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Article

Mineralogy of Particulate Suspended Matter of the Severnaya Dvina River (White Sea, Russia)

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Abstract: This paper seeks to generalize the data obtained over 4 years of investigation of the suspended sediment mineral composition in Severnaya Dvina River. The sampling of the river water to isolate suspended particulate matter (SPM) using the method of sedimentation from large water volumes (200–800 L) was carried out at two points of the delta with different hydrological regimes every month for four years. SPM samples weighing 1 g and more allowed us to obtain and preserve for different analytical procedures the grain size fractions from 1.0–0.5 to <0.001 mm (from sands to pelit). The analyses of fractions revealed a sharp prevalence of pelitic fractions (<0.01 mm) (near 90% on average), while the share of silt was 4%–5%. Coarse fractions were found in the SPM of the main stream of the river but were absent in the samples taken at the point near the river–sea boundary. The determinations of clastic, clay, and some other minerals using the method of X-ray diffraction analysis have shown that in the group of clastic minerals, quartz and plagioclase prevail. Among the clay minerals, smectite and illite were present in high quantities, and chlorite and kaolinite were in lower quantitative. The distribution of minerals in the grain size fractions showed that the sum of clastic minerals reached its highest content of up to 84% in silt fractions (0.05–0.01 mm), while the sum of clay minerals in this fraction was minimal (about 15%). Investigations of seasonal variations of clastic and clay minerals during the whole period showed that the contents of minerals in the SPM of the Severnaya Dvina did not change much over the year. As a result of this work, the following trend was established on the behavior of all kinds of minerals: during the periods of high water in spring and autumn, a slightly increased quantity of clastic minerals was detected in comparison to winter and summer, while the variations in the quantities of clay minerals were insignificant.

Keywords: Severnaya Dvina River; suspended particulate matter; mineral composition; granulometrical properties; seasonal distribution

Citation: Gordeev, V.V.; Dara, O.M.; Filippov, A.S.; Belorukov, S.K.; Lokhov, A.S.; Kotova, E.I.; Kochenkova, A.I. Mineralogy of Particulate Suspended Matter of the Severnaya Dvina River (White Sea, Russia). *Minerals* **2022**, *12*, 1600. https://doi.org/10.3390/min12121600

Academic Editor: Georgia Pe-Piper

Received: 23 October 2022
Accepted: 10 December 2022
Published: 12 December 2022

1. Introduction

The river discharge is one of the most important sources of delivery of solid sedimentary material of continental origin into the seas and oceans. Almost half a century ago, the outstanding marine geologist and academician A.P. Lisitzin showed [1,2] that of the total volume of terrigenous material delivery to the World Ocean, the share of rivers made up 73.1% (or 18.5 $\times$ 10^6 t/y), while the contribution of other factors was much lower, i.e., ice discharge—5.9%, discharge of dissolved substances—12.6%, aeolian material—6.3%, and abrasion of shores—2%.

The Hydrometeosurvey Monitoring System of the former Soviet Union was the greatest in the world at the time. One of its main priorities was to observe water and suspended matter discharges and their chemical composition in practically all large rivers and the majority of medium and small rivers. Significant attention was paid to studying the grain size and mineral and chemical composition of suspended particulate matter (SPM) in the USSR's rivers. Already in the 1930s–1940s, several publications [3–5] addressed seasonal variations of SPM concentrations and their grain size composition.

Delivery of about 36,000 km^3/y of river water and near 19×10^9 t/y of solid terrigenous sedimentary material into the World Ocean [6] exerts a fundamental impact on the processes of sedimentation in the ocean that have attracted the attention of many marine geologists and geochemists. The assessments of global volumes of SPM riverine discharge fluctuate in a wide range from 12.7×10^9 t/y [7] to 51.1×10^9 t/y [8]. Recent and probably the most accurate data on the global fluvial discharge of sediments presented in a fundamental monograph [6] cites the volume at 19.1×10^9 t/y, which practically coincides with earlier assessments by J.N. Holeman [9] and A.P. Lisitzin [2].

Results of the studies of suspended matter chemical composition in rivers are presented extensively in a number of scientific publications. One of the first among them was the work by F.W. Clarke [10] with the first data on the composition of suspended sediments in the Mississippi and the Nile. The first estimations of average global contents of the main petrogenic elements in riverine SPM were published in [11]. The estimations of average global contents of several dozen major and trace elements in SPM in more than 100 rivers across the world appeared in a paper [12] and almost simultaneously in the work of French authors [13]. As of today, the most complete results of 72 chemical element determinations in SPM of about 160 world rivers were summarized in a monograph [14]. A generalizing paper [15] prepared by a group French specialists, who used the ICP-MS determinations method to assess the concentrations of elements in river SPM, states that the data obtained by the authors are the most reliable to date. In our own work [16], significant information was presented on the most characteristic concentrations of major and trace elements in dissolved and suspended forms in river and ocean waters, and the geochemical interaction between freshwater (riverine) and marine environments was characterized.

Publications with the results of mineralogical investigations in river SPM are less numerous. Still, in the late 1960s, interrelations were established between clastic and clay minerals and grain size composition of the Amazon River SPM [17]. A. Ivanova and G. Konovalov [18] made the determinations of a group of clastic and clay minerals in SPM of 14 rivers of the former USSR. The SPM samples from nine rivers of the Black, Azov, and Caspian seas were collected in the spring of 1976, and mineralogical data were published in [19].

The papers [20,21] considered clay minerals in river SPM. The mineral composition of SPM and its input to the ocean from the rivers of India were investigated in detail in [22]. Significant contributions to our knowledge of river SPM mineralogy were made through publications with the results of a large international project under the aegis of SCOPE/UNEP "Transport of Carbon and Minerals in Major World Rivers" (1982–1989) [23–29]. The most significant work among those is the review paper by J. Konta, "Minerals in Rivers" [28]. Another noteworthy publication is a monograph by D. Eisma [30], in which grain size and mineral composition of not only river SPM but also of SPM from other water environments were considered in great detail.

In the 1990s and 2000s, a lot of work was conducted in the Arctic Region by Russian and German scientists [31–33].

The first data on the mineralogical composition of suspended matter in the Severnaya Dvina and other rivers of the White Sea in the former USSR were collected in the mid-1970s [34,35]. Clay mineral and grain size distribution in surface sediments of the White Sea with the analyses of the Severnaya Dvina and Onega SPM were presented to characterize the recent sedimentation processes, reconstruct transport pathways, and identify the sources of terrigenous components [35].

This paper considers the new data on the mineral composition of the Severnaya Dvina SPM (White Sea, Russia). In 2000–2016, under the leadership of A.P. Lisitzin, multidisciplinary investigations were carried out as part of "The White Sea System" project. The results were published in four volumes of collective monographs under the same title in Russian [36–39] and in two volumes in English [40,41]. The features of grain size and chemical and mineral composition of the Severnaya Dvina SPM were considered in [42–44].

From 2015 to the present, systematic studies of those river discharges and the processes of transformation of riverine sedimentary material in the transitional river–sea zone that was called the marginal filter by Lisitzin [45,46] have been carried out as part of the "Observatory of the Marginal Filter of the Severnaya Dvina" project. The results of the first phase of the project (May 2015–May 2019) were published in several papers, including [47–49]. The data on grain size and mineral composition of SPM of the river for 2016–2017 are presented in [50].

The main aim of this work is to present the determinations of the most typical clastic and clay minerals in total suspended sediment samples and their grain size fractions of the Severnaya Dvina and also the features of their seasonal variations for the period of 2015–2019.

2. Sampling and Analytical Methods

2.1. Study Area and Sampling

The Severnaya Dvina belongs to the White Sea basin and represents a pristine river draining taiga forest without permafrost development (Figure 1a). The river has a length of 744 km and a watershed area of 357 × 10^3 km^2; the average water discharge over the period from 1882 to 1998 was 108 km^3/y and the suspended sediment discharge was 4.4 × 10^6 t/y [51]. The coldest month in the river delta is January, with an average temperature of about −12.5 °C; the warmest month is July (+15.6 °C). Average annual precipitations are 400–500 mm, about 70% of which falls during the warm period of the year (April–October) [51].

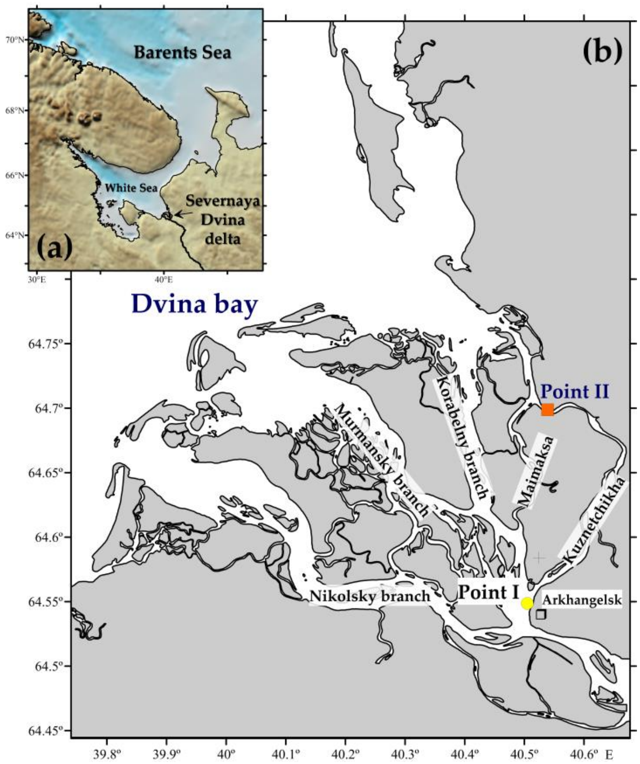


Figure 1. The White Sea position (**a**) and location of two sampling points (**b**).

The watershed area is covered with a thick layer of glacial deposits and underlined by sedimentary rocks. The most typical rocks are dolomites, limestone, clays, and sandstones. A total of 80%–85% of the watershed territory is covered with forests and about 8.5% with bogs that play an important role in the river geochemistry due to significant input of organic and organo-mineral colloids [52,53].

The river delta has a length of 45 km and an area of 900 km^2. One of the main characterizing features of the water level regime in the delta is semidiurnal tides. At the marine edge of the delta, a big tide may reach 1.3 m and neap tide 0.8 m. In the period of low water flux, a front of saline waters (with salinity near 1‰) may penetrate the river upward of the delta for 10–15 km.

Sampling was carried out at two points: on the mooring of a yacht club in the City of Arkhangelsk (point I) and upstream of the Economiya port (point II) (Figure 1b). Point I presents the main stream of the river while point II was used as a river end-member in the river–sea transects to study the processes in the river–sea mixing zone (marginal filter (MF) of the river). Typical flow velocity during spring flooding in the main stream is near 1.0–1.5 m/s and lower in the low-water period of summer, while at point II, velocity is near zero.

Water samples of 200–800 L (the largest volumes were taken during the periods of very low SPM concentrations) collected from the surface with a plastic bucket on a nylon berth were then poured into 50 L polyethylene tanks. After 3–4 days of settling, most of the water was decanted. The residues were transferred to 5 L polyethylene bottles for daily settling after which the excess water was removed, and SPM with residual water was transferred to ceramic dishes and dried in an oven at +45 °C. Dry samples were put into a Petri dish, sealed, and transported to a laboratory in Moscow. This method allows us to obtain a significant quantity of SPM (near 1 g or more). An important negative factor in this procedure is the loss of some of the finest particles that would not settle to the bottom of the tank in the given period of 3–4 days. A longer period of sedimentation does not improve the situation to any significant extent either. Thus, unfortunately, the real share of these particles remains unknown. A preliminary attempt to assess this loss using the Stokes Law showed that in 3–4 days in a 50 L tank, all fractions would settle except the subcolloidal fraction (<0.001 mm). The application of the Stokes Law to this fraction is questionable, so the issue requires further special investigation.

To measure the real concentration of SPM, the samples were filtered through a Nuclepore filter (0.4 μm pore size, 47 mm diameter). A separate 5 L bottle was filled for this purpose.

2.2. Analytical Methods

Total SPM samples were separated into grain size fractions using Petelin's hydraulic (water-mechanical) method [54] and fractions were preserved for further analyses. In a number of cases, the weights of the samples were not enough to fulfill such separation and it was necessary to combine samples of the same months but of different years.

The mineral composition of the Severnaya Dvina SPM samples was studied using X-ray diffractometry (XRD). A D8 ADVANCE (Bruker AXC) diffractometer with irradiation of Cu-Kα, Ni 0.02-filter, 40kV tension, 40 mA current, and LYNXEYE linear detector were used for this purpose. Scanning was carried out in a discrete regime with step 0.02° θ, exposition 8 s/step in interval 2.5°–70° 2θ with rotation. For primary treatment, deciphering of spectrums, and calculations, the DIFFRAC.EVA program was used. For quantitative analyses of total SPM samples, the corundum numbers from the PDF-2 ICDD database (International Centre for Diffraction Data) were employed. To identify the minerals of the montmorillonite group, the samples were saturated with ethylene glycol. All the distortions of the diffraction profile (hallow, high background, and others) may characterize the crystallinity of the samples. This index (Cry., %) is a calculated value that does not have any physical meaning. The calculation is based on the deviation between the sample diffractogram profile and the profile of an ideal crystalline substance. The profile deviation may increase for multiple reasons, i.e., the existence of organics, amorphous silica, imperfections of crystal structure of minerals, etc. However, a comparison of this index in a group of SPM samples may give an idea of their relative crystallinity. This index was calculated for all investigated samples using the DIFFRAC.EVA program.

The samples were reduced to powder consistency in a jusper mortar with alcohol, and the suspension was then put on a flat cell. To obtain the diffractograms from a low quantity of material, the low-background cells are made of a single silicon crystal. Sample preparation for detailed X-ray graphic investigations was carried out using the Stemi 508 stereoscopic microscope (Carl Zeiss Microscopy GmbH, Jena, Germany).

Si and Al in total samples and their fractions were determined using a photometric method on the UNIKO spectrophotometer (Russia). The samples were decomposed through fusion with a mixture of soda and borax in the presence of a small amount of nitrate and subsequently dissolved in distilled water with an addition of HCl (1:3). Detection limits are 0.02% for Al and 0.04% for Si, while standard deviation is 3%–4%. To assess the accuracy of determinations, standard reference materials of oceanic sediments (SDO-1 and SDO-3, Russia) were treated using the same procedure.

3. Results and Discussion

3.1. Water and SPM Discharges

Calculations of river water discharges from the Severnaya Dvina into the White Sea were carried out based on Rosgidromet's (Russian Hydrological Survey) data. The daily water discharges (water level) at the Ust-Pinega gauging station (125 km from the sea) were used to re-count the volumes to monthly and annual discharges (in km^3/month and in km^3/year). The monthly and annual suspended sediment discharges were calculated based on our own determinations of their concentrations at two sampling points and the Rosgidromet measurements slightly upstream of Arkhangelsk city (Figure 2).

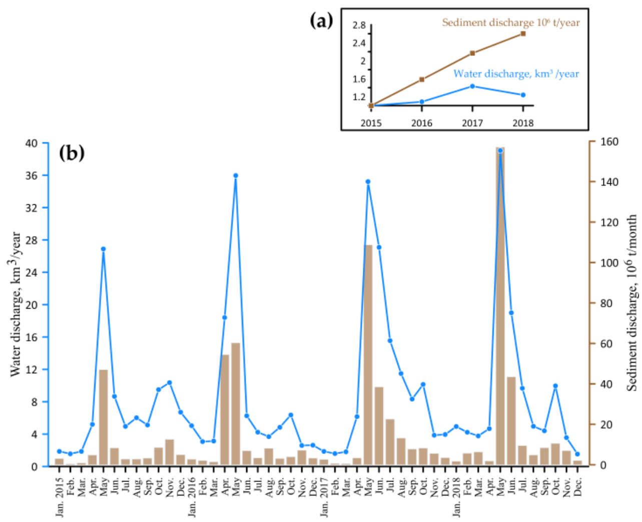
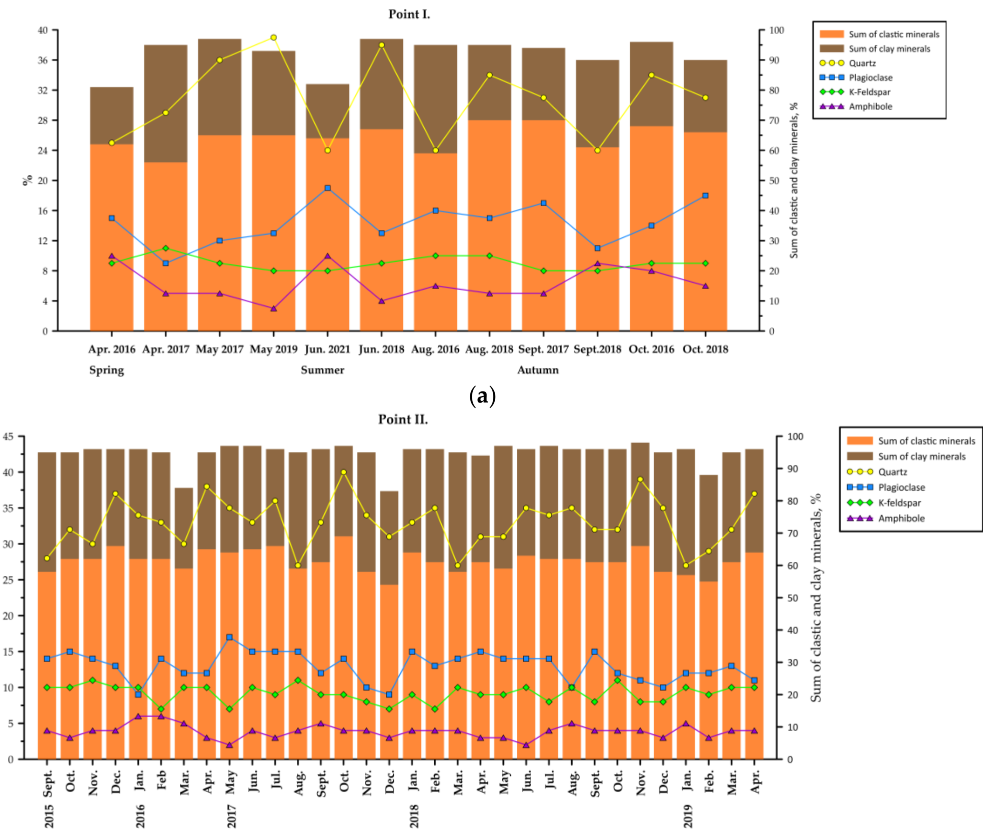


Figure 2. Water and suspended sediment discharges of the Severnaya Dvina in the period from 2015 to 2019 (**a**); inter-annual variation of water and sediment discharges in the period of investigations (**b**).

Two peaks of high water and SPM discharges are clearly distinguished in the periods of spring flood and autumn rain seasons. The river runoff observations were started at Ust-Pinega station at the end of the 19th century. The authors [55] have shown that with time, the water discharge decreased gradually. The downward trend for the period between 1882 and 2014 was detected also in [53] with significant inter-annual variations (from 42 to 170 km^3/y). However, in almost 150 years, there were also periods with significant increases in annual runoff. For instance, in the period from 1981 to 1985, it increased by almost 1.5 times (up to 151.2 km^3/y) [56]. During the period of our investigations, we found a 43% increase in runoff. At the same time, the increase in SPM flux from 2015 to 2018 was much higher—about 2.5 times.

3.2. Mineralogy of Total Samples of SPM

The investigations of the mineral composition of 13 total SPM samples from sampling point I (Table S1a; Figure 3a) demonstrate that the clastic mineral group represents quartz (24%–39%, av. 30.8%), plagioclase (9%–19%, av. 14.3%), potassium feldspars (8%–11%, av. 9.0%), minerals of the amphibole group (hornblende, tremolite) (3%–10%), and minerals of the pyroxene group (diopside, augite) (2%–3%). In the samples of August, September, and October, epidote was diagnosed (from trace to 7%). The total content of all clastic minerals varied from 56% to 70%.



(**a**)

(**b**)

Figure 3. (**a**) Variations of the sum of clastic minerals and the sum of clay minerals and of the main clastic minerals for sampling point I. (**b**) The same as (**a**) but for sampling point II.

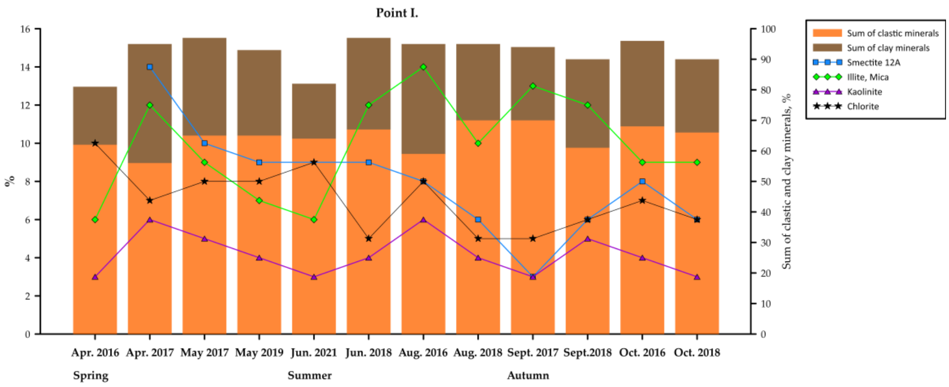
The 32 total SPM samples from point II showed the following results: quartz (27%–40%, av. 28.9%), albite (plagioclase) (9%–17%, av. 11.0%), potassium feldspars (7%–11%, av. 9.1%), minerals of the amphibole group (hornblende, tremolite) (2%–6%), and minerals of the pyroxene group (diopside, augite) (2%–4%). The sum of all clastic minerals varies in the range of 54%–69% (Table S1b; Figure 3b).

The results demonstrate quite a similar composition of clastic minerals in total SPM samples from both sampling points. It is worth pointing out, however, that the average contents of main clastic minerals are higher in samples from point I compared to samples from point II. The sum of clastic minerals in the first point (64.4%) is higher than that in the second one (59.6%). This fact may be related to differences in hydrological regimes at two sampling points.

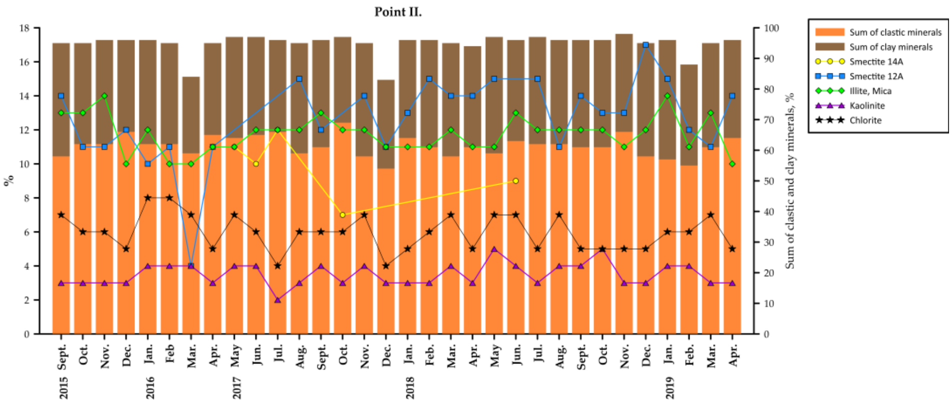
Epidote was found in three samples from point I (up to 7%), while in the 32 samples from point II, this mineral was not detected.

According to our data, among the clastic minerals in the SPM samples of the Severnaya Dvina, quartz, plagioclase, and potassium feldspar are the primary ones. This result coincides with the data by J. Konta [25–27], who investigated large rivers in North and South America, Europe, Africa, and Asia and identified the same clastic minerals that commonly occur in suspended solids of rivers across the globe. The mineralogical analyses of SPM from 14 rivers of the former USSR [18] showed that the quartz content in total samples was on average near 44% (27% in pelit fraction). In our expedition to the rivers of the Black, Azov, and Caspian seas in May of 1976 [19], the SPM samples were collected using the same method of sedimentation. The content of quartz was in a range from 10% (The Volga) to 30% (The Danube) and the content of potassium feldspars ranged from 3% (the Volga) to 12% (The Ingury, Georgia). The sum of clastic minerals varied between 13% (The Volga) and 48% (The Terek, Caspian Sea). The mineralogical data for the Severnaya Dvina are scarce [42,44,47]. The results were presented for the 14 samples collected in May 2004 [42] and five samples collected in May and August of 2006 [44]. The content of quartz was in the range of 9%–29%, av. 20%; plagioclase—5%—21%, av. 11.8%; and potassium feldspar—1%–20%, av. 8.2%. The sum of clastic minerals was on average about 55%, while in the present work, this value equals 59%. The comparison of our data with the results of previous works demonstrates that in spite of the fact that the majority of samples in [42,44] were collected in the spring of 2004, the average content of quartz (20%, n = 19) in those was 1.5 times lower than in our samples (29.8%, n = 44). At the same time, the average contents of plagioclase and potassium feldspar appeared to be similar.

Clay minerals (sampling point I) (Table S1b; Figure 4a) include illite (6%–14%, av. 9.7%), magnesia–ferriferous chlorite (5%–10%, av. 7.0%), and kaolinite (3%–6%, av. 4.2%). Dioctahedral smectite (from traces to 14%, av. 6,6%) was diagnosed with small variations of inter-layer basal reflection value d_{001} (from 12.2 to 13.5Å) that indicates constancy in the composition of inter-layered cations with priority of Na^{1+} and K^{1+}. Traces of mixed-layered formations were found in several SPM samples. The total quantity of clay minerals was in the range of 18%–39%, av. 27.7%. Sometimes minerals from the serpentine group were detected, without any connection to seasonal prevalence.



(**a**)



(**b**)

Figure 4. (**a**) Variations of the sum of clastic and clay minerals and main clay minerals for sampling point I. (**b**) The same as in (**a**) but for sampling point II.

The samples from point II contained illite (10%–14%, 10.7%) and magnesia–ferriferous chlorite (4%–8%, av. 6.0%). Dioctahedral smectite was found (7%–17%, av. 10.7%) with variable values of inter-layered basal reflection d_{001} (12.3–14.5 Å) connected with variations in the composition of inter-layered cations (Ca^{2+}, Mg^{2+}, K^{1+}, Na^{1+}). The sum of clay minerals in these SPM samples varied in the range of 25%–39%, av. 33.3% (Table S1b; Figure 4b).

Comparison of clay mineral composition in the samples from sampling points I and II demonstrates significant similarity for illite, chlorite, kaolinite, and smectite; however, the average total sum of clay minerals in contrast to clastic minerals is higher at point II.

The first determinations of clay minerals in the SPM samples of 14 rivers of the former USSR showed the following results (on average): illite—22%, chlorite—11%, kaolinite—4% [18]. The SPM in the rivers of the Black, Azov, and Caspian seas contained chlorite in the range from 15% (The Danube) to 23% (The Rioni, Georgia), illite from 16% (The Kura, Azerbaijan) to 36% (The Volga), and kaolinite from 5% (The Terek) to 21% (The Sulak) [19]. Clay minerals in pelit (<0.01 mm) and subcolloidal (<0.001 mm) fractions of SPM in the Severnaya Dvina were determined in [42,44,47]. The results from [42,44] were as follows in pelit and subcolloids accordingly: illite—46.9% and 41.9%, chlorite—20.0% and 17.9%, smectite—15.9% and 20.9%, kaolinite—18.9%–18.2%. The sum of clays is on average (n = 19) 47.4%, which is close to the sum of clastic minerals. Therefore, according to our data, the total SPM of clay minerals (n = 44) is as follows: illite—10.4%, smectite—9.6%, chlorite—6.3%, kaolinite—3.7%. It is difficult to compare our results with those from [42,44] as we do not have the determinations in total SPM samples. Another reason for the differences in our results and the results of these two papers (both for clastic and clay minerals) may be related to the fact that we used different sampling points. The authors of the aforementioned works took samples from multiple points across all the main arms of the river—Nikolsky, Murmansky, Korabelny, Maimaksa, and Kuznetchikha (Figure 1). Illite is the most widespread clay mineral in the SPM of the Severnaya Dvina. The quaternary ice-born sediments in the White Sea catchment area are enriched by illite. The content of illite in the glacial deposits (<0.01 mm and <0.001 mm fractions) is varying between 40% and 90% [35,44]. These deposits are among the main sources of terrigenous material supplied by the river to the sea. Ref. [21] showed that the distribution of clay minerals in riverine SPM was climate-dependent. Different climatic zones vary significantly in terms of prevailing clay minerals. In the cold humid temperate climate that is typical for the Severnaya Dvina catchment area, illite is the dominating clay mineral, and our results support this classification.

Besides the clastic and clay minerals, several minerals from other groups were detected in our samples. For instance, from the carbonate group, calcite (trace—5% in point I), dolomite (2%–6%), and aragonite (1%) were detected. Three samples from point II contained halite (8%–12%) and gypsum (1%–2%). As was mentioned above, during the periods of high tide, the brackish waters may reach the location of point II. SPM samples may contain a low quantity of halite which is dissolved in fresh water used to prepare the samples for analyses.

The crystallinity of the total SPM samples is decreasing along with the increasing content of vegetation detritus. The lowest crystallinity degrees were detected in the sample of August 2017 (point II)—43%, and the sample of September 2017 (point I)—40%. As particle dimensions in grain size fractions decrease, crystallinity is also usually decreasing.

3.3. Grain Size Composition of SPM

The results of grain size determinations of SPM samples collected at the two points are presented in Table S2 of the Supplementary Materials and in Figure 5.

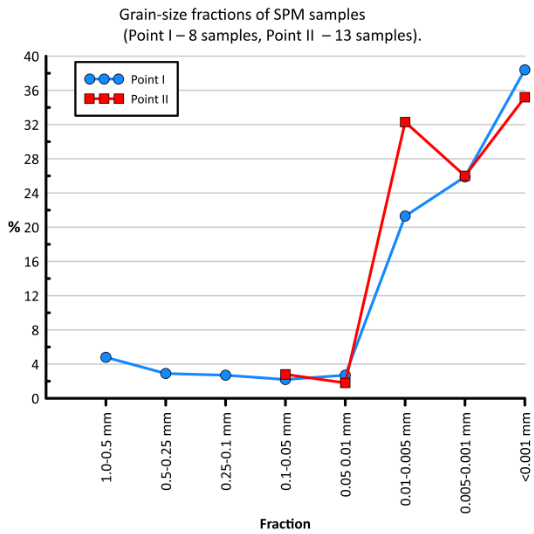
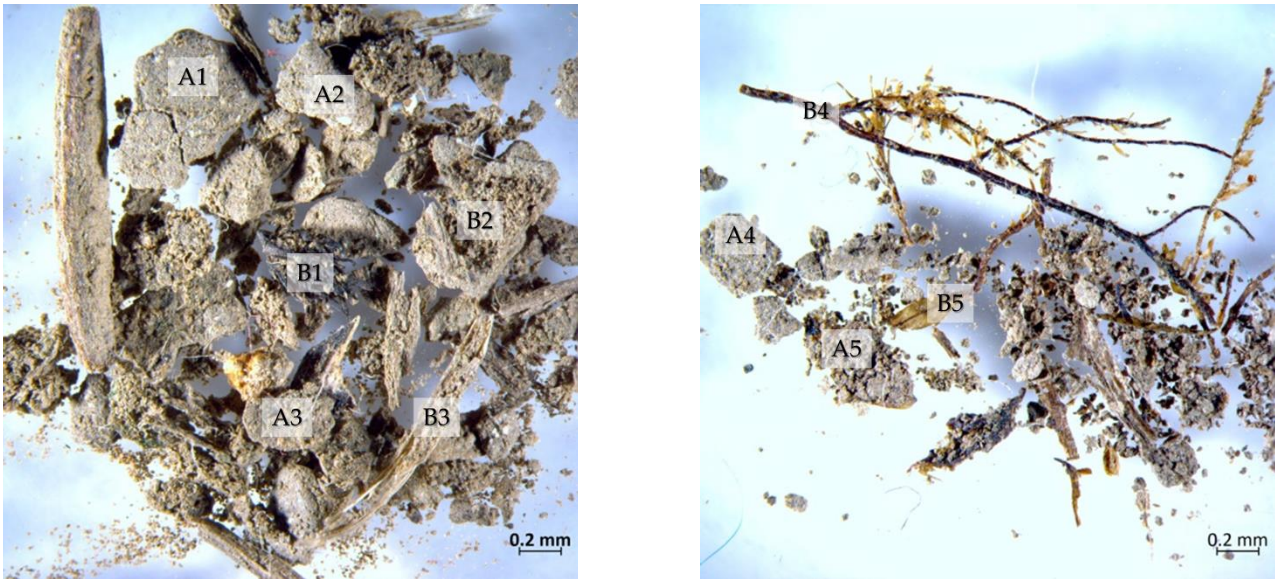


Figure 5. The content of grain size fractions in samplings from point I and II.

The data show that pelitic fractions (<0.01 mm) predominate in the Severnaya Dvina suspended matter (85.7% on average at point I and 93.4% at point II, percentages of subcolloidal fraction are 38.4% and 35.2%, respectively). The share of silt fraction (0.01–0.1 mm) is much lower at about 4%–5%. Unexpected results were obtained for the so-called sandy fractions (0.1–1.0 mm): while at point I, 0.1–0.25 mm, 0.25–0.5 mm, and 0.5–1.0 mm fractions were detected in all eight samples, at point II, these fractions were absent in 13 samples. Among the studied samples, those of September (2017 + 2018) and October (2016 + 2018) (Table S2) stand out the most with the content of the three coarsest fractions reaching 36.52% and 11.77%, respectively. That made a significant impact on the decreasing share of finer fractions.

The main part of these fractions at point I was detected as particles of wood debris and anthropogenic detritus. Typical SPM particles in 0.5–1.0 mm fractions of the two samples are shown in Figure 6. The sources of these particles are most probably the woodworking enterprises located along the shores of the river. The mineral part is rarely presented here as terrigenous fragments of minerals; more frequently, these are the mineral multi-phase aggregates.



(**a**) (**b**)

Figure 6. Photo of 1–0.5 mm fraction: (**a**) August 2016; (**b**) June 2021. A1–A5—terrigenous fragments of minerals d; B1–B5—wood debris.

The 0.05–0.01 mm fraction of May 2017 sample (point I) evidently stands out with 12.82% against a background of 1%–2% in other samples that resulted in 1.5 times higher average share of this fraction in samples from point I in comparison with samples from point II (Figure 5).

The comparison of average annual contents of different fractions from samples taken at points I and II shows that besides the differences between the coarsest fractions, the fraction of coarse pelit (0.01–0.005 mm) (21.3% at point I and 32.4% in point II) is notable. Attempts to explain the reasons for these occurrences are made after the examination of mineralogical analyses in Section 3.4.

The difference in grain size composition of SPM from sampling points I and II depends largely on different hydrological regimes in these points. While point I is located in the main stream of the river upper-mouth area where the rate of flow is near 0.5–1.5 m/sec, point II is situated in quite a calm place where the flow is practically absent. Judging by the absence of 0.1–1.0 mm fractions in all SPM samples at point II, it may be suggested that the main part of the wood debris in these fractions is carried by the stream of the main navigable arm of the Maimaksa delta into the sea and does not penetrate the Economiya port area.

In May 2004 [42] after SPM sampling using the method of sedimentation, the determinations of grain size fractions were made using the same hydraulic method as in the present work [53]. The comparison of data demonstrates that in the 2004 flood period, seven SPM samples contained on average 0.5% of 0.25–0.1 mm fractions, 9% of silts (0.1–0.01 mm), and 90.4% of pelit (<0.01 mm), including 57.3% of subcolloidal fraction (<0.001 mm), while in the two samples from sampling point I in the present work in May 2017 and 2019, the same fractions were 4.3%, 8.4%, 87.5%, and 38.4%, respectively. It is easy to see that the coarse fractions in May 2004 were almost one order lower with about 1.5 times higher share of subcolloidal fractions. We explain this difference by the absence of coarse fractions with woody debris in May 2004.

3.4. Mineralogy of SPM Grain Size Fractions

The results of mineralogical determinations in grain size fractions of river SPM samples are presented in Table S3a,b. The group of clastic minerals consists of the same minerals as the total samples: quartz, albite, potassic feldspars, the amphibole group (hornblende, tremolite), the pyroxene group (diopside, augite), and muscovite. The sum of these minerals is fluctuating in a range from 49% to 67% in the 1–0.5 mm fraction to a maximum of 61%–87% in the 0.05–0.01 mm fraction. Quartz is the main clastic mineral and its fluctuations play the leading role in these variations. Antigorite (the mineral from the serpentine group) was found in all fractions. Its highest content (12%) was measured in a sample of 2021, 0.25–0.1 mm fraction. The exceptional case is the sample of May 2017 in which serpentine was found in two fractions only—0.25–0.1 mm and 0.1–0.05 mm. Muscovite is drawn towards to crude fractions—from 1.0 to 0.05 mm.

Clay minerals are present in all grain size fractions. This fact looks a little unexpected: how can pelitic-size clay appear in fractions of a much bigger size? Alternatively, how can we explain the fact that quartz, which is a typical clastic mineral found in sand and silt size fractions, is sometimes found in subcolloidal fractions? Our data demonstrate that practically all minerals of different types may be present in any fraction (Table S3). In all <0.001 mm fractions of the SPM samples taken from rivers of the Black, Azov, and Caspian seas, besides clay minerals, we also found quartz and potassium feldspars, and often calcite. Additionally, in the same size fraction of the Volga SPM, we identified the presence of barite and palygorskite [19]. Depending on their genesis, the particles mfound in different fractions may be aggregates of several minerals or monominerals.

The group of these minerals includes kaolinite, magnesia–ferriferous chlorite, illite, and dioctahedral smectite. The total content of clay minerals in different fractions is widely variable. As was expected, the lowest clay content (7%) was detected in the combine sample of August 2016 + 2018 in 0.05–0.01 mm fraction, and the highest (up to 56%) was found in the subcolloidal fraction of the same sample (<0.001 mm) (Table S3). The results show that the quantity of clays primarily depends on the contents of smectite and illite.

Figure 7 demonstrates the fragments of diffractogram of two different fractions that confirms the presence of all kinds of minerals in every fraction of suspended matter.

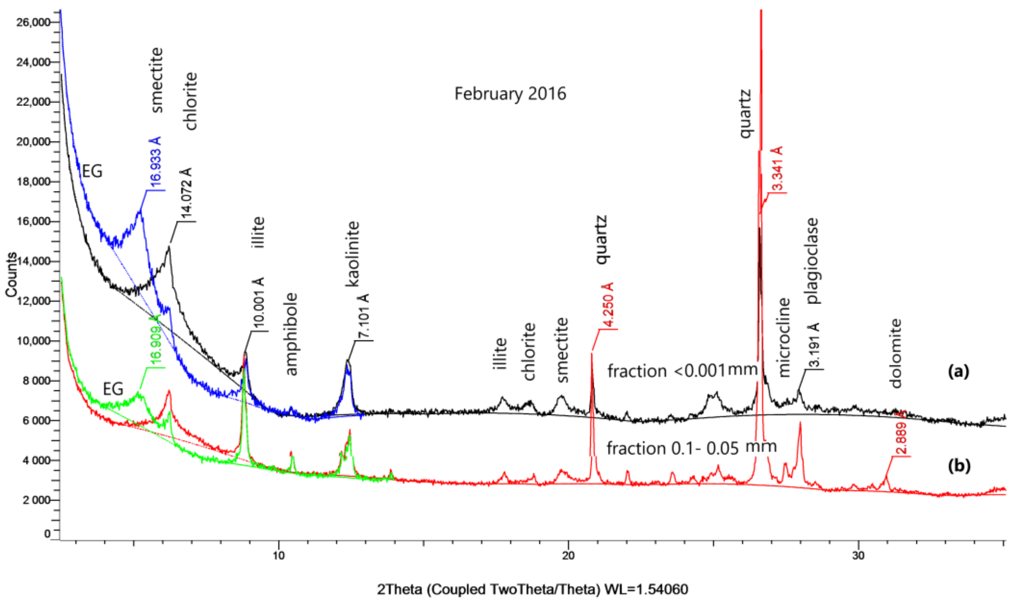


Figure 7. Fragments of X-ray diffraction patterns of suspended matter of Severnaya Dvina. River: (**a**) <0.001 mm fraction; (**b**) −0.1–0.05 mm fraction. EG—fragments of diffraction patterns of the same samples saturated with ethylene glycol for the identification of smectite.

It should be noted that in all grain size fractions of each SPM sample, dolomite was detected in quantities varying from traces to 12%. Its minimum content was found in the sample of May 2017 in coarse fractions (1.0–0.05 mm), while the maximum content was measured in the combined sample of October 2016 + 2018 in the 0.05–0.01 mm fraction.

Among other minerals, up to 4% of calcite was found in the sample of September 2017 + 2018 (1.0–0.5 mm fraction).

Figure 8 demonstrates the distribution of average values of the sums of clastic and clay minerals in different fractions.

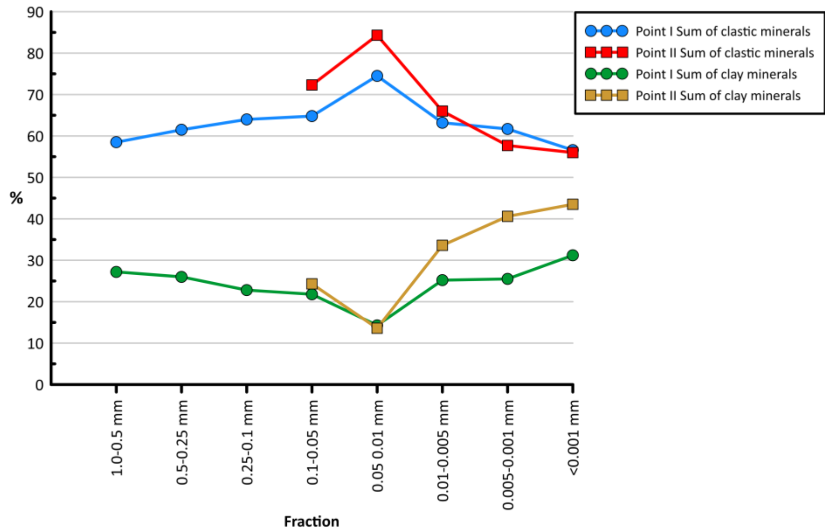


Figure 8. Variations in the sum of clastic and clay minerals at sampling points I and II.

The silts stand out clearly. The sum of clastic minerals reaches its maximum in the 0.05–0.01 mm fraction (up to 85%). This was not unexpected. As far back as in 1988, the author [27] wrote that the content of silicon largely increased with the rising portion of silt (in the Mississippi and Colorado Rivers). Our data support this conclusion: within these fractions, the content of quartz is near its highest, 66%–72%, when the median values are about 40%–60% and the content of silica is near 29%–34% vs. the typical 25%–27%. The sum of clay minerals in this fraction is minimal.

Let us consider the possible reasons for the differences between fractions in SPM samples from the two sampling points.

The 0.05–0.01 mm fraction in the sample of May 2017 stands out against a background of all other samples in this fraction. The coefficient of crystallinity is the lowest here. The results of mineralogical analyses show that the fraction of this sample is enriched with clay minerals, and it is these minerals that are the reason for a higher content of clay fraction. At the same time, the overall trend in clay behavior in terms of fraction distribution (Figure 8) indicates that May 2017 is an anomalous one.

The share of 0.01–0.005 mm fraction in samples from point II is higher than that in samples from point I (32.4% against 21.3%) (Figure 5). The mineralogical composition of these fractions (Table S3a,b) and Figure 8 shows that the sum of clay minerals in the 0.01–0.005 mm fraction is significantly higher in samples from point II in comparison to the samples from point I (33.6% against 25.5%). At the same time, the sums of clastic minerals are similar (66.0% and 63.2%). The excess of clay minerals at point II is mainly due to the high content of smectite and illite. The conclusion is following that the prevailing of this fraction in samples from point II is, first, the result of the high sum of clay minerals and second, may be due to an insignificant excess of a sum of clastic minerals in this fraction.

Lastly, the subcolloidal fraction (<0.001 mm) of the March 2017 sample (point II) is enriched significantly comparing the fractions of this type for other months. In our previous paper [47], it was shown that a significant part of this fraction is mainly connected with the contribution of fine dispersed clastic minerals (mainly quartz).

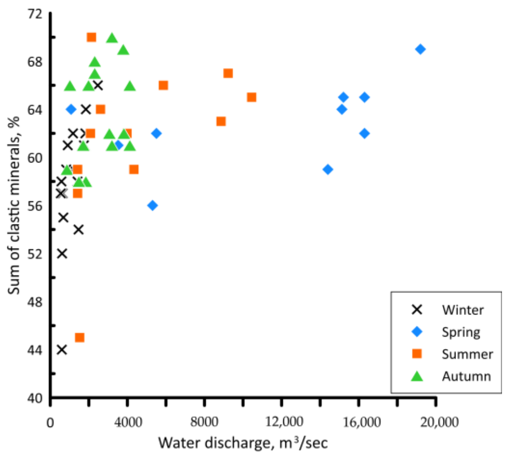
Figures 5 and 8 demonstrate the general trends of variations of clastic and clay minerals from crude fractions to the finest ones. From the fractions of sands to the fractions of silts, the role of clastic minerals is increasing, while the role of clay minerals is decreasing. The content of clay minerals reaches the minimal level in the 0.05–0.01 mm fraction (fine silts) with a corresponding increase in the sum of clastic minerals. This is based on the results of at least six samples and causes no doubts on this ground. The quantity of clay minerals is growing with decreasing sizes of particles at both sampling points, with a visible excess at point II, and a slight decreasing in the sum of clastic minerals at both sampling points. At the same time, it is necessary to point out that this sampling method entails the loss of the finest fractions during sedimentation, and the percentage of these losses is bigger for those samples with lower SPM concentrations.

So, we can see that the variations in clastic and clay minerals in different grain size fractions of suspended matter in the Severnaya Dvina are not very stable. If in one case (0.05–0.01 mm fraction in the sample of May 2017, point I) the percentage of the fraction is higher in samples from point I, then in the second case (0.01–0.005 mm fraction in samples of point II), it is higher in samples from point II due to a higher content of clay minerals, while in the third case (<0.001 mm fraction in sample II), the reason of high content of the fraction was its enrichment with the finest clastic minerals.

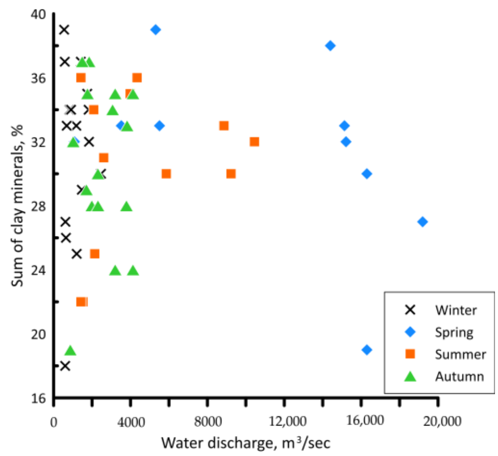
3.5. Seasonal Variations of Mineralogical Composition of SPM Samples

The results of the investigations of seasonal variations of chemical elements in water and suspended matter in the Severnaya Dvina were presented in a number of recent papers [46,48,51,52].

The variable hydrological water regime and significant changes in the concentration of organic carbon and colloidal fraction of dissolved carbon, iron, and many other elements lead to seasonal variations of their concentrations. It is interesting to see if there are any seasonal variations of mineral composition in this river. In Figure 9a,b, the dependences of the sum of clastic minerals and the sum of clay minerals on the seasons are shown.



(**a**)



(**b**)

Figure 9. Dependence of the sum of clastic (**a**) and clay (**b**) minerals on the water discharge across the seasons.

We take into account the seasonal variations of water and suspended sediment discharges (Figure 2) and only include two months in the spring—April and May—while adding another month to the winter: December, January, February, and March. In this case, we combine all total SPM samples from the two sampling points together. Scattering of figurative points is significant in both figures. The average contents of both types of minerals for every season are shown in Figure 10.

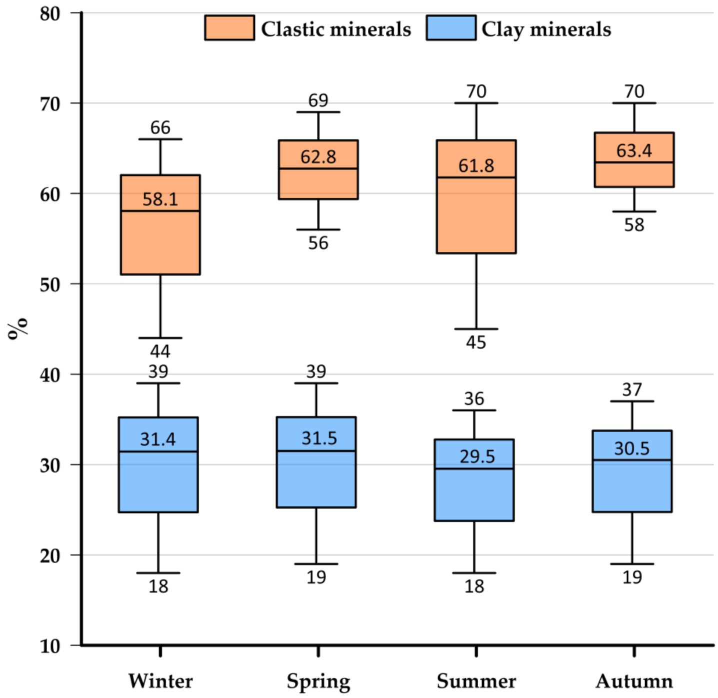


Figure 10. Average content and standard deviations of clastic and clay minerals in different seasons of a year.

As shown, the real difference between the seasons is of low significance. We may point to the following trend in terms of increasing sums of clastic minerals: winter–summer—-spring–autumn. Seasonal variations of clay minerals are not meaningful.

Coefficients of correlations between the sums of clastic and clay minerals and water runoff (m3/sec) confirm this trend: clastic minerals—0.344, $p < 0.05$, $n = 54$; clay minerals—-0.043, $n = 54$. The crystallinity is increasing in the same direction as the clastic minerals. So, the highest content of clastic minerals is found in the spring and autumn when the water discharge and SPM concentrations are highest, while the lowest mineral content is typical for the summer and winter during the periods of low discharges and SPM concentrations.

The determinations of Si and Al in all total SPM samples (Table S1a,b) were carried out to identify any confirmations of clastic and clay mineral seasonal distribution, as the interrelations between clastic minerals and Si and clay minerals and Al are well-documented and known [27]. The calculations show that the coefficient of correlation between Si and clastic minerals is 0.385, $p < 0.05$, $n = 40$, and between Al and clay minerals, it is 0.390, $p < 0.05$, $n = 40$.

These results demonstrate that seasonal variations of mineral composition of SPM in the Severnaya Dvina are quite insignificant and we may only speak about its tendency towards a slight increase in the clastic mineral content in periods of high water (spring and autumn) and a slight decrease in the summer and winter. The variation of the sum of clay minerals is practically negligible. The average content of the sum of clastic minerals changes only slightly (from 58% in winter to 63% in spring), while the sum of clay minerals is stable all year around (from 30% to 32%, that is within the limits of error). At the same time, our previous publications and literature data on the chemical composition of the Severnaya Dvina SPM [48,51,52] demonstrate much more significant seasonal variations in multiple chemical elements in comparison to mineral composition.

This can be linked to variations in amorphous and poorly crystalline phases including Fe and Mn oxides, allophanes, but also organic materials of both allochthonous and autochthonous nature. These phases can accommodate sizable amounts of trace metals and micronutrients. Unfortunately, they were not assessed in the present study and should be subjects of further research.

In these considerations, we must keep in mind the losses of the finest fractions during the application of the water-mechanical method of grain size analyses. This question requires special investigation and discussion in future publications.

4. Conclusions

The mineralogical composition of SPM in rivers attracts the attention of many marine geologists because these data appear to be useful in the investigations of riverine sedimentary material transport and sedimentation processes in the sea basins. A brief review of scientific literature shows that extensive research in this field was started nearly a century ago.

The present work is devoted to the generalization of the results of the analyses of mineralogical composition of suspended matter in Severnaya Dvina River (the White Sea basin) over 5 years. Sampling of SPM was carried out every month in all these years at two sampling points with different hydrological regimes that permitted us, for the first time, to obtain the information not only about the contents of the clastic, clay, and some other minerals in the total SPM samples but also in different grain size fractions, as well as to characterize their seasonal variations. The prevailing element in the group of clastic minerals is quartz, and further in order of decreasing of content are plagioclase, potassium feldspars, minerals of the amphibole group (hornblende, tremolite), and minerals of the pyroxene group (diopside, augite). The sum of these minerals varies from 54% to 70%. Among clay minerals, illite and smectite have the highest content in SPM, while kaolinite and chlorite are found in lower quantities. The sum of clay minerals varies from 18% to 39%.

Some other minerals such as those of the carbonate group (calcite, dolomite, aragonite), as well as serpentine, epidote, anatase, and gypsum were detected from time to time. Halite was found in three samples collected at sampling point II, which may be reached periodically by saline waters.

The results of the water-mechanical (hydraulic) method of analyses of the total SPM samples showed evident prevalence of pelitic fractions (<0.01 mm)—85.7% at point I and 93.4% at point II (the finest subcolloidal fraction of <0.001 mm is 38.4% and 35.2% accordingly). Silt fractions (0.1–0.01 mm) were much lower—near 4%–5%. Unexpected results were received for the so-called sandy fractions—between 1.0 and 0.1 mm. The microscopic investigation of these fractions shows a lot of wood debris and anthropogenic detritus. It is interesting that while at point I, 0.1–0.25 mm, 0.25–0.5 mm, and 0.5–1.0 mm fractions were detected in all eight samples (from 2.5% to 36.5%), at point II, these fractions were absent in 13 samples. The mineral part is presented here by terrigenous fragments of minerals or mineral multi-phase aggregates.

Clay minerals are presented in all grain size fractions. Smectite and illite are the most important clay minerals in all fractions. This conclusion coincides with the dependence of clay minerals on the climate zone of Severnaya Dvina with a cold, moderately wet climate.

The comparison of average values of the sums of clastic and clay minerals in different fractions reveals the highest content of clastic minerals in silts (0.05–0.01 mm) and the lowest content of clay minerals in the same fraction. Our results support the conclusion that clastic quartz occurs in a higher proportion compared to the content of quartz in clay.

For the first time, we observed the seasonal and annual variations of mineral composition of river suspended matter in the Severnaya Dvina. Our data testify in general a quite stable mineral composition of the river SPM both during the year and also between years. The results show that the sum of clastic minerals varies between seasons within less than 8%, reaching the maximum levels in the spring and autumn during the seasons with high discharge and minimum levels in the summer and winter. Seasonal variations of the sum of clay minerals could not be resolved within the uncertainties of our measurements.

Further work on ultrafine mineral fractions and suspended amorphous and poorly crystalline minerals as well as organic matter is necessary to quantify the total export of solid material from the Severnaya Dvina watershed to the Arctic Ocean.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/min12121600/s1, Table S1(a): Mineralogical composition of suspended sedimentary material at Point I. XRD diffractometry; Table S1(b): Mineralogical composition of suspended sedimentary material at Point II. (Ekonomiya). XRD diffractometry; Table S2: Grain-size fractions at Point I and Point II; Table S3(a): Mineralogy of SPM grain-size fractions at Point I; Table S3(b): Mineralogy of SPM grain-size fractions at Point II.

Author Contributions: Conceptualization, V.V.G.; Methodology, V.V.G. and O.M.D.; Software, A.S.F.; Validation, V.V.G. and O.M.D.; Formal Analysis, V.V.G.; Investigation, S.K.B. and A.S.L.; Resources, E.I.K.; Data Curation, A.I.K.; Writing—Original Draft Preparation, V.V.G. and O.M.D.; Writing—Review and Editing, V.V.G.; Visualization, A.S.F. All authors have read and agreed to the published version of the manuscript.

Funding: The work was partly supported by Russian Science Foundation grant N 19-17-00234p: "Biogeochemical accumulation and migration of organic compounds, heavy metals and radionuclides in the components of ecosystems of the Arctic seas". Analyses were performed as part of the State Assignment of the IO RAS for 2022, on topic no. FMWE-2021-0006.

Data Availability Statement: The data are available upon request with reasonable purpose by e-mail to the corresponding author.

Acknowledgments: The authors would like to express their appreciation and thanks to Oleg S. Pokrovsky for his valuable recommendations on improvements to the manuscript. We thank Tatiana N. Alekseeva for the grain size analyses and isolation of fractions for the following investigations, and Elena O. Zolotykh for determinations of Si and Al in suspended sediments.

Conflicts of Interest: The authors declare no conflict of interest.

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