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Original article

Physical and Chemical Characteristics of the Near-Bottom and Pore Waters, and the Bottom Sediments in the Northeastern Part of the Black Sea Shelf

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Abstract
Purpose. The investigation is purposed at studying the features of the vertical distribution of geochemical parameters and chemical profile of pore waters in bottom sediments in the coastal zone of the northeastern part of the Black Sea.
Methods and Results. 24 samples of the bottom sediments surface layer were obtained in three cruises on the R/V "Professor Vodyanitsky" in September 2018, July 2019 and August 2021. 5 samples of cores and 9 samples of water from the near-bottom layer were taken in August 2021. The profiles of vertical distribution of oxygen and hydrogen sulfide, the iron oxidized and reduced forms in pore waters, and also oxygen concentration and the degree of oxygen-saturation of the near-bottom waters were studied; the geochemical characteristics of bottom sediments in the surface layer (0–5 cm) and in the cores were also determined. It was noted that the sediments surface layer in the northeastern region was formed by a silty material, the maximum portion of which (83–97%) was recorded in the areas where large rivers of the Krasnodar region flowed into the sea. In the sediments surface layer, the C_{org} content varied within 0.2–1.9% dry mass. The vertical distribution of C_{org} in the bottom sediments cores is characterized by a tendency to decrease in its content with depth; its maximum concentrations were observed in the upper (0–4 cm) layer.
Conclusions. It is established that in the area under study, the sediments upper layer is characterized by the aerobic conditions (oxygen penetrated up to 10 mm); in the sediments, the organic matter is oxidized mainly with the participation of iron (Fe(II, III)), that leads to developing the suboxic conditions. In some areas, the redox conditions in the sediments changed from suboxic (the region of Anapa and the area between the cities of Gelendzhik and Tuapse) to anoxic (the region of Novorossiysk and Tuapse), that is conditioned by occurrence of the sulfur reduced forms in the bottom sediments layer. It has been revealed that in the northeastern part of the Black Sea, the distribution of Fe(II, III) in the pore waters of bottom sediments is inversely proportional to the distribution of pH value (the correlation coefficients are –0.68 and –0.73, respectively).

Keywords: bottom sediments, pore waters, oxygen, voltammetry, granulometric composition, organic carbon, Black Sea

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Introduction

Bottom sediments are a quasi-stationary system and are an integral indicator of the state of marine ecosystems, including their pollution [1]. It is with bottom sediments that the final stage of transformation, migration, and accumulation of various substances is primarily associated [2]. Pore waters are an integral part of bottom sediments [3, 4]. A comprehensive study of bottom sediments makes it possible to study the biogeochemical processes that determine the redox and acid-base properties of sediments, the content of mobile forms of metals, the possibility of their binding and transition to a solid form or to the water column, as well as to assess the state of the marine ecosystem as a whole and describe its mechanism of possible changes [5, 6]. This is of great scientific and practical interest.

One of the main indicators for assessing the ecological state of marine ecosystems is the presence of oxygen and its concentration in the near-bottom water layer and the upper sediment layer, as well as the content and lability of organic matter, and the sediment granulometric composition. An additional factor determining the ecological situation is hydrodynamics: the hampered water exchange and stratification of the water column limit the oxygen flux into the near-bottom water layer and bottom sediments, which leads to the development of oxygen deficiency and the emergence of ecological risk zones.

The region of the Black Sea northeastern part includes the area from the Taman Peninsula to the town of Adler. This area is characterized by intense water dynamics [7]. The authors of [8] note that coastal currents here are very variable: their velocity and direction can change significantly; countercurrents, which propagate towards the southeast in the direction opposite to the Rim Current, are regularly recorded.

The Caucasus coast of the Black Sea, due to its natural and climatic conditions, is a region where recreational and sanitary-resort activities traditionally develop. As a result, the volumes and rates of development of a narrow coastal strip are constantly growing [9]. The continuous impact of the anthropogenic factor on coastal ecosystems also contributes to the supply of additional amounts of organic matter and nutrients to them. This determines a high variation rate of the characteristics of coastal ecosystems and contributes to the intensification of sedimentation processes in them.

The pollution of the coastal zone of the Black Sea northeastern part is also significantly affected by various treatment facilities built on the coast and the pollution transport with continental runoff. The main areas of risk for the development of oxygen deficiency in the water column are the areas adjacent to the cities of Novorossiysk, Sochi, Gelendzhik, and Tuapse, as well as anchorages [10, 11].

According to [12], coarse terrigenous material is deposited in the coastal shallow part (down to depths of 20–30 m). This provides oxygen supply to the upper layer of sediments, however, as a result of resuspension and redeposition (due to shallow depths), uneven profiles of the characteristics of pore water and bottom sediments can be observed.

Insufficient attention is paid to the modern study of the bottom sediment geochemistry in the Black Sea northeastern region. In most of the available data, the granulometric composition distribution of the sediment and the organic carbon content in it are described in a generalized form. The main attention is paid to the content of various pollutants in bottom sediments, including organochlorine compounds [13], petroleum hydrocarbons [9–11, 14, 15], and heavy metals [9, 16–18]. It was determined that the most polluted areas are the exits from the Tsemess and Gelendzhik bays, the traverses of the Abrau Peninsula and the mouths of the rivers Vulan, Tuapse, Psezuapse, Shakhe, Dagomys Zapadny, Sochi, Mzymta.

At the same time, the effect of hydrological and hydrochemical characteristics of the near-bottom water layer and the geochemical characteristics of bottom sediments on the formation of the chemical composition of pore waters and redox conditions in them in the northeastern part of the Black Sea was not considered.

This work is purposed at studying the features of vertical distribution of geochemical parameters and the chemical profile of the pore waters of bottom sediments in the coastal zone of the Black Sea northeastern part using new expeditionary data.

Materials and methods

The samples of the surface layer of bottom sediments were obtained during three cruises of R/V "Professor Vodyanitsky": the 103th cruise (September 2018), 108th (July 2019), and 117th (August 2021). In total, 24 samples were taken in the surface layer of sediments (Fig. 1, *a*). The samples of cores (5 cores) and water from the bottom layer (9 samples) were also obtained during the 117th cruise.

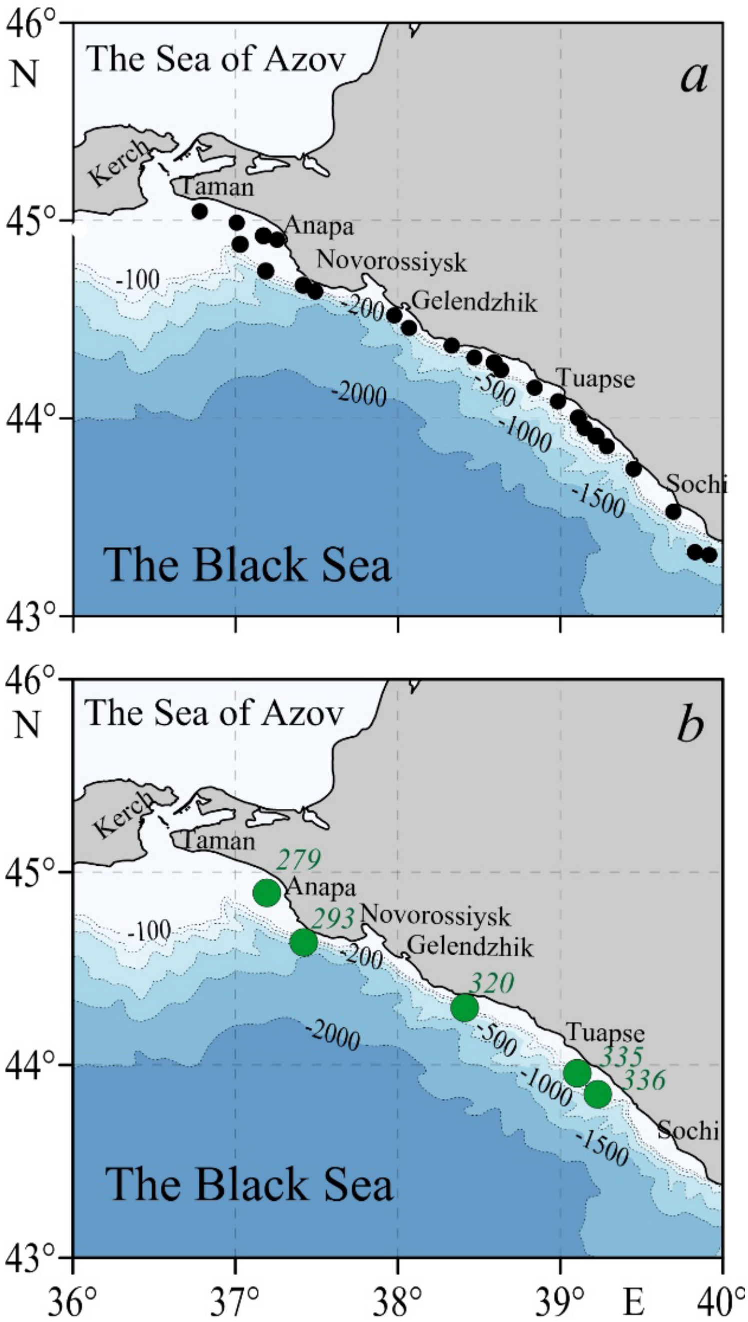
The sampling and preparation of bottom sediment samples were carried out in accordance with regulatory documents (GOST 17.1.5.01-80, ISO 5667-19:2004). The samples of bottom sediments and water from the near-bottom horizon were taken in the depth range from 30 m in the area of the Kerch pre-strait region to 90 m in the continental slope area in between Tuapse and Sochi. The samples of the near-bottom water layer were taken 10 m above the surface of bottom sediments.

The sea water sampling from near-bottom horizons for chemical analysis was carried out using bathometers. The dissolved oxygen content in water samples was determined by the Carpenter modification of volumetric Winkler titration method [19]. The technique provides results with an accuracy of l ± 0.010 ml/l (±0.4 μM). The degree of water saturation with oxygen (%) was calculated in accordance with [20]:

$$\ln C = A_1 + A_2\,(100/T) + A_3 \ln (T/100) + A_4\,(T/100) + S\,[B_1 + B_2\,(T/100) + B_3\,(T/100)^2],$$

where C is the oxygen solubility at a total pressure of 1 atm, taking into account the pressure of saturated water vapor, ml/l; $A_{(1,\,2,\,3,\,4)}$ and $B_{(1,\,2,\,3)}$ are constants ($A_1 = -173.4292$, $A_2 = 249.6339$, $A_3 = 143.3483$, $A_4 = -21.8492$, $B_1 = -0.033096$, $B_2 = 0.014259$, $B_3 = -0.0017$); T is the absolute temperature, K; S is salinity.

The samples of the upper layer of sediments (0–5 cm) were taken using the Peterson bottom grab. The columns of the bottom sediments for studying the sediment vertical structure were taken using a manual sampler in the form of an acrylic soil tube (60 mm inner diameter) with a vacuum hydraulic seal. This method of sampling provided preservation of fine structure of the surface layer of bottom sediments and the bottom water layer.



F i g. 1. Scheme of sampling stations: *a* – in the bottom water layer and in the surface layer (0–5 cm) of sediments, *b* – cores of bottom sediments (numerals indicate the station numbers)

To obtain the chemical profile of pore water, a polarographic method of analysis with a glass Au-Hg microelectrode was applied [21, 22]. An electrode saturated with silver chloride was applied as a reference electrode, and a platinum electrode was used as an auxiliary one. Profiling of bottom sediment cores was carried out with 1–10 mm vertical resolution. The main advantage of the method is the ability to analyze the composition of pore waters of bottom sediments under the conditions as close as possible to the natural ones, without sample destruction and additional sample preparation. The detection limit for oxygen was 5 µM, for hydrogen sulfide it was 0.5 µM, for Fe(II) – 10 µM, for Mn(II) – 5 µM. For all measurements, the determination error varied within 0.4–10% depending on the concentration. The concentration of Fe(III) and FeS cannot be analytically determined due to the formation of complicated set (Fe(III)) and colloidal solutions (FeS) [23]. Therefore, the paper considers their semi-quantitative distribution in microamperes (µA).

To analyze the physico-chemical characteristics, the columns were separated into 1–2 cm thick layers using a manual extruder and an acrylic ring.

The granulometric composition of bottom sediments was determined by the combined method of decantation and dispersion. The separation of the aleurite-pelitic fraction (≤ 0.05 mm) was performed by wet sieving, followed by gravimetric determination of the dry mass. The coarse-grained fractions (> 0.05 mm) were separated by a dry sieving method using standard sieves (GOST 12536-2014).

The organic carbon content (C_{org}) was determined coulometrically using AN-7529 express analyzer according to a procedure adapted for marine bottom sediments [24, 25]. The root-mean-square deviation for samples with C_{org} content < 0.5% was 0.03%, with C_{org} content > 1.5% – 0.08% [25].

The determination of pH value in bottom sediment columns was carried out using I-160MP ion meter and Hanna Instruments HI 10530 combined pH electrode.

Correlation coefficients, as well as their reliability degree, were calculated using the *Statistica* program. The reliability level of the obtained correlation coefficients chosen in the work was 95%.

Results and discussion

The surface sediment layer in the northeastern region is formed mainly by silty material (average percentage 79%) (Fig. 2, *a*). Coarse-grained gravel-sand material was distributed in shallow water (~ 30 m) in the areas of the Kerch pre-strait region and Anapa. An increased proportion of the finely dispersed clay fraction (up to 97%) was noted in the areas of the inflow of large rivers of Krasnodar Krai (Pshada, Tuapse, Sochi, Mzymta).

On average, C_{org} content in the sediment surface layer in the northeastern region was 1.0% dry weight, which, according to [1], indicates the recovery nature of bottom sediments. Nevertheless, the obtained values were almost less than half of that in the northwestern region and on the southern coast of Crimea, and slightly higher than in the Kerch pre-strait region [29, 30].

C_{org} content in the surface layer of sediments (Fig. 2, *b*) varied from 0.2–0.5% dry weight at stations near the Taman Peninsula and in the Anapa region up to 1.3–1.9% dry weight in fine-grained silty sediments in the area from Tuapse to Adler, as well as in the area of the Abrau Peninsula.

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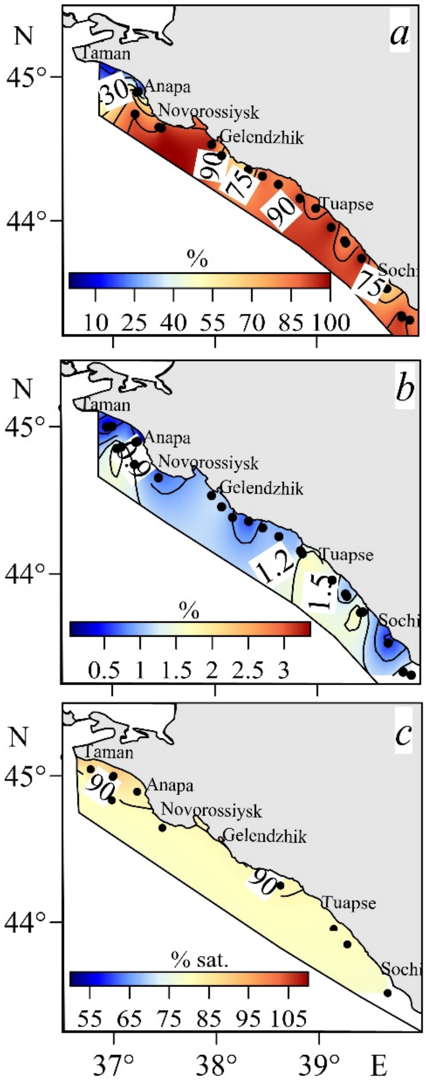
[1] Volkov, I.I., 1984. *Geochemistry of Sulfur in Ocean Sediments*. Moscow: Nauka, 272 p. (in Russian).



F i g. 2. Spatial distribution of physical and chemical characteristics of: *a* – clay fraction; *b* – C_{org} in the surface layer of sediments and *c* – the degree of oxygen saturation of the near-bottom water layer in the northeastern part of the Black Sea

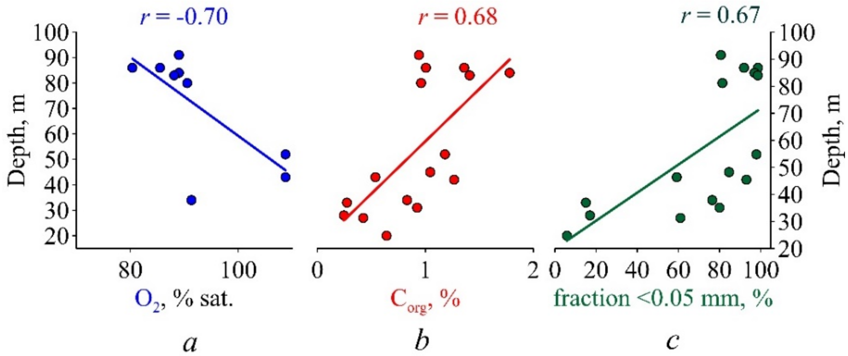
One of the causes for the localization of areas with an increased proportion of the fine-grained fraction of sediments and high C_{org} content in them can be considered the influx of organic matter and nutrients with river runoff [26]. The authors of [27] suggested that in the area of the drift of the Mzymta, Kudepsta, and Sochi rivers, the "secondary" accumulation of organic matter is associated not with its allochthonous drift, but with a significant influx of nutrients with river runoff, which stimulates the development of phytoplankton at the seaward boundary of the mixing zone [26] and is consistent with the field data obtained in 2016–2019 during the expedition research at R/V "Professor Vodyanitsky" [28]. Low C_{org} content is primarily determined by the sediment granulometric composition: in the area of the Taman Peninsula coastal zone, the fraction of clay sands with the inclusion of shell detritus prevailed; the proportion of the clay fraction did not exceed 30% on average.

According to the data obtained in this work, the oxygen content in the bottom water layer corresponded to 88–91% saturation, which is quite typical for the studied depth range. The exception was station *279* (sampling depth 34 m) with 91% oxygen saturation. This is not quite typical for this depth and indicates an active oxygen consumption in the bottom layer of waters and, probably, may be due to the oxygen consumption for the oxidation of organic matter coming with the Kerch Strait waters.

It is known that depth affects the formation of hydrochemical structure of waters and geochemical characteristics of bottom sediments [2]. An analysis of the results obtained in the work revealed that with increasing depth, the degree of water saturation with oxygen decreases (Fig. 3, *a*), the proportion of finely dispersed pelite-

[2] Rukhin, L.B., 1969. [*Fundamentals of Lithology*]. 3rd Edition. Nedra: Leningrad, 703 p. (in Russian).

aleuritic fraction (Fig. 3, *b*) and C_{org} content in the sediment increase (Fig. 3, *c*), which is consistent with the literature data. The exception is shallow water areas, in which, depending on the hydrodynamic regime, sediments of different granulometric composition can accumulate.



F i g. 3. Dependence of the distribution of: the degree of oxygen saturation in the near-bottom water layer (*a*), and the C_{org} content (*b*) and the portion of pelite-aleuritic fraction (*c*) in the sediments surface layer upon the sampling depth in the northeastern part of the Black Sea

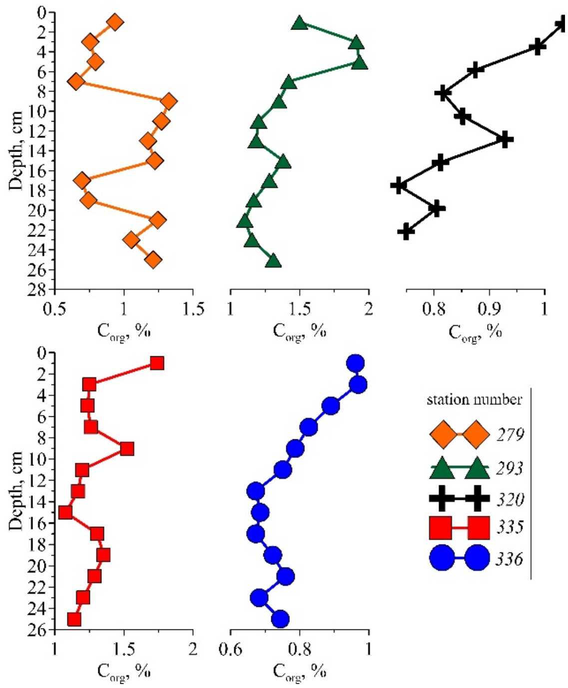
In general, the correlations identified during the work turned out to be not unique for the Black Sea ecosystem. However, the fact that they are consistent with previously obtained results for other regions [1, 29, 30] confirms the existence of common mechanisms for the functioning of the Black Sea ecosystem.

The vertical C_{org} distribution is heterogeneous, which may be due to the heterogeneous supply of organic matter. However, the maximum concentrations are observed mainly in the upper (0–4 cm) layer and there is a trend towards a decrease with depth (Fig. 4), except for station *279* and *293*. At station *279*, two maxima of the C_{org} content were noted: in the depth interval of 8–16 and 20–22 cm. This indicates a different rate of input and accumulation of organic matter, the main sources of which, apparently, are the terrigenous runoff of the Anapa River and the Sea of Azov waters coming from the Kerch Strait [31]. At station *293*, the maximum C_{org} content was noted in the 4–6 cm layer, which may indicate a decrease in the level of organic matter input in this area at the present time.

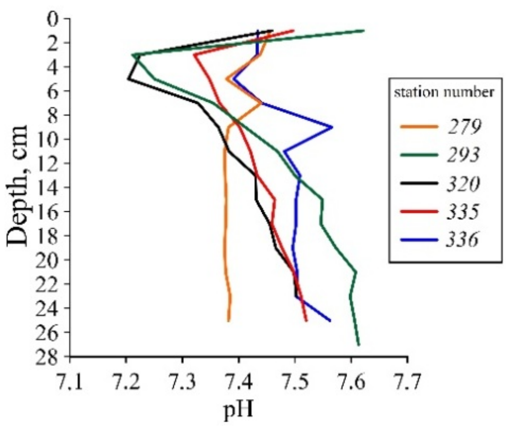
The nature of the pH value vertical distribution in the cores of bottom sediments of the northeastern part of the Black Sea is shown in Fig. 5.

Active redox processes in bottom sediments with participation of organic matter lead to a significant decrease in pH compared to the bottom layer (the bottom layer pH is 8.23–8.24).

Increased values (7.43–7.62) are observed in the 0–2 cm layer, which is explained by its saturation with water and constant contact with the bottom layer. Further, the values sharply decrease, in the 2–6 cm layer, the minimum values (7.20–7.37) are noted for all cores. With depth, the pH values steadily increase for all cores, except for station *279* (Fig. 5). The pH shift towards the alkaline environment indicates the development of anaerobic conditions as a result of accumulation of reduced forms of sulfur and ammonium ions in the bottom sediments. It was determined that the vertical pH profile is predominantly inversely proportional to the C_{org} vertical distribution in the sediment (correlation coefficient –0.6).



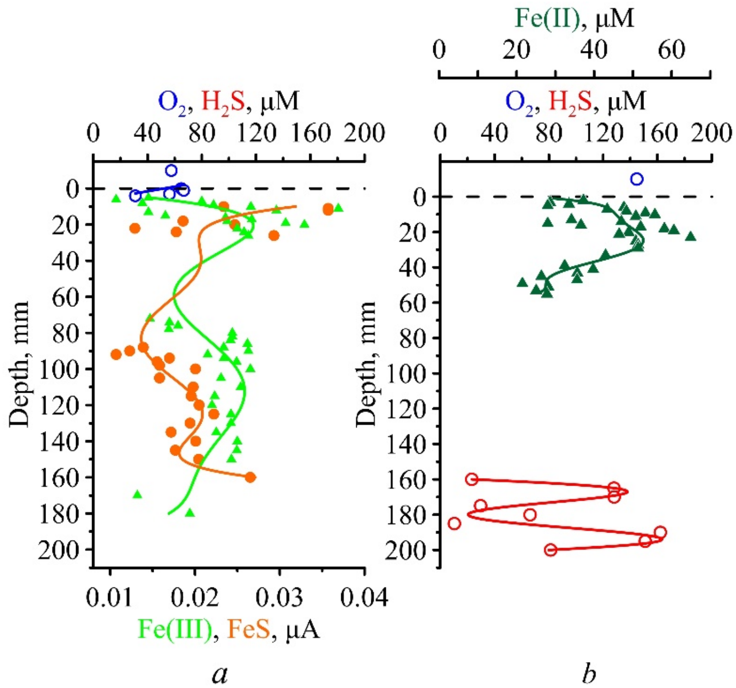
F i g. 4. Vertical profiles of C_{org} content in the bottom sediments



F i g. 5. Vertical profiles of the pH value in the bottom sediments

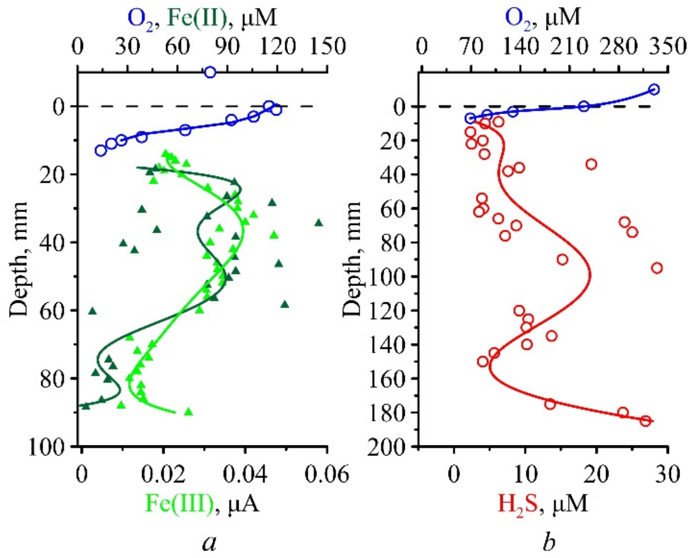
In the near-bottom water layer (sampling depth ~ 24 m) near Anapa (station *279*), the oxygen concentration corresponded to 91% saturation (254 µM) (Fig. 2, *c*). On the sediment surface, oxygen was present in an insignificant concentration (65 µM, 26% sat.) and penetrated up to 4 mm (Fig. 6, *a*). Two depth intervals with the presence of Fe(III) were noted: 5–26 and 72–180 mm (Fig. 6, *a*). From a horizon of 10 mm, iron monosulfide (FeS) appeared; its distribution along the depth was uneven. Thus, various forms of iron were the main components of the pore waters; there was no oxygen in the sediment, which led to the development of suboxic conditions in the upper layer of bottom sediments.

In the area of Novorossiysk (station *293*), in the near-bottom water layer (sampling depth ~ 74 m), the oxygen concentration reached 285 μM (89% sat.). At the same time, oxygen was not found in the surface layer of sediments. In the upper layer of pore water (0–50 mm), reduced forms of iron (Fe(II)) dominated. Its maximum concentration (50–60 μM) was noted in the 10–30 mm layer (Fig. 6, *b*). Hydrogen sulfide appeared in deeper layers (from 160 mm), its distribution was nonuniform, the average concentration was 87 μM (Fig. 6, *b*). No polarographically active compounds were observed in the depth interval of 60–160 mm. Thus, suboxic conditions were noted in the upper layer of bottom sediments, while anaerobic conditions were noted in deeper layers. Such a distribution of the chemical components of pore water is quite typical for these depths, while the iron content was significantly lower than in anthropogenically loaded areas [32].



F i g. 6. Vertical distribution of the main components of pore waters in the bottom sediments at stations *279* (*a*) and *293* (*b*) in the northeastern part of the Black Sea

In the area between Gelendzhik and Tuapse (station *320*, sampling depth ~ 81 m), despite the saturation of the near-bottom water layer with oxygen (91% sat.), in the layer 10 cm above the sediment, an oxygen deficiency was noted – 25% sat. At the same time, oxygen penetrated into the bottom sediments up to 13 mm (Fig. 7, *a*). Oxidation of organic matter occurred presumably with the participation of iron (Fe(II, III)) (Fig. 7, *a*; Table) [33]. The distribution of Fe(II) was inhomogeneous, its concentration varied within 5–145 μM with an average value of 63 μM. Starting from 92 mm, no polarographically active components were recorded [21, 34].



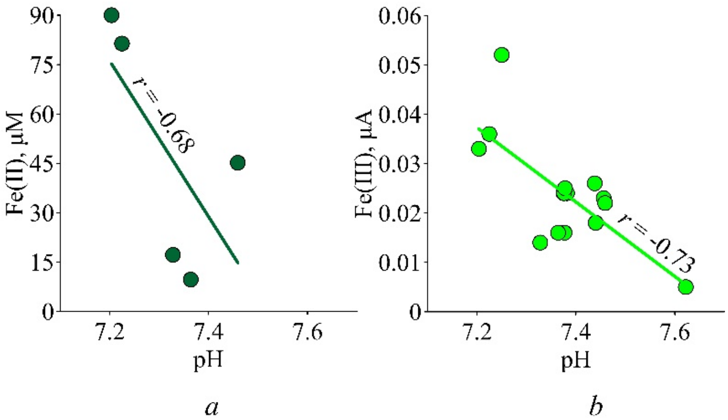
F i g. 7. Vertical distribution of the main components of pore waters in the bottom sediments at stations *320* (*a*) and *335* (*b*) in the northeastern part of the Black Sea

Sequence of the reactions of organic matter oxidation in bottom sediments

Name of the process	Scheme of the reaction
Aerobic oxidation	$C_{106}H_{175}O_{42}N_{16}P + 150\ O_2 \rightarrow 106\ CO_2 + 16\ HNO_3 + H_3PO_4 + 78\ H_2O$
Denitrification	$C_{106}H_{175}O_{42}N_{16}P + 104\ HNO_3 \rightarrow 106\ CO_2 + 60\ N_2 + H_3PO_4 + 138\ H_2O$
Manganese reduction	$C_{106}H_{175}O_{42}N_{16}P + 260\ MnO_2 + 174\ H_2O \rightarrow 106\ CO_2 + 8\ N_2 + H_3PO_4 + 260\ Mn(OH)_2$
Iron reduction	$C_{106}H_{175}O_{42}N_{16}P + 236\ Fe_2O_3 + 410\ H_2O \rightarrow 106\ CO_2 + 16\ NH_3 + H_3PO_4 + 472\ Fe(OH)_2$
Sulfate reduction	$C_{106}H_{175}O_{42}N_{16}P + 59\ H_2SO_4 \rightarrow 106\ CO_2 + 16\ NH_3 + H_3PO_4 + 59\ H_2S + 62\ H_2O$
Methanogenesis	$C_{106}H_{175}O_{42}N_{16}P + 59\ H_2O \rightarrow 47\ CO_2 + 59\ CH_4 + 16\ NH_3 + H_3PO_4$

Near Tuapse (station *335*) the sampling depth was 83 m. The oxygen concentration in the bottom layer (73 m) corresponded to 88% saturation. Oxygen was also present in the upper layer of sediments and penetrated to a horizon of 7 mm (Fig. 7, *b*), its average concentration in the surface layer of the sediment was 231 μM (72% sat.). However, hydrogen sulfide was the main component of pore water (Fig. 7, *b*). Its concentration varied within 3–76 μM with an average value of 19 μM. In this area, aerobic conditions were observed in the upper 6 mm layer, which was probably due to the influx of waters enriched with oxygen due to their good ventilation [7, 35]; the anaerobic conditions that developed as a result of sulfate reduction are recorded below (Table). The absence of other polarographically active components may indicate a “fresh” source of oxygen for the surface layer of sediments and intense organic matter oxidation.

Relationships between the vertical distribution of iron (Fe(II, III)) in the pore waters of bottom sediments and the pH value were determined (Fig. 8). An inverse dependence of Fe(II, III) content on the sediment pH was noted, the correlation was –0.68 and –0.73, respectively. This demonstrates that the presence of oxygen and other redox components, in particular, dissolved iron in the sediment, determines the formation of redox conditions in the sediment, which is further confirmed by the data of physicochemical characteristics. The obtained results are consistent with the ones of [36].



F i g. 8. Relationship between the Fe(II) (*a*) and Fe(III) (*b*) content in pore water, and the pH value in the bottom sediment cores

Conclusion

The features of the geochemical composition of bottom sediments and the chemical composition of pore water in the coastal areas of the Black Sea northeastern part were considered. New expeditionary data were obtained, and the features of the vertical and spatial distribution of the chemical profile of pore waters were studied using an adapted method of polarographic analysis.

It was noted that the sediment surface layer in this area was formed mainly by clay material, the maximum concentrations of which were recorded in the areas where large rivers of Krasnodar Krai (Pshada, Tuapse, Sochi, Mzymta) flow into the sea. Gravel-sand material accumulated in shallow water in the areas of the Kerch pre-strait region and Anapa.

C_{org} content in the sediment surface layer varied from 0.2% dry weight near the Taman Peninsula up to 1.9% dry weight in the Adler region, which is on average half as high as in the northwestern region of the Crimean shelf and at the southern coast of Crimea. The maximum C_{org} concentrations were characteristic of the upper (0–4 cm) layer of bottom sediments; its content mainly decreased with depth.

The obtained data on the vertical distribution of the main components of pore water made it possible to identify the areas where the formation of bottom sediments occurs under aerobic, suboxic, and anaerobic conditions. On the whole, it can be concluded that aerobic conditions prevail at most coastal stations. At the same time, in the deeper layers, the conditions in the sediment changed from suboxic (Anapa region and the area between Gelendzhik and Tuapse) to anaerobic (Novorossiysk and Tuapse regions).

It was determined that Fe(II, III) content in the pore water was inversely proportional to the pH value of the sediment.

It is noted that in the northeastern part of the Black Sea, as a result of water ventilation oxygen penetrated into the upper sediment layer, and the main factor determining redox conditions is water dynamics. However, for the inflow areas of the North Caucasus large rivers, the organic matter accumulation in bottom sediments is typical due to the influx of a significant amount of suspended matter. This led to the formation of finely dispersed sediments, which hindered oxygen supply to the underlying sediment layers.

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Natalia A. Orekhova – general scientific supervision of the research, data preparation; participation in the discussion of the paper materials; editing and supplementing the text of the paper, advisory assistance

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