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MARINE CHEMISTRY

Fluorescence of Dissolved Organic Matter as a Marker for Distribution of Desalinated Waters in the Kara Sea and Bays of Novaya Zemlya Archipelago

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Abstract—The optical properties and distribution of dissolved organic matter in the surface waters of the Kara Sea and bays of Novaya Zemlya archipelago were studied during the 63th cruise of the R/V *Akademik Mstislav Keldysh*. The fluorescence of dissolved organic matter has been studied over wide excitation (230–550 nm) and emission (240–650 nm) wavelength ranges. Based on the results of fluorescence measurements, we propose a simple technique for estimating the relative content of humic compounds entering the Kara Sea shelf region with Ob and Yenisei river runoff. We have found that the blue shift parameters of the DOM fluorescence are $\Delta_{270-310} = 28 \pm 2$ nm and $\Delta_{355-310} = 29 \pm 2$ nm. The highest contents of humic compounds in surface waters were measured on the transect across the desalinated layer of the Kara Sea, near the continental slope on the transect along the St. Anna Trough, and in the area of Sedova, Oga and Tsivol'ki bays. Traces of labile terrigenous organic matter were found in the region of the Voronin Trough, in the bays of the Severny Island of Novaya Zemlya, as well as in some freshwater reservoirs and ice samples of the archipelago. We established a conservative distribution of dissolved organic matter, whose content in water varied from 1.25 to 8.55 mg/L.

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1. INTRODUCTION

Dissolved organic matter (DOM) of natural origin is one of the most important components of marine ecosystems and the carbon cycle [8]. In the Arctic Basin, the DOM pool was estimated as 9×10^{15} g [20]. The annual supply of C_{org} from large Siberian rivers to the Arctic shelf is roughly 3×10^{13} g [7, 21]. The Kara Sea receives the largest river runoff over the entire Arctic Basin, which is about 45% of the total runoff of the Arctic region [11, 22].

Currently, optical methods are widely used in studying the origin and dynamics of DOM [12, 18, 19, 22, 25]. They and based on the ability of the colored fraction of DOM, in particular, fluorescent DOM, to absorb the ultraviolet (UV) light and to emit UV and visible light. A typical spectrum of UV-excited fluorescence of DOM in natural water involves two superimposed bands. The first one represents a peak at 300–350 nm and is due to emission of proteins, aromatic amino acids, and phenolic compounds, while the second band is due to fluorescence of humic substances (HS) in the blue spectral range [16, 23]. The composition and ratios of DOM components are liable to differ from one aquatic area to another. That is why optical markers are successful in identifying natural waters [3, 6] and investigating the degradation processes of DOM [17]. A high HS content is typical of Siberian rivers, including the Ob and Yenisei [2, 15]. This makes it possible to use the peculiar features of riverine DOM fluorescence described in [2, 14] to reveal the occurrence of terrigenous DOM in the Arctic Basin. For example, traces of terrigenous DOM have been discovered in the Kara Sea north of 76° N [2].

The goal of the present study is to examine the distribution and fluorescence of DOM in the upper water layer of the Kara Sea and the bays of Novaya Zemlya. We investigated the spreading of desalinated waters in the Kara Sea as well as in the Voronin and St. Anna troughs and revealed the areas of predominance of labile autochthonous DOM in the surface waters.

2. MATERIALS AND METHODS

The water samples were collected during cruise 63 of the R/V *Akademik Mstislav Keldysh* in August–October 2015 at 28 stations in the Kara Sea and bays of Novaya Zemlya. Oga, Tsivol'ki, and Sedov bays are located on the eastern shore of Severny Island of the archipelago, while Stepovoy and Abrosimov bays are situated on Yuzhny Island (Fig. 1). Ten samples of river, lake, and ice water were collected to study the

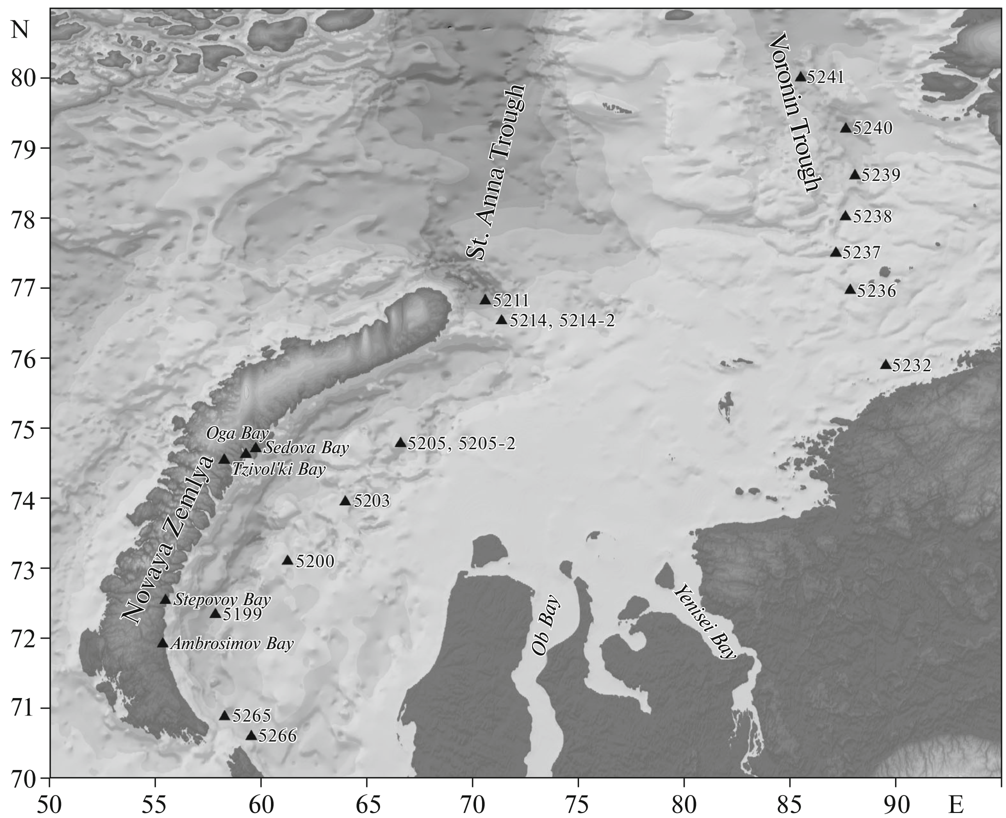

Fig. 1. Stations of water sampling for content and optical properties of DOM (August 30–October 6, 2015).

optical properties of fresh waters entering the Kara Sea from Novaya Zemlya.

Water samples were filtered through precombusted at 430°C Whatman GF/F filters with an average pore size 0.6–0.7 μm. The filtrate for recording the absorption spectra was placed into glass vials 22 mL in volume and stored at 2–3°C for subsequent analysis under stationary conditions. For the dissolved organic carbon (DOC) measurements, the filtrate was additionally acidified with hydrochloric acid up to pH 2. Fluorescence of water was measured on board immediately after filtration without additional conservation of samples.

The DOC content was determined by high-temperature combustion with a Shimadzu analyzer TOC-$V_{CPH/CPN}$. Its instrumental error is 1%. The reproducibility of the results was ±5%.

The fluorescence spectra were recorded at indoor temperature using a cell 1 cm in length and a Lumex Fluorat-02-PANORAMA spectrofluorometer. Considering that the spectral response of natural DOM depends on the excitation wavelength [3, 23], the spectra were recorded from 240 to 650 nm with a 1-nm increment, and the excitation wavelengths from 230 to 650 nm with a 5-nm increment. The 2D fluorescence spectra, known also as excitation emission matrices (EEMs), are widely used to identify fluorophores of natural DOM [9, 18]. The wavelength accuracy of excitation and emission monochromators is ±1 nm, and their spectral resolution is 15 nm.

A Unico spectrophotometer was used to record the **absorption spectra**. It was performed with respect to distilled water in the 1-cm cell over the spectral range 200–900 nm with an increment of 1 nm. The spectral resolution was 2 nm.

The quantum yield of fluorescence (QYF) was determined for each excitation wavelength by referencing to the fluorescence of quinine sulfate fluorophore [5, 23], the QYF of which is 0.55 [23].

3. RESULTS AND DISCUSSION

Station 5205, located on a transect across a lens of desalinated waters along the eastern coast of Novaya Zemlya, was chosen as the reference site, distinguished by minimal salinity (16.4 psu). Figure 2a shows the fluorescence spectra of water from this station at different excitation wavelengths, (Fig. 2b) the 2D fluorescence spectrum, and (Fig. 2c) the wavelength dependence of QYF. There was no fluorescence of proteins or aromatic amino acids in the short-wavelength spectral range from 310 to 380 nm, but fluorescence of HS within the range 400–550 nm was quite intense (Figs. 2a and 2b). This example shows that the maximum of HS fluorescence depends on the excitation wavelength: the fluorescence band maximum shifts towards shorter wavelengths when the excitation wavelength increases from 270 to 310 nm. A further increase in the excitation wavelength results in a shift of the fluorescence maximum towards longer wavelengths. This peculiarity of fluorescence differentiates the colored fraction of natural DOM from other organic luminophores. The numerical characteristic of this effect, namely, the parameters of the blue shift of fluorescence, is a distinctive feature of natural DOMs of different origin [3, 6]. Table 1 gives the parameters of the blue shift and fluorescence band maxima of the HS transported with river runoff to the Kara Sea shelf. Similar to the DOM spectra of Ob and Yenisei fresh waters given in [13], the 2D spectrum of water from station 5205 (Fig. 2b) reveals the presence of fluorophores *A* and *C* according to Cobble's classification [16]. The latter provides additional evidence of abundance of riverine HS in the water samples. The QYF depends on the excitation wavelength and varies from 0.5 to 2.3%.

It is shown in [4] that melting of the Novaya Zemlya ice cover may contribute to desalination of surface waters of the coastal zone of Novaya Zemlya in addition to westward propagation of Ob and Yenisei waters in the Kara Sea Basin. To assess the applicability of fluorescence techniques to identify waters near the eastern coastal zone of Novaya Zemlya, we investigated the land water basins of this archipelago. Figure 3 shows the typical 2D fluorescence spectra of ice and water samples, collected on the coasts of Oga, Tsivol'ki, Stepovoy, and Abrosimov bays. Fluorophores *A* and *C*, indicating the presence of terrigenous HS, were absent in the spectra of riverine and melt waters (Figs. 3a–3d). In contrast, the same fluorophores were well expressed in the spectra of riverine and lake waters on the coasts of Stepovoy and Abrosimov bays (Figs. 3e–3h). Thus, the 2D fluorescence spectra provide a graphical evidence of the absence of HS in the samples of water and ice from the Severny Island and growth of the HS fraction in DOM from streams and lakes of the Yuzhny Island. Herein the fluorescence band of HS from Yuzhny Island is slightly blue-shifted (Table 1). This may be an indication of lower degree of humification of organic matter and/or the presence in water of autochthonous colored DOM characterized by an absorption maximum at 290–310 nm and fluorescence in the range 370–410 nm (fluorophore *M*) [16]. In addition, most samples were found to contain tryptophan and/or proteins containing this amino acid [16], as evidenced by peak of fluorophore *T* (Fig. 3).

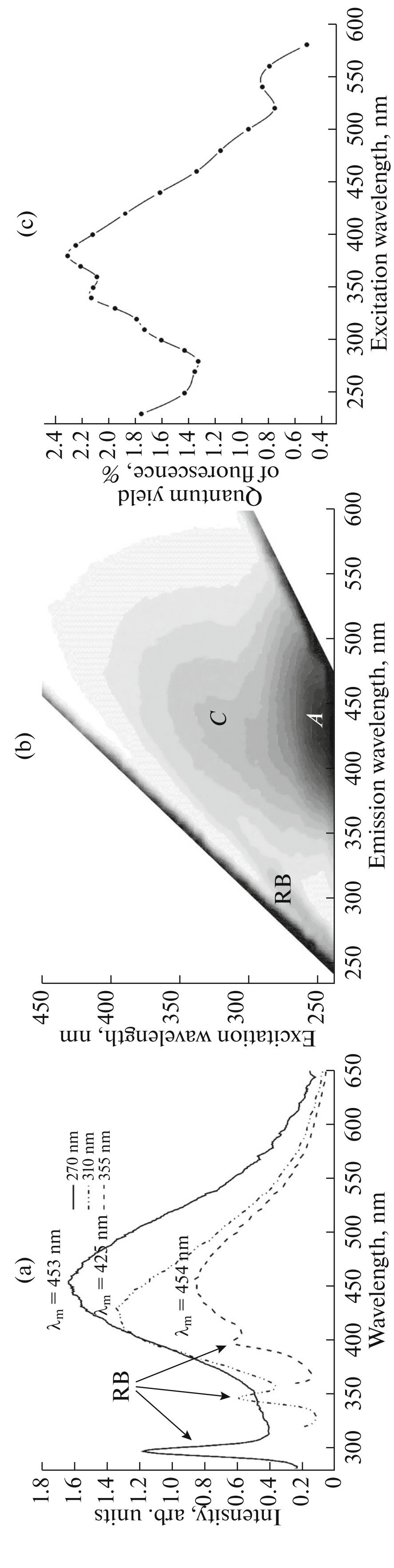

Fig. 2. Properties of DOM fluorescence at station 5205: (a) fluorescence spectra excited at 270, 310, and 355 nm; (b) 2D fluorescence spectrum; (c) dependence of quantum yield of fluorescence on excitation wavelength. RB is Raman scattering band of water molecules. *A* and *C* are fluorophores according to classification in [16].

Table 1. Position of maximum of fluorescence band of humic substances and magnitude of blue shift of emission spectra of desalinated layer of Kara Sea (Ob and Yenisei river waters) and Yuzhny Island of the Novaya Zemlya archipelago. Absolute error in determining position of fluorescence maximum is ±1 nm; same error for blue shift of fluorescence is ±2 nm

Samples	Position of maximum of fluorescence band of HS, nm			Blue shift, nm	
	λ_{ex} = 270 nm	λ_{ex} = 310 nm	λ_{ex} = 355 nm	$\Delta_{270-310}$	$\Delta_{355-310}$
Station 5205	453	425	454	28	29
Stream, Abrosimov Bay	449	422	453	27	31

In order to assess the distribution of Ob and Yenisei river waters over the Kara Sea Basin, we calculated the ratios of integral intensities of HS fluorescence to the one at the reference station 5205 (I/I_{5205}). To minimize the influence of fluorescence of proteins and the Raman peak, we chose 360–585 nm as the integration range and 310 nm as excitation wavelength. This approach, however, is applicable if the HS spectrum is

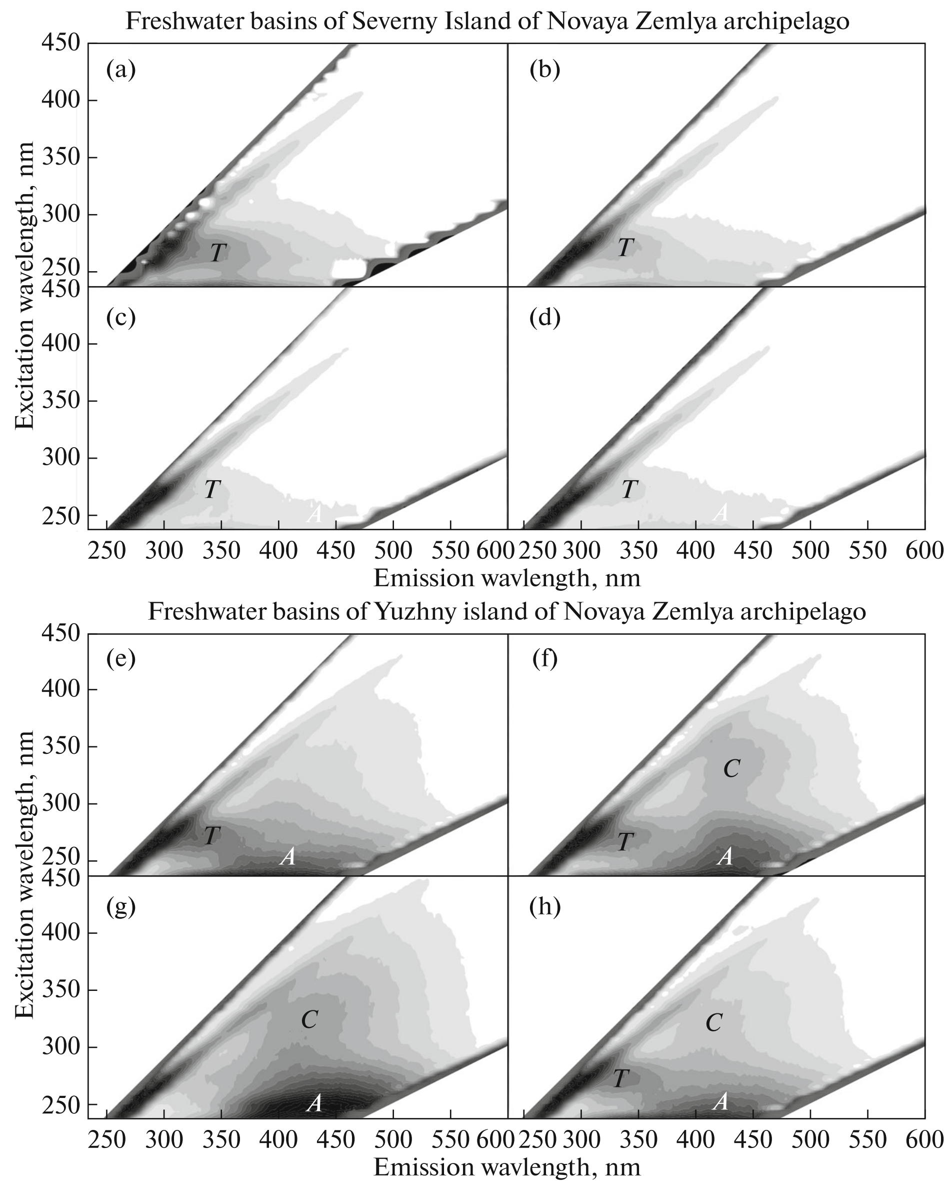

Fig. 3. Typical 2D spectra of DOM fluorescence of freshwater basins of Novaya Zemlya archipelago: melt ice (a), riverine water of Oga Bay (b); melt ice (c), riverine water of Tsivol'ki Bay (d); samples of surface (e) and bottom (f) waters from lake near Stepovoy Bay; stream water (g) and lake water (h) near Abrosimov Bay. *A*, *C*, *T* designate fluorophores according to classification in [16].

not affected by fluorescence of autochthonous organic matter (fluorophore *M*). For the studied samples the broadening or displacement of the HS fluorescence band in the range from 370 to 410 nm were not observed. Table 2 shows the salinity and DOC content, as well as the results of calculation of I/I_{5205} ratios. The DOC content in samples varied from 1.25 to 8.55 mg/L (Table 2, Fig. 4a). As this takes place, the conservative distribution of DOC is observed, which follows from the linear dependence of DOC on salinity. Our results agree with the 1997, 1999, and 2000 data reviewed in [13], and with the results of feild observations carried out in 2007 and summarized in [1]. The dependence of the fluorescence intensity on salinity obeys an inverse linear function ($R^2 = 0.90$) and is shown in Fig. 4b.

The highest HS content was found on the transect across the lens (stations 5205 and 5203), close to the continental slope on the transect along the St. Anna Trough (stations 5211 and 5214), and in the area of Sedov, Oga, and Tsivol'ki bays. In 21 days, station 5214-2 appeared beyond the influence of fresh runoff, and the effect of the latter at station 5205-2 considerably decreased: the HS content was 1.6 times lower, and salinity increased up to 23.1 psu. These changes may be due both to the displacement of the upper desalinated layer because of wind forcing and to changes in the local hydrology as a whole. The lack of fluorescing HS in fresh water samples (Figs. 3a–3d) along with an intense fluorescence within the 400–550 nm range in the waters of the bays allow us to state with certainty that Ob and Yenisei river waters flow in the eastern coastal zone of Severny Island of the Novaya Zemlya archipelago. Desalination of the surface layer in Tsivol'ki Bay (station 5254) is substantially contributed by melt water from the Serp i Molot glacier. This follows from the fact that HS content was twice as low as water of the central part of the basin with the same salinity of ~24 psu (Fig. 4b).

Marine waters of Stepovoy and Abrosimov bays were desalinated insignificantly (salinity ranged from 28.1 to 32.5 psu). The influence of Ob and Yenisei river waters is not evident in this region, since fresh waters entering the bays from the Island contain soil-related HS. Nevertheless, a comparative analysis of the fluorescence intensity revealed that the content of HS fluorophores in water basins of Yuzhny Island is about five times as low as in waters of Siberian rivers. This fact and the dependence given in Fig. 4 point to dominance in the bays of HS supplied by Ob and Yenisei runoff.

The concentration of HS at station 5236 on the eastern transect was roughly twice as low as at reference station 5205; and salinity was 27 psu. A gradual decrease in HS content in the surface waters (Table 2) occurred offshore on the transect along the Voronin Trough at stations 5236–5241. The DOC content also decreased from 2.73 to 1.67 mg/L. The jump in DOC content at station 5238 up to 3.26 mg/L could be due to the presence of labile autochthonous organic matter. This is evidenced by intense fluorescence in the spectral range from 310 to 380 nm, excited at 270 nm, which is characteristic of aromatic amino acids. Traces of autochthonous organic matter have been also found at neighboring stations 5237 and 5241.

Table 2. Relative intensity of fluorescence of humic substances and content of dissolved organic carbon in surface waters of Kara Sea and bays of Novaya Zemlya

Station	Salinity, psu	I/I_{5205}	DOC, mg/L
5199	31.7	0.138	4.19
5200	32.0	0.089	–
5200-2	31.7	0.090	2.39
5203	23.8	0.604	–
5205	16.6	1.000	6.12
5205-2	23.1	0.627	3.04
St. Anna Trough			
5211	24.7	0.597	–
5214	28.2	0.818	8.55
5214-2	33.8	0.065	1.25
5265	30.4	0.280	2.67
5266	31.6	0.154	–
Vil'kitskiy Strait	–	0.193	–
Voronin Bay			
5232	31.4	0.215	1.68
5236	27.0	0.491	2.73
5237	26.1	0.351	2.31
5238	26.1	0.397	3.26
5339	26.7	0.305	2.05
5240	27.4	0.283	1.89
5241	29.1	0.153	1.67
Sedova Bay			
5243	23.7	0.514	3.01
Oga Bay			
5247	21.6	0.502	1.75
5248	23.2	0.517	2.52
Tsivol'ki Bay			
5251	23.9	0.478	2.84
5254	23.9	0.266	1.92
Stepovoy Bay			
5257	28.1	0.283	–
5258	26.8	0.279	1.95
5260	26.9	0.272	–
Abrosimov Bay			
5263	32.5	0.092	–

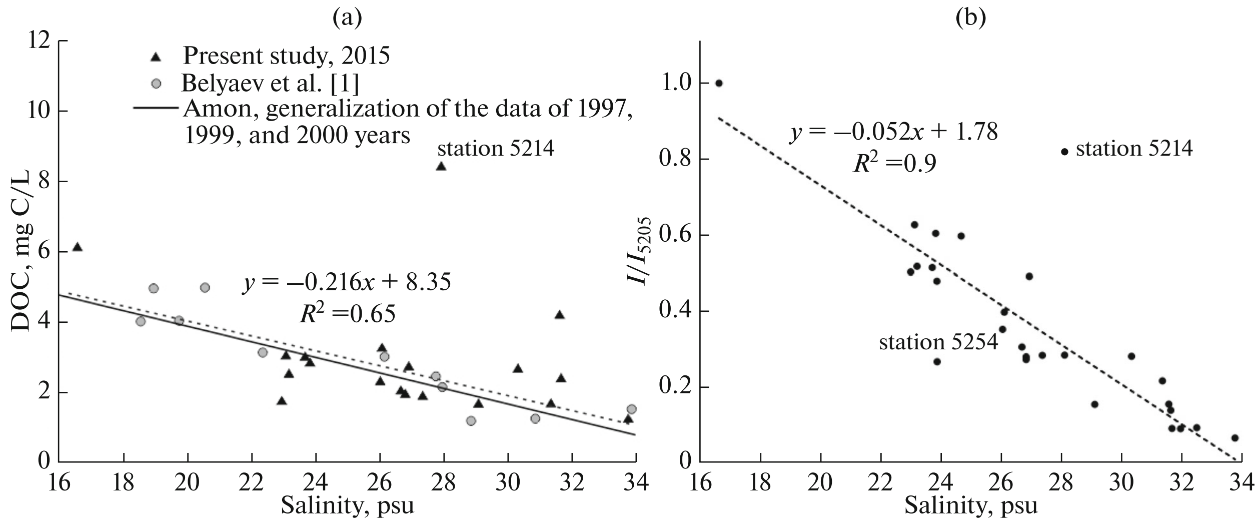

Fig. 4. (a) Dependence of DOC content on salinity in surface layer. Our data are supplemented with results of field measurements conducted in 2007 [1] and dependence $y = -0.221x + 8.326$ representing conservative DOC distribution relative to salinity data obtained in 1997, 1999, and 2000 [13]; (b) dependence of relative integral intensity of fluorescence of HS on salinity. Dashed lines approximate dependences of DOC and integral fluorescence on salinity.

The high HS and DOC contents against a background of salinity of 28.2 psu were found in waters of the slope front zone on the transect along the St. Anna Trough at station 5214 (Table 2 and Fig. 4). It is known that the propagation of desalinated water in the Kara Sea may last several years [10]. This allows us to assume that HS reached station 5214 with river runoff during preceding years and that the higher HS and DOC contents in seawater are due to displacement of 60–90% of DOM [24] during ice formation. It is more likely, however, that such a deviation of the results at station 5214 from the others was caused by nonuniformity of the water structure in the front zone area where salinity of surface waters varied from 19.5 to 33.8 psu.

5. CONCLUSIONS

The DOC content in our samples varies from 1.25 to 8.55 mg/L. We have revealed the conservative character of DOC distribution. The dominant component of colored DOM in the Kara Sea Basin in August–September 2015 was demonstrated to be terrigenous HS. It has been established that terrigenous DOM propagated up to 80° N on the transect along the Voronin Trough. The higher DOC content at the station 5238 is related to the presence of labile autochthonous organic matter in water samples. In the areas of the St. Anna Trough, the Novaya Zemlya Depression, and the desalinated lens, the content of autochthonous DOM is an order of magnitude lower compared to station 5238 and increases in the freshwater basins and the bays of Novaya Zemlya. The blue shift parameters of fluorescence of the desalinated surface waters of the Kara Sea are $\Delta_{270-310} = 28 \pm 2$ nm and $\Delta_{355-310} = 29 \pm 2$ nm. The specific features of DOM fluorescence allowed us to use the wavelength range 360–585 nm at excitation wavelength 310 nm to calculate the integral intensities of HS fluorescence. We have established a linear dependence of HS fluorescence on salinity in the case of conservative DOC distribution.

Based on the presence/absence of fluorophores *A* and *C* in the samples from freshwater basins of Novaya Zemlya, we have established, first, the lack of HS in waters entering the Kara Sea Basin due to melting of Novaya Zemlya glaciers, second, the increase in the HS content in freshwater basins of Yuzhny Island, which might be explained by more active processes of soil formation under warmer climate conditions in the absence of ice cover, typical for the Severny Island of the Novaya Zemlya archipelago. The data on HS fluorescence in Oga, Tsivol'ki, and Sedova bays unambiguously corroborate the inflow of fresh waters from the Ob and Yenisei into these bays. The westward propagation of desalinated waters, probably, took place in 2015 as it was previously observed in 2007. The salinity, however, did not drop below 23.2 psu in the eastern coastal zone of Novaya Zemlya (the minimum salinity was 16 psu in 2007).

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