toroidal particles induced by chiral acidic amino acids (D-enantiomer) (Jiang et al., 2017)

In the bottom sediments of drainless lakes of the arid zone with extremely high salinity, another polymorphic modification of CaCO₃, aragonite, becomes stable. The most striking example of aragonite formations are the famous ooids of the Grand Salt Lake. Sample 5 was selected a few meters from the water's edge. But since the surface area of the lake is highly variable, we can confidently assert that it can characterize the bottom sediments of the lake. The content of CaO in the sample is high, more than 16 %. Indeed, the sample contains multiple ooids (Figure 5*a*) for the most part of regular spherical shape up to 350–400 µm in size. As shown earlier (Rey et al., 2016) these are aragonite formations. Ooids were found with both a perfectly smooth surface (Figure 5*b*) and ooids in which the processes of recrystallization and the formation of calcite began, leading to their destruction (Figure 5*c*, *d*) and a reduction of their size to 250–300 µm. The transformation is not a direct solid phase reaction, but recrystallization of calcium carbonate throught the dissolution of aragonite and growth of calcite. Porous microaggregates up to 200 µm in size (Figure 5*e*) from micritic particles <4 µm (Figure 5*f*) are also encountered. Apparently, this is the result of the destruction of aragonite ooids outside the "habitat" – the salt water of the lake.

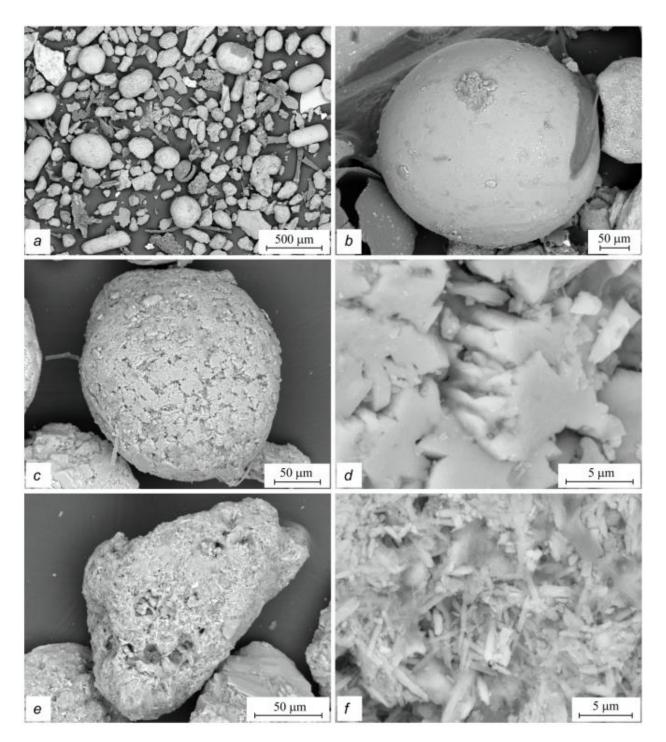


Fig. 5. SEM images of CaCO₃ from sediments of Grand Salt Lake: a - a sample as a whole with many aragonite ooids; b - a separate unweathered ooid; c and d - partially destroyed ooid; <math>e, f - calcite microaggregate

4. Conclusion

The paper presents data on manifestations of metastable modifications of calcium carbonate, vaterite and aragonite, in the river suspensions, river and lake bottom sediments of the different natural zones – the Amur and the Simmi Rivers (the Amur River basin, Khabarovsk Region, Russia); the Jordan River (Jordan); the Great Salt Lake (USA). In natural conditions vaterite, metastable polymorphic modification of calcium carbonate, can occur in cold (river ice) and extremal cold (supraglacial springs) climate as spherical particles up to $10-100 \mu m$ in size. Vaterite formation is sporadic, triggered by an as yet unknown mechanism, and can recrystallized spontaneously to calcite.

In laboratory *ex situ* vaterite forms stable toroidal suprastructures in the presence of acidic amino acids Asp and Glu. These microforms of $CaCO_3$ are characterized with internal diameter of 8–10 µm and cross-sectional radius of about 1 µm. *In situ* similar toroidal vaterite suprastructures we discovered in the river sediments of the arid and humid zone (the Jordan and the Simmi Rivers), and in the peat stratum (the Amur River basin, Khabarovsk Region). The age of peat stratum, in which these varerite microforms were found, is more than 6000 years. Apparently, its formation and stability in natural, as in laboratory conditions, are associated with the presence of low molecular amino acids in natural water.

Another polymorphic modification of $CaCO_3$, aragonite, are stable in the bottom sediments of drainless lakes of the arid zone with extremely high salinity, e.g. the famous ooids of the Grand Salt Lake. However, outside the "habitat" – the salt water of the lake – aragonite ooids are destroyed.

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Карбонат кальция в эдафических компонентах экосистем

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Аннотация. Карбонат кальция – важный компонент эдафических компонентов экосистем (почвы, речные и озерные донные отложения, взвеси). Из трех полиморфных модификаций СаСО₃ (кальцит, арагонит, ватерит) наиболее изучен кальцит. Две другие, менее стабильные и более растворимые модификации, изучены недостаточно, хотя они являются важными прекурсорами многих карбонат-образующих систем. В работе рассмотрены некоторые примеры проявления ватерита и арагонита в речном льду, торфяных отложениях, речных и озерных донных отложениях различных природных зон реки Амур, Симми (бассейн Амура, Хабаровский край, РФ) и Иордан (Иордания), Большое Соленое озеро (США). Показано, что ватерит в присутствии низкомолекулярных аминокислот может стабилизироваться в виде тороидных супраструктур ex и in situ. Эти микроформы µ-CaCO₃ имеют внутренний диаметр 8–10 мкм и радиус поперечного сечения около 1 мкм. Считается, что в природных условиях ватерит неустойчив. Однако, наши исследования показали, что его тороидные формы весьма устойчивы. Так, возраст торфяных отложений Приамурья, в которых тороидные микроформы ватерита были обнаружены, составляет более 6 тыс. лет. Другая полиморфная модификация CaCO₃, арагонит, стабилизируется при экстремальной высокой концентрации солей, как например, знаменитые арагонитовые ооиды Большого Соленого озера, формирование которых проходило на протяжении большей части голоцена. Однако на побережье озера вне зоны "обитания" (соленая вода озера) они начинают разрушаться.

Ключевые слова: карбонат кальция, кальцит, арагонит, ватерит, речные и озерные донные отложения.

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