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Re-assessing the nitrogen signal in continental margin sediments: New insights from the high northern latitudes

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Abstract

Organic and inorganic nitrogen and their isotopic signatures were studied in continental margin sediments off Spitsbergen. We present evidence that land-derived inorganic nitrogen strongly dilutes the particulate organic signal in coastal and fjord settings and accounts for up to 70% of the total nitrogen content. Spatial heterogeneity in inorganic nitrogen along the coast is less likely to be influenced by clay mineral assemblages or various substrates than by the supply of terrestrial organic matter (TOM) within eroded soil material into selected fjords and onto the shelf. The $\delta^{15}N$ signal of the inorganic nitrogen ($\delta^{15}N_{inorg}$) in sediments off Spitsbergen seems to be appropriate to trace TOM supply from various climate- and ecosystem zones and elucidates the dominant transport media of terrigenous sediments to the marine realm. Moreover, we postulate that with the study of sedimentary $\delta^{15}N_{inorg}$ in the Atlantic–Arctic gateway, climatically induced changes in catchment's vegetations in high northern latitudes may be reconstructed. The $\delta^{15}N_{org}$ signal is primarily controlled by the availability of nitrate in the dominating ocean current systems and the corresponding degree of utilization of the nitrate pool in the euphotic zone. Not only does this new approach allow for a detailed view into the nitrogen cycle for settings with purely primary-produced organic matter supply, it also provides new insights into both the deposition of marine and terrestrial nitrogen and its ecosystem response to (paleo-) climate changes.

Keywords: Spitsbergen; continental margin; sediments; stable isotopes; nitrogen; inorganic and organic

1. Introduction

The stable nitrogen isotope ratio ($\delta^{15}N$) of organic matter (OM) has been regularly used to track relative nutrient utilization in nitrate replete environments (e.g. [1–3]). The isotopic signature produced in the photic zone depends on the isotopic composition of nitrate and the degree to which this inorganic nitrogen pool is utilized [4]. For instance, isotopically light particulate OM (low $\delta^{15}N$ values) is produced during nutrient (nitrate) replete conditions when physical supply of nitrate exceeds biological demand, and vice versa (e.g. [2]). This simple relationship can be complicated in regions of significant denitrification [5] and/or atmospheric nitrogen fixation [6]. Diagenetic overprinting during water column transport and/or after burial may alter the $\delta^{15}N$ value of the organic detritus further [7,8].

Nitrogen isotope ratios in Arctic Ocean sediments have been shown to be reliable indicators for relative

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nutrient utilization in surface waters [9,10]. Their application in sediments from the high latitudes or from continental margins with significant supply of terrestrial organic matter (TOM) [11], however, is more complicated because of mixing of isotopic signals from different sources. Atmospheric nitrogen fixed by land plants and soil organic matter can significantly modulate the total $\delta^{15}N$ record towards lower values [12]. Peters et al. [13] concluded that mixing of terrestrial and marine organic matter is the dominant process that determines the $\delta^{15}N$ isotopic signature in coastal sediments of the northeast Pacific. Schubert and Calvert [9] outlined the major impact of ammonium bound between the lattices of clay minerals on the bulk nitrogen content and its isotopic signature in Arctic Ocean surface sediments. They showed constant $\delta^{15}N$ values of bound ammonium ($\delta^{15}N_{inorg}=3.0\pm1.2‰$) in the central Arctic Ocean indicating a major supply of soil from adjacent coastal settings by sea ice and ocean current transport mechanisms [14]. Accordingly, the importance of inorganic nitrogen and its distinct isotopic signature as known not only from the Arctic [9], but also the South China Sea [11] may require re-evaluation of interpretations of the bulk nitrogen content and isotopic composition of coastal sedimentary organic matter.

In this follow-up study, we build on the results by Schubert and Calvert [9] and Kienast et al. [11] and present data from continental margin sediments off

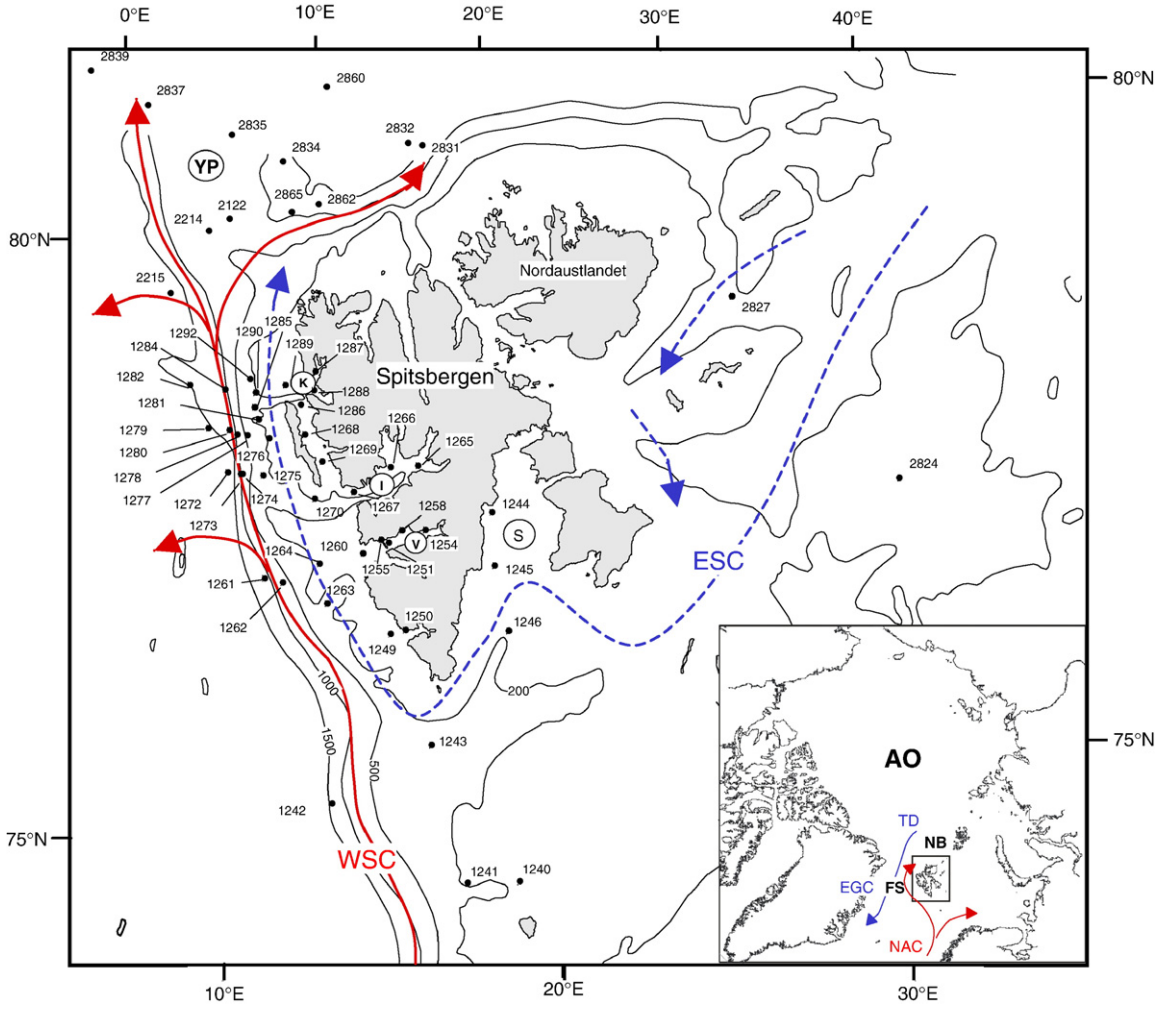

Fig. 1. Bathymetry and major surface water currents in the study area around Spitsbergen. Blue arrows indicate the East Spitsbergen Current (ESC), red arrows the warm Atlantic water derived West Spitsbergen Current (WSC). Sample locations are also displayed. S = Storfjorden, V = van Mijenfjorden, I = Isfjorden, K = Kongs–Krossfjorden, YP = Yermak Plateau. The water depth isolines are labelled. The inset shows a simplified map of surface waters in the Nordic seas. Blue arrows indicate the cold Transpolar Drift (TD), and East Greenland Current (EGC), red arrows the North Atlantic Current (NAC). Abbreviations are: AO = Arctic Ocean, FS = Fram Strait, NB = Nansen Basin.

Spitsbergen. We show that the $\delta^{15}N$ of these sediments is strongly influenced by inorganic nitrogen, which can make up 70% of the sedimentary nitrogen. The new data demonstrate a close link between the inorganic nitrogen content of sediments and distance to the shore, the hinterland geology and the sediment transport processes (e.g., sea ice drift and ocean currents). We also introduce the $\delta^{15}N$ isotopic signature of the inorganic nitrogen as a tracer of the origin of TOM and show that it tracks climatically induced changes in the global pattern of $\delta^{15}N$ values of soil organic matter [15]. Finally, we elucidate the response of the $\delta^{15}N_{org}$ signature in surface sediments to nutrient-rich Atlantic water inflow and topographically steered local upwelling cells.

2. Modern hydrography

The northward extension of the Norwegian Atlantic Current (NAC) (Fig. 1, inset) flows along the continental slope of the western Barents Sea and west off Spitsbergen to become the West Spitsbergen Current (WSC). The upper 600 m of the WSC are composed of relatively warm and saline water, which causes the western coasts of Spitsbergen to be essentially free of ice for most of the year [16]. When Atlantic water leaves the Nordic Seas through the Fram Strait, its temperature is lowered by 5 °C compared to its entry and salinity varies around 35.0 [17]. The WSC supplies the largest heat, water mass and salt amounts to the Arctic Ocean [18].

The WSC splits during its northward flow into three major branches, (1) the western branch or Return Atlantic Current, (2) the northward-directed Yermak Branch and (3) the north- and later eastward flowing Spitsbergen branch [19]. The waters of the Spitsbergen Branch enter the Arctic Ocean northwest off Spitsbergen. The relatively warm (>0 °C) and saline (>35) waters advect further East on the continental slope at intermediate depths. The Return Atlantic Current meets the cold East Greenland Current (EGC), which transports sea ice from the Transpolar Drift into the Atlantic Ocean (Fig. 1). A second Arctic type current (<0 °C and salinities <34.4), the East Spitsbergen Current (ESC), enters the Barents Sea from north between Franz Joseph Land and Nordaustlandet, flows south-west around Edgeøya and Sørkapp and then turns north along the west coast of Spitsbergen (Fig. 1) [20].

3. Materials and methods

Sediment samples (n=45) were collected west of Spitsbergen and on the adjacent shelf to the south on a scientific cruise with "RV Heincke" in summer 2001. Short cores were either taken with a multicorer or with a boxcoring device (Table 1). The surface sediment of all short cores (first centimeter of core depth) were sampled onboard and stored at −20 °C for further analysis onshore. Sediment samples were freeze-dried and one part was ground for geochemical analysis while the remainder was used for mineralogical analysis [21].

One aliquot of each sediment sample was analysed for the bulk nitrogen content (N_{tot} in wt.%). A second aliquot was treated with KOBr–KOH solution to remove organic nitrogen (N_{org}) and to measure the amount of inorganic nitrogen (N_{inorg} in wt.%) bound as ammonium to the fine-grained sediments [22,23].

The KOBr–KOH treatment of soil samples removes in excess of 98% of the organic nitrogen fraction. This value was determined on different soil samples with variable content of total organic carbon (0.8–10%), total nitrogen (0.9–0.08%) and clay (42.7–19.8%) [22]. It is mainly based on the difference of total nitrogen content determined by a modified Kjeldahl method [24] including the pretreatment of soil samples by HF–H_2SO_4 solution to decompose silicate minerals and release fixed ammonium, and the summation of exchangeable and fixed ammonium according to the method described in Silva and Bremner [22]. Stevenson and Cheng [25] and Müller [23] have shown using independent analytical methods that this approach is reliable for the recovery of fixed inorganic ammonium in marine sediments. The approach has been further developed and applied in various marine environments for both paleoclimatic research [11,21,26] and diagenetic processes [5,27,28] (see "Supplementary data" for further information).

Total N and $\delta^{15}N$ were analysed by elemental analyser isotope ratio mass spectrometry (EA-IRMS) on an ANCA-GSL/20–20 system (Europa Scientific, Crewe, UK). Approximately 20% of the samples were analysed in duplicate with a mean standard deviation of 0.16‰. Results in Table 1 are standard δ-values in the per mil (‰ vs. air). The reference material used during analysis of the samples (IA-R001 wheat flour, Iso-Analytical Ltd.) had a $\delta^{15}N$ value of 2.55‰ vs. air and contained 1.88% N (w/w). Control samples were analysed to check the accuracy of the measurements. The precision for treated and untreated samples was better than ±0.2‰.

N_{org} content and $\delta^{15}N_{org}$ values were calculated from the measured amounts of N_{tot} and N_{inorg} and isotopic values $\delta^{15}N_{tot}$ and $\delta^{15}N_{inorg}$ using a simple isotope mass balance [9]. The error in determining the $\delta^{15}N_{org}$ values including processing, measurement, and calculation was estimated to be better than ±2.1‰.

Table 1
Geochemical data from surface samples

Station	Gear	Latitude	Longitude	Water Depth (m)	TOC (wt.%)	N_{tot} (wt.%)	N_{inorg} (wt.%)	N_{org} (wt.%)	N_{org} (% of total)	$\delta^{15}N_{tot}$ (‰)	$\delta^{15}N_{inorg}$ (‰)	$\delta^{15}N_{org}$ (‰)	Rock Eval Tmax (°C)	Illite (rel.%)	Chlorite (rel.%)	Smeetite (rel.%)
1240	Box core	74.48.87 N	19.10.77 E	94	0.53	0.065	0.017	0.049	74.7	6.07	5.99	6.09	415			
1241	MUC	74.49.08 N	17.34.54 E	297	2.14	0.292	0.046	0.245	84.2	5.08	4.65	5.16	416	43.4	26.7	0
1242	MUC	75.30.18 N	13.19.97 E	1297	1.07	0.163	0.028	0.135	82.8	6.03	4.47	6.35	417	47	28.8	0
1243	MUC	75.59.95 N	16.35.40 E	333	1.82	0.229	0.051	0.177	77.5	5.30	4.29	5.59	427	40	31.6	0
1244	MUC	77.56.95 N	19.09.98 E	96	2.17	0.242	0.102	0.141	58.1	4.56	3.10	5.61	424	43.7	26.7	0
1245	MUC	77.29.97 N	19.07.80 E	180	2.37	0.271	0.099	0.171	63.3	5.06	3.52	5.95	420	46.1	32.4	0
1246	MUC	76.46.10 N	19.26.45 E	153	2.07	0.228	0.087	0.142	62	4.69	3.48	5.44	422	47.5	32.9	0
1249	MUC	76.57.06 N	15.15.07 E	156	1.91	0.226	0.082	0.144	63.6	4.86	3.77	5.48	418	49.3	36.1	0
1250	MUC	76.59.04 N	15.45.46 E	228	1.65	0.179	0.080	0.099	55.5	4.76	3.49	5.77	445	56.9	38	0
1251	MUC	77.45.03 N	14.55.09 E	115	1.84	0.172	0.089	0.083	48.1	4.50	3.33	5.76	420	55	33.5	0
1254	MUC	77.50.09 N	16.35.27 E	76	2.03	0.137	0.096	0.041	30.2	4.36	3.32	6.77	428	49.3	33	0
1255	MUC	77.43.19 N	15.11.25 E	83	1.74	0.154	0.087	0.068	43.9	4.63	3.78	5.71	427	58.7	30.1	0
1258	MUC	77.49.72 N	15.41.61 E	43	1.72	0.135	0.085	0.050	36.9	3.94	3.39	4.89	426	53.1	32.8	0
1260	MUC	77.38.01 N	14.12.56 E	162	1.77	0.234	0.078	0.156	66.6	5.00	3.76	5.63	412	60.1	33.7	0
1261	MUC	77.23.38 N	10.35.84 E	1291	0.89	0.118	0.039	0.079	67.2	5.64	3.32	6.77	429	60.2	23.6	0
1262	MUC	77.21 56 N	11.16.30 E	603	0.82	0.087	0.036	0.051	58.5	5.50	3.65	6.81	437	44.9	30.1	0
1263	MUC	77.11.96 N	12.55.67 E	196	1.96	0.239	0.079	0.159	66.7	5.09	3.97	5.66	416	45.9	37.7	0
1264	Box core	77.32.11 N	12.36.29 E	103	1.53	0.191	0.058	0.133	69.5	4.98	3.86	5.47	424	48.2	39.7	0
1265	MUC	78.22.00 N	16.21.95 E	87	1.88	0.166	0.078	0.088	53.1	4.89	3.54	6.08	431	50.2	22	0
1266	MUC	78.21.89 N	15.15.51 E	256	2.24	0.280	0.083	0.198	70.6	5.00	3.40	5.67	418	56.9	28.9	0
1267	MUC	78.09.00 N	13.50.01 E	416	2.68	0.366	0.079	0.287	78.3	4.54	3.83	4.74	413	53.5	35.5	0.1

1268	MUC	78.37.47 N	11.38.27 E	102	1.19	0.187	0.071	0.116	61.9	4.66	3.54	5.35	399	64.8	35.2	0
1269	MUC	78.22.02 N	12.18.27 E	169	2.00	0.249	0.088	0.161	64.7	5.24	3.69	6.08	421	60.9	36.9	0
1270	MUC	78.04.98 N	12.17.99 E	259	2.41	0.313	0.090	0.223	71.2	4.80	3.32	5.4	409	53.8	35.3	0
1272	MUC	78.15.15 N	08.49.28 E	1400	1.20	0.155	0.045	0.110	70.8	5.46	3.80	6.15	425	52.8	21.7	6.1
1273	MUC	78.15.13 N	09.18.99 E	600	0.85	0.081	0.040	0.041	50.7	4.47	3.67	5.25	434	43.9	31.6	2.3
1274	Box core	78.15.00 N	09.23.59 E	430	0.89	0.097	0.045	0.05 1	53	4.45	4.03	4.82	434	53.5	29.9	0
1275	MUC	78.14.97 N	10.12.57 E	297	2.21	0.264	0.075	0.188	71.4	4.32	3.91	4.48	426	53.9	32.7	0
1276	Box core	78.34.02 N	10.20.19 E	131	2.14	0.306	0.054	0.252	82.2	4.75	4.45	4.82	417	59	34.1	0
1277	MUC	78.34.91 N	09.26.33 E	404	1.44	0.179	0.051	0.128	71.5	4.67	4.56	4.72	417	53.1	34.7	0
1278	MUC	78.35.01 N	09.01.77 E	601	1.13	0.133	0.043	0.089	67.4	4.81	4.52	4.96	430	51.7	30.7	0
1279	MUC	78.36.58 N	07.49.41 E	1203	1.36	0.176	0.047	0.129	73.3	5.19	3.77	5.71	426	48.4	31.6	0
1280	MUC	78.36.45 N	08.41.75 E	787	1.31	0.161	0.052	0.109	68	5.36	4.23	5.89	428	49.3	32.5	0
1281	Box core	78.43.12 N	09.49.81 E	106	1.29	0.174	0.040	0.134	77.2	4.59	4.96	4.48	418	58.7	31.9	0
1282	MUC	78.56.87 N	06.49.01 E	1400	1.18	0.161	0.046	0.115	71.6	5.54	4.36	6.01	418	53.4	24.7	0
1284	MUC	78.56.96 N	08.26.18 E	604	0.93	0.106	0.061	0.045	42.4	4.91	3.70	6.55	429	52.5	32	0
1285	Box core	78.49.45 N	09.37.33 E	91	1.96	0.223	0.060	0.163	73	4.37	3.99	4.52	419	55.6	34.9	0
1286	MUC	78.52.04 N	11.19.97 E	159	1.13	0.165	0.043	0.122	73.7	4.74	3.85	5.05	413	60.7	35.5	0
1287	MUC	79.10.09 N	11.45.94 E	364	0.97	0.133	0.027	0.106	79.8	5.17	6.26	4.9	402	79.5	16.7	0
1288	MUC	78.58.83 N	11.49.36 E	308	1.38	0.191	0.044	0.147	77.2	4.89	4.58	4.98	410	69.6	20.2	0
1289	MUC	79.01.98 N	10.50.10 E	319	2.23	0.290	0.067	0.222	76.7	4.33	3.69	4.53	412	68.3	24.2	0
1290	MUC	78.57.00 N	09.37.25 E	250	0.95	0.125	0.028	0.097	77.6	4.69	3.51	5.03	410	52.9	37.1	0.3
1292	Box core	79.03.48 N	09.19.72 E	80	1.51	0.208	0.037	0.171	82.2	4.71	4.73	4.7	413	61.3	29.3	0

Data published in Schubert and Calvert [9] are not included.

Table 2
Exchangeable nitrogen in percent of the total available nitrogen of marine and limnic sediments

Marine sediments				Lake sediments			
Namibia (cm)	$\%N_{KCl}$	Chile (cm)	$\%N_{KCl}$	Lake Zug (cm)	$\%N_{KCl}$	Lago di Cadagno (cm)	$\%N_{KCl}$
0–1	0.05	0–1	0.28	0–1	1.03	0–1	0.78
4–5	0.04	4–5	0.18	4–5	1.37	4–5	1.65
8–9	0.01	8–9	0.14	8–9	1.40	8–9	0.89
14–16	0.04	14–16	0.14	14–16	0.73	14–16	1.09
18–20	0.02	18–20	0.12	18–20	0.88	18–20	0.80
22–24	0.01	22–24	0.16	22–24	0.96	22–24	0.48
Latitude 25°31′ S	Longitude 13°02′ E	Latitude 36°32′ S	Longitude 73°03′ W	Latitude 47°10′ N	Longitude 8°29′ E	Latitude 46°33′ N	Longitude 08°43′ E

To estimate the possible influence of exchangeable nitrogen that is bound to clay mineral surfaces, we measured this fraction in lake sediments (Lake Zug and Lago di Cadagno, Switzerland) and marine sediments (Chile and Namibia) (Table 2). Sediments were extracted with 2 M KCl to release the ammonium bound to the surface of clay minerals. NH_4^+ was analysed using the phenolhypochlorite method on a "Merck SQ300" photometer [29]. Concentrations of exchangeable nitrogen varied from 0.01 to 1.7% of the total nitrogen, lower values being associated with marine samples (average 0.03 to 0.9%) and higher values with lake samples (0.5 to 1.7%). These results indicate that the exchangeable nitrogen fraction represents a minor fraction, and hence this fraction was not further considered in this research. Our results confirm the results by Müller [23] and Freudenthal et al. [28], who showed that exchangeable nitrogen is below 5 and 1% of the total nitrogen, respectively.

4. Results and discussion

4.1. The spatial variability of organic and inorganic nitrogen

The total nitrogen concentration (N_{tot}) in continental margin sediments off Spitsbergen varies between 0.05 and 0.37 wt.% (Fig. 2A). The distribution of N_{tot} shows no distinct pattern with relatively high and low values are present in fjords and on shelf areas (Fig. 2A). However, generally high N_{tot} are characteristic for the Isfjorden and Storfjorden areas, while the lowest N_{tot} are found on the shelf/slope off W-Spitsbergen.

The distribution of the inorganic nitrogen (N_{inorg}), which is mainly lattice-bound ammonium (NH_4^+) in clay minerals [23,30,31], is clearly influenced by the distance to shore. Concentrations are highest (up to 0.1 wt.%) in the fjord areas but steeply decrease towards open marine or shelf environments (Fig. 2B). The concentration of inorganic nitrogen correlates negatively with the $\delta^{13}C$ values of the organic matter ($R^2=0.65$) (Fig. 3), implying that fixed ammonium in surface sediments is considered to be land-derived (see also [9,28]). Low $\delta^{13}C$ values also indicate enhanced supply of TOM [21]. Both inorganic nitrogen (Fig. 2B) and terrestrial organic carbon [21] are particularly abundant in the more vegetated central and southern Spitsbergen fjords, while low concentrations are found in the more glaciated northwestern fjords, the Kongs- and Krossfjorden. The low concentrations of organic carbon and inorganic nitrogen (Fig. 2B) in sediments from the northwestern fjords may be attributed to variations in the sediment matrix and/or clay mineral assemblages, the physiogeographic settings, the hinterland geology and/or the catchment areas and are discussed below.

Numerous studies of soils indicate that ammonium ions are preferentially fixed in illites [32,33]. Illite is the dominant clay mineral (on average accounting for 54% of the total clay fraction) in all surface sediments around Spitsbergen (Table 1). However, highest illite abundances (up to 79%) are found in surface sediments with low concentrations of inorganic nitrogen prevail, namely in the Kongs- and Krossfjorden areas (Table 1, Fig. 2B). This lack of correlation suggests two possibilities. First, illite is not the sole carrier of ammonium. Other matrix-supported processes or carriers such as K-feldspars or zeolites may also bind ammonium [27]. Unfortunately, data supporting this assertion are currently not available for the study area. Second, the proportionality between illite and inorganic nitrogen is expected if all the illite entering the system contains the same concentration of ammonium, a rather unrealistic assumption considering the variable bedrock/ecosystem types present on Spitsbergen. The heavily glaciated, sparsely vegetated hinterland with predominantly metamorphic strata in the Kongs- and Krossfjorden catchment area [34] is less likely to provide significant amounts of inorganic nitrogen-bearing soil

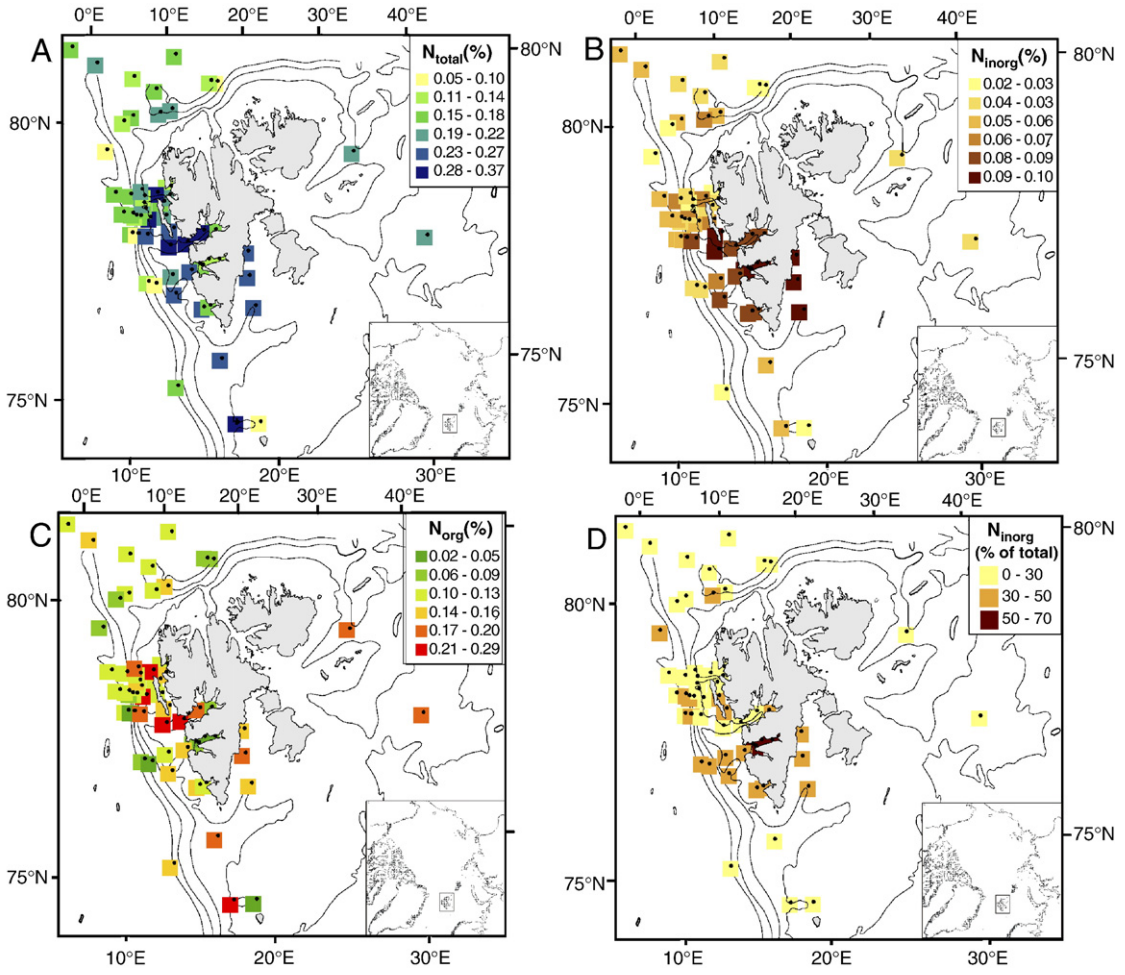

Fig. 2. Distribution patterns of (A) total nitrogen concentration (N_{tot} wt.%), (B) inorganic nitrogen concentration (N_{inorg} wt.%), (C) organic nitrogen concentration (N_{org} wt.%), and (D) percentages of inorganic nitrogen (% N_{inorg}) of the total nitrogen content in surface sediments off Spitsbergen.

material to the marine environment than are the more vegetated areas in central and southern Spitsbergen. This is reflected by the low abundance of TOM (<25% of total organic carbon input) in the north and high values (>60%) in the south [21]. All this suggests that the inorganic nitrogen of marine sediments off Spitsbergen is composed of the inputs from soil (terrestrial) organic matter from rivers, coastal erosion and sea ice. This is also supported by the correlation between low $\delta^{13}C$ values of the organic matter – indicative for enhanced terrestrial organic matter supply [21] – with relatively high proportions of inorganic nitrogen (Fig. 3). The substrate in all fjord settings typically consists of fine-grained, soft sediments, so differences in glacimarine sedimentation in proximal settings may not be responsible for the variations in inorganic nitrogen supply.

The organic nitrogen (N_{org}) content as determined by the difference between N_{tot} and N_{inorg} significantly

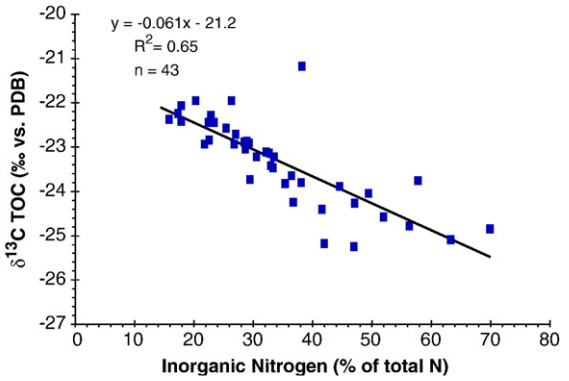

Fig. 3. Crossplot between the inorganic nitrogen content (%) and $\delta^{13}C$ of total organic carbon, which is a proxy for the terrigenous fraction of the organic matter [21].

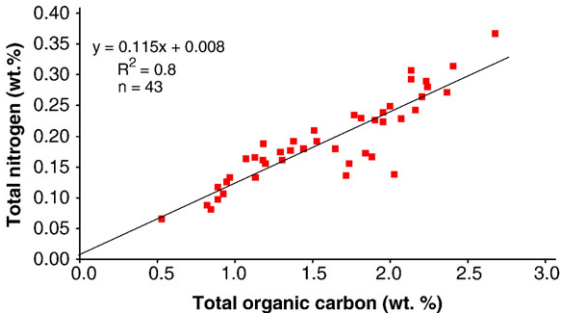

Fig. 4. Crossplot between total organic carbon (wt.%) and total nitrogen (wt.%) contents in surface sediments off Spitsbergen.

varies (0.02–0.3 wt.%) and shows no distinct distribution pattern (Fig. 2C). Generally, higher values occur at fjord heads and in the central and southern Barents Sea, while lower values are found along the upper slope, in van Mijenfjorden, and towards northern Spitsbergen and the Yermak Plateau (Fig. 2C). However, when the N_{inorg} percentages of the total nitrogen content (Fig. 2D) are inspected, the large fluctuations indicate that dilution of the organic signal by land-derived inorganic nitrogen is most observed in van Mijenfjorden and Storfjorden and their adjacent outlets (Fig. 2D). Minimum N_{inorg} proportions of total nitrogen, and thus maximum N_{org} proportions, occur south off Spitsbergen, where they may reflect the predominance of the Atlantic Water inflow, and across the Kongs- and Krossfjorden trough, where they may be the result of a topographically steered upwelling via vertical mixing (Fig. 2D) [20].

The substantial but variable contribution of inorganic nitrogen to the total nitrogen in the study area is not detectable in an N_{tot} versus TOC plot (Fig. 4). Plots such as this are, however, widely used to discriminate between the organic and inorganic fractions of nitrogen [11,14,35]. Usually, a large contribution of N_{inorg} to N_{tot}

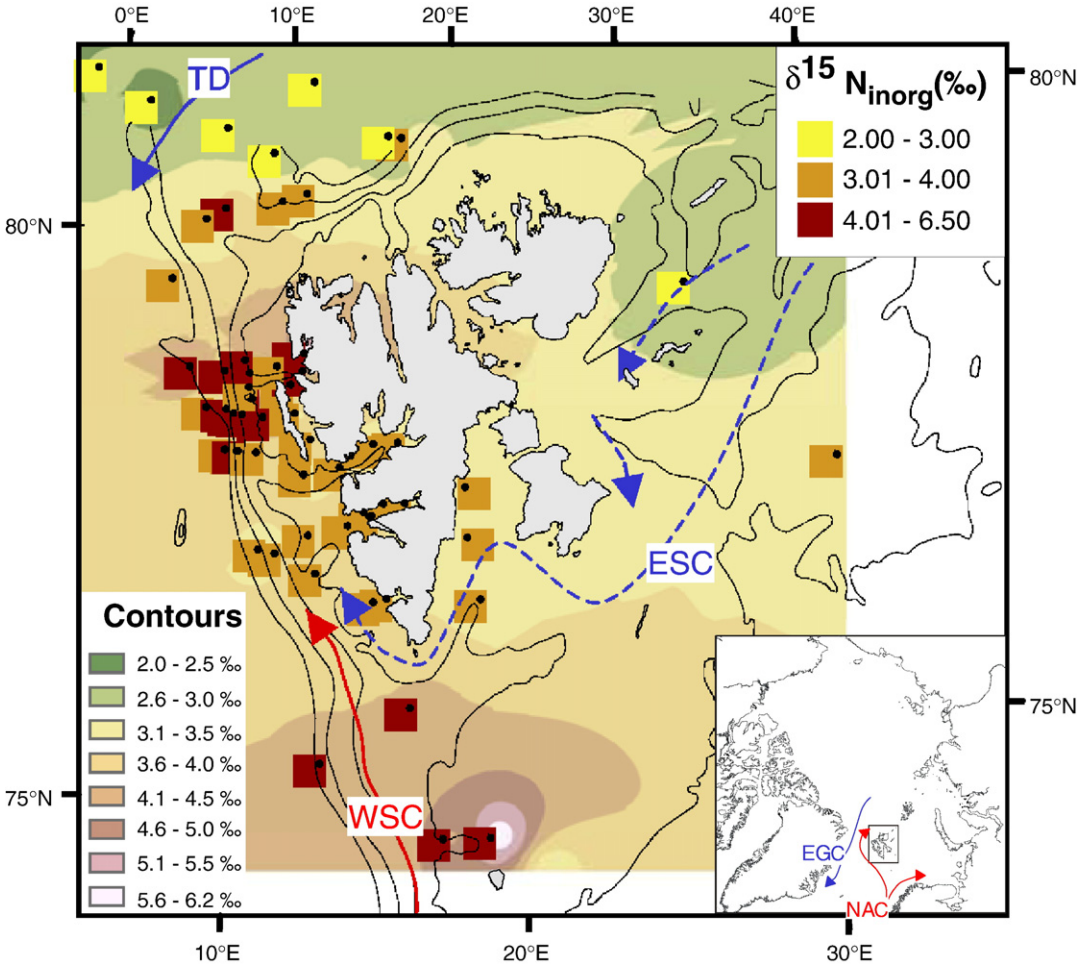

Fig. 5. $\delta^{15}N$ of inorganic nitrogen (‰) in continental margin sediments off Spitsbergen. Major surface water currents in the study area around Spitsbergen are marked (see abbreviations in Fig. 1).

is often inferred from nonzero intercept in plots of N_{tot} versus TOC. Here, the regression is not significantly different from zero implying a small contribution of N_{inorg} to the total signal (Fig. 4). This is obviously not the case for Spitsbergen sediments and may justify a re-evaluation of this approach. Recently, Calvert [35] stated that the intercept on the N axis is often the case when organic carbon and organic nitrogen show close proportionality while the inorganic nitrogen is devoid of carbon. Considering the close correspondence of the $\delta^{13}C$ isotopic composition of organic matter and inorganic nitrogen content (Fig. 3) in our data set, a proportionality between *both* the marine (marine organic carbon and N_{org}) and the terrigenous (terrigenous organic carbon and N_{inorg}) C–N components is more than likely and may explain the zero-intercept of N_{tot} vs. TOC plot. Hence, this may call for inspection of the various nitrogen fractions before interpretation (i.e. C/N ratios or nitrogen isotopic signatures) at least for continental margin sediments with known TOM supply.

4.2. The $\delta^{15}N$ of inorganic ammonium nitrogen: response to climate-induced changes in soil organic matter?

The $\delta^{15}N_{inorg}$ value of marine surface sediments is generally assumed to be equivalent to the inorganic ammonium nitrogen signal obtained on land (e.g. [9,28]). First studies of Arctic Ocean surface sediments show narrow ranges of $\delta^{15}N_{inorg}$ values with an average of 3.0±1.2‰ [9]. Surprisingly, the $\delta^{15}N_{inorg}$ value of surface sediments in this study shows a wide and regular variation with relatively low (2.0–3.0‰) values in the north, intermediate (3.0–4.0‰) values adjacent to the main fjord heads and troughs west off Spitsbergen, and high (4.5–6.0‰) values in the south (Fig. 5).

Although the relation between $\delta^{15}N_{inorg}$ in marine and bulk $\delta^{15}N$ in terrestrial ecosystems is not well known, the strong gradient in the isotopic signal may be linked to various terrestrial biomes. Amundson et al. [15] noted from a compilation of worldwide $\delta^{15}N$ values in soil organic matter that there is a systematic decrease of $\delta^{15}N$ values with decreasing mean annual temperature and (less significantly) with increasing mean annual precipitation (see Fig. 2 in [15]). $\delta^{15}N$ measurements of recent soil (upper 50 cm) are lowest within boreal ecosystems [15]. While climate plays a significant role in global $\delta^{15}N$ patterns in soils, the mechanisms behind these patterns are not fully understood. A likely reason could be variations in isotopic fractionation due to climate-dependent differences in the isotopic composition of nitrogen leaving the system [15,36]. Particularly, the greater loss of ^{15}N-depleted forms of nitrogen (e.g. NO_3^-, N_2O) relative to total soil nitrogen may explain the systematic increase of $\delta^{15}N$ in soil organic matter with globally decreasing mean annual precipitation and increasing mean annual temperature, respectively [15]. Another possibility could be spatial variations in the $\delta^{15}N$ isotopic signature of atmospheric nitrogen input [15].

Our data suggest that, similar to the $\delta^{15}N$ in soils, the isotopic signal of land-derived N_{inorg} preserved in marine sediments may provide insight into systematic changes in the supply of material derived from specific ecosystem types and thus indicate a close relationship to climate zones. This assumption requires that during transfer of nitrogen from continents to coastal oceans the bulk soil $\delta^{15}N$ value within the respective climate zone be preserved in clay minerals deposited in marine sediments, a question that remains unresolved. Considering that in terrestrial ecosystems the biologically fixed nitrogen is converted to organic nitrogen within the fixing organisms, and lost from the organism as reduced inorganic nitrogen or converted to NH_4^+ by mineralisation of dead organic matter [37] (as long as nitrogen stays in its reduced form (NH_4^+)), we speculate that it remains in the local environment because of its affinity for soil adsorption and its rapid uptake by biota. Austin and Vitousek [38] showed in soils from Hawaii that despite isotopic fractionation during mineralization of NH_4^+, nitrification, denitrification, and ammonia volatilization [39], the general pattern of decreasing soil $\delta^{15}N$ with increasing annual precipitation in Hawaii prevailed, suggesting that no competing processes within various nitrogen pools exist. Koba et al. [40,41] further showed that ranges of $\delta^{15}N$ of total N (1–6.8‰) and NH_4^+ (2.5–15‰) are similar in forest mineral soils in Japan and generally increase with soil depth. This implies that inorganic ammonium nitrogen bound in clays may be useful for tracing bulk $\delta^{15}N$ in soil. However, given the uncertainties from this limited set of data, we can only speculate that erosional processes and river transport supply the adsorbed fraction of inorganic ammonium to the marine environment leading to a land-derived isotopic signal being preserved in marine sediments.

The lowest $\delta^{15}N_{inorg}$ values found in the study area are limited to the summer-ice covered areas in the north and northeast of Spitsbergen. As is widely known from previous studies [14], the carrier of terrestrially-derived N_{inorg} in these areas are the sediments transported by sea ice via the Transpolar Drift and released along the ice-edge north of Spitsbergen. The organic matter within these sediments is dominated by terrestrial organic

carbon [14]. The sediments released along the ice edge still contain between 0.15 and 1 wt.%, making a contribution of 40–60% of TOM to the total organic carbon [14]. This suggests that significant amounts of soil material – consistent with 0.04–0.06% N_{inorg} in the sediment (Fig. 2B) – derived from northern Siberian coastal plains and tundra reach the study area by lateral sea ice processes. This interpretation is consistent with $\delta^{15}N$ values of northern Siberian soils and $\delta^{15}N_{inorg}$ values in our sediments. Low values with very narrow ranges (1.0–3.0‰) (Fig. 5) (Fig. 2 [15]) in both areas support the hypothesis that the terrestrial organic material may be derived from northern Siberia.

The $\delta^{15}N_{inorg}$ signals in the southernmost samples of the study area are significantly heavier (4.5–6.0‰) than in the north (Fig. 5). This area is strongly influenced by northward flowing AW-derived water masses (Fig. 1) suggesting that the land-derived isotopic signal in the underlying sediments is derived from more southerly or easterly sources. Indeed, cascading of dense water in the Barents Sea is an important process known for transporting terrigenous sediments from the shallow Barents Sea shelf to the adjacent continental margin [42,43]. Moreover, in the vicinity of northward flowing AW-derived water masses, internal wave erosion processes along the slope and sediment winnowing further control sediment import from the south into the study area [43,44]. The land-derived isotopic signal is therefore not source-specific as described for the northern sample set, but possibly reflects a composite signal derived from various sources (fresh and reworked). In fact, the narrow $\delta^{15}N_{inorg}$ ranges in our data set (4.0–6.0‰) suggest potential source areas in the eastern North Atlantic and North Sea, where $\delta^{15}N$ values of soil material are in the same range [15].

Sedimentary $\delta^{15}N_{inorg}$ values in the central part of the study area are mainly influenced by the input of soil material from coastal Spitsbergen. This is clearly shown by the distribution patterns of N_{inorg} concentration in the sediments (Fig. 2B), and also by the intermediate $\delta^{15}N_{inorg}$ values (3.0–4.0‰) (Fig. 5). The latter are consistent with the $\delta^{15}N$ values in soil material on Spitsbergen [15] and may indicate that currents and sea ice may transport material originally deposited in fjords by coastal erosion, meltwater and riverine discharge onto the shelf. Hence, the lateral transport of TOM from the hinterland dilutes the $\delta^{15}N_{inorg}$ signal supplied by the Atlantic water inflow and decreases sedimentary

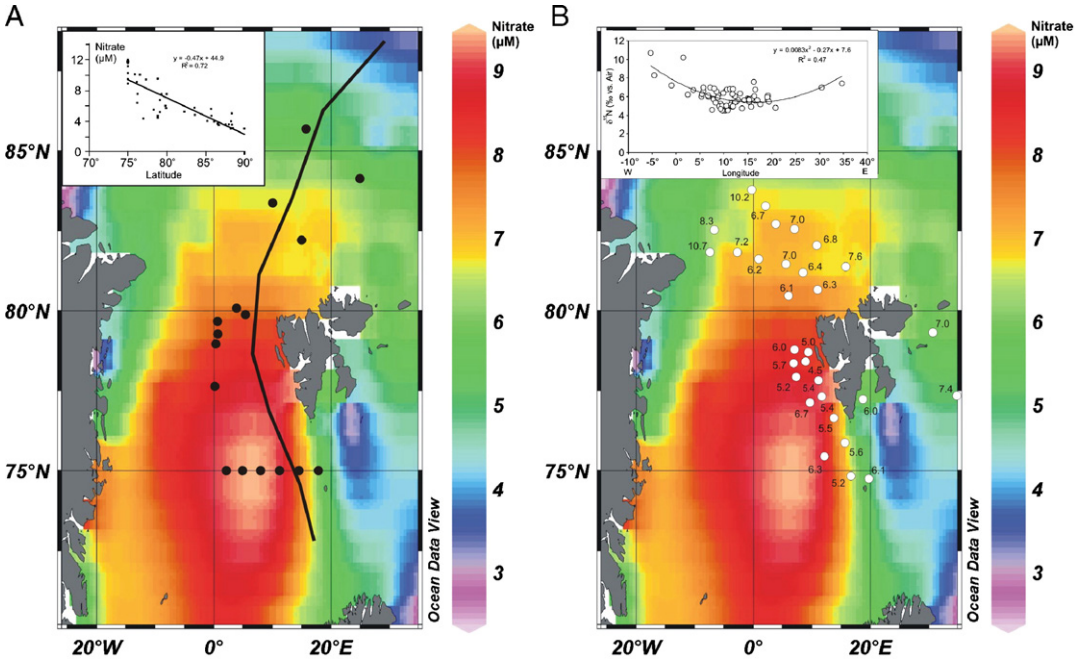

Fig. 6. (A) Map shows dissolved nitrate concentrations (µM) in 30 m water depths taken from the World Ocean Database [46]. Black dots indicate stations where measured dissolved nitrate concentrations (µM/kg) from various investigators (Leif Anderson, University of Göteborg, Eva Falck, University of Bergen) and years/seasons (1993–2004) are available. The respective data in the inset shows the positive correlation between decreasing nitrate concentration and minor influence of nitrate-rich Atlantic water towards the north. (B) Map with dissolved nitrate concentrations (µM) in 30 m water depths taken from the World Ocean Database [46] and $\delta^{15}N_{org}$ values in selected surface samples. The inset shows the distribution patterns of $\delta^{15}N_{org}$ along an East–West transect (5° W–35° E) from Greenland towards the Barents Sea.

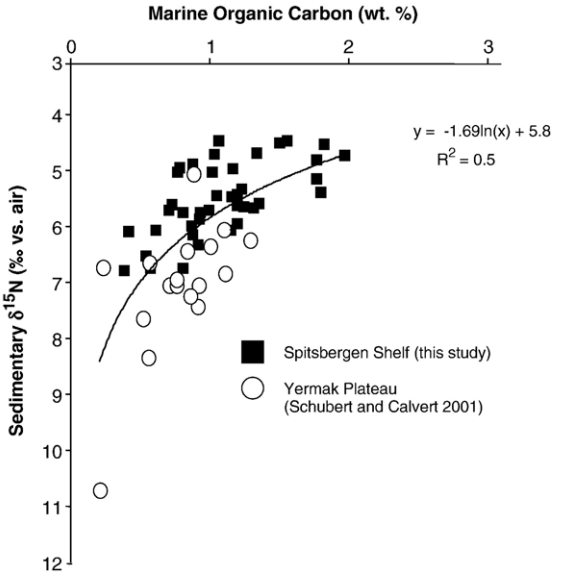

Fig. 7. A crossplot of $\delta^{15}N$ of organic nitrogen (‰) and marine organic carbon (wt.%) in continental margin sediments off Spitsbergen (this study) and the Yermak Plateau/Fram Strait [9].

$\delta^{15}N_{inorg}$ values. Higher $\delta^{15}N_{inorg}$ values towards the north, namely in the Kongs- and Krossfjorden troughs, may be caused by a lower influence of the hinterland signal due to the minimal supply of inorganic nitrogen (Fig. 2B). This correlates with a lower supply of TOM and a predominance of primary-produced organic carbon in the underlying sediments [21].

Accordingly, we suggest that $\delta^{15}N_{inorg}$ values in marine surface sediments from the Arctic gateway are closely related to TOM supply from various climatic- and ecosystem zones and may be source-specific tracers for climatically induced changes in catchment vegetations in high northern latitudes. Existing core data from the middle (NW Africa) to lower (South China Sea) latitudes [11,28], however, show average $\delta^{15}N_{inorg}$ values of ~3–6‰, which is significantly lighter than would be expected from the data set of soil $\delta^{15}N$ presented by Amundson et al. [15]. This suggests that factors other than climate (soil $\delta^{15}N$) influence the marine $\delta^{15}N_{inorg}$ signal. However, results from NW African appear to be hardly influenced by TOM, and are characterized by constantly low (<0.02%) amounts of inorganic nitrogen, and shows less variable $\delta^{15}N_{inorg}$ values (5–6‰), which is typical for the mid-latitudes [15]. The fact that the terrigenous background signal is mainly detrital and finely dispersed in the sediment matrix underlines the possibility of long distant transport. Previous studies indicate that TOM is mainly brought to NW African margin via eolian fallout and that river influence can be excluded [45]. The latter may indicate an admixture of aeolian transported TOM from various sources, owing to the influence of the North East trade winds in the region, which would explain the slightly deviating $\delta^{15}N_{inorg}$ marine data from the soil $\delta^{15}N$ signal of the hinterland (6–7‰). A plausible explanation for the differences between the $\delta^{15}N_{inorg}$ core data in the South China Sea (3.1–4.8‰) and modern soil $\delta^{15}N$ in the catchment area (5–7.5‰) can at this stage not be provided. A randomly distributed set of surface sediments samples must be studied first, before the approach of tracking various terrestrial biomes by $\delta^{15}N_{inorg}$ marine data may be verified in lower to middle latitudes.

4.3. The $\delta^{15}N$ value of organic nitrogen: tracking nutrient utilization and Atlantic water inflow

Schubert and Calvert [9] introduced the use of the $\delta^{15}N_{org}$ signal in Arctic Ocean surface sediments instead of $\delta^{15}N_{tot}$ as an indispensable tool for tracing different relative phytoplankton nitrate utilization rates in various regions of the Arctic Ocean. In contrast to the $\delta^{15}N_{tot}$ signal, the regional pattern of $\delta^{15}N_{org}$ values is not influenced by the admixture of ^{15}N depleted TOM and is thus better resolved. This is consistent with our results, which indicate slightly larger ranges for $\delta^{15}N_{org}$ (~4.5–7‰) values than for $\delta^{15}N_{tot}$ values (~4–6‰) (Table 1). Schubert and Calvert [9] confirmed that a significant fraction of the variance in the $\delta^{15}N_{org}$ values in these sediments is due to changes in nitrate concentration. Denitrification, in contrast, is very unlikely in this well-oxygenated setting and should not have affected the sedimentary $\delta^{15}N_{org}$ signal.

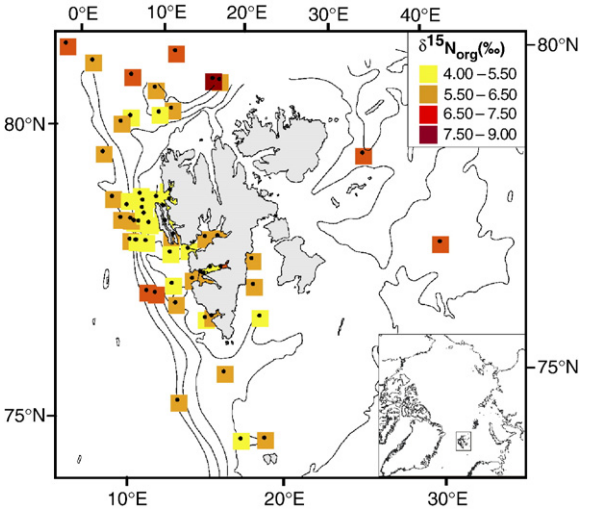

Fig. 8. $\delta^{15}N$ of organic nitrogen (‰) in continental margin sediments off Spitsbergen.

A compilation of dissolved nitrate concentrations [NO_3^-] in surface waters (0–50 m) found in the World Ocean Database [46], and other data provided by various investigators (Leif Andersen, University of Göteborg; Eva Falck, University of Bergen) show a good correlation between Atlantic Water influenced areas with relatively high nitrate concentrations and the gradual decrease and enhanced utilization by phytoplankton towards the ice-covered Arctic Ocean (Fig. 6A). The $\delta^{15}N_{org}$ pattern in surface sediments generally mirrors the differences in oceanic current systems with decreasing influence of Atlantic water masses towards the north and northeast (Fig. 6B). Values within the core of nitrate-rich Atlantic Water inflow vary between 4.0 and 7.0‰, which is consistent to the source $\delta^{15}N$ nitrate (~4.5‰) [47], while towards the nitrate-depleted polar domains in the east (East Spitsbergen Current) and west (East Greenland Current), significantly higher values (7.0–10.0‰) occur (Fig. 6B). These gradients indicate, as in the central Arctic Ocean [9], that the cumulative effect of NO_3^- uptake in less replenished Arctic waters with the remaining substrate that is enriched in $^{15}NO_3^-$, result in higher $\delta^{15}N_{org}$ values in the polar regions. Hence, considering only the organic signal in the sediments, a strong relationship between surface water NO_3^- depletion and an increase in $\delta^{15}N_{org}$ values, as clearly demonstrated for the North Atlantic [48,49], is also postulated for the gateway into the Arctic Ocean. The positive trend between marine organic carbon content and $\delta^{15}N_{org}$ (Fig. 7) further indicates a relationship between primary-produced organic matter and availability of nitrate in surface waters. This strengthens the theory that the $\delta^{15}N_{org}$ signal in high northern latitudes is appropriate for tracing past nutrient utilization in Arctic Ocean sediments [9].

Diagenetic changes in particulate $\delta^{15}N$ values in the water column during particle settling and in the sediment after burial (e.g. [1,8,28]) have not been determined in the study area. However, there are no systematic $\delta^{15}N_{org}$ shifts with increasing water depth (Fig. 8) suggesting that diagenetic modifications are minimal. This agrees with observations of little diagenetic fractionation during burial in shelf and slope sediments [50], although, in the absence of $\delta^{15}N_{org}$ values in sinking particles from the study area, a diagenetic overprint of at least 1‰ (as known from the sediment–water interface in various marginal environments (e.g. [28])), cannot be excluded.

Another factor that might influence the $\delta^{15}N_{org}$ distribution patterns from south to north is erosion and out-washing of fine-grained material by bottom currents at the sediment surface along the shelf edge. The occurrence of winnowed lag deposits on the outer shelf, which represent erosional surfaces, may have led to the sampling of non-representative surface samples (cf. [21]) and thus may have produced some artefacts. This might explain the few high $\delta^{15}N_{org}$ values (~6.8‰) along the shelf break west off Spitsbergen (stations 1261, 1262) (Fig. 8). Moreover, a topographically steered local upwelling center in the Kongsfjorden trough may control a steady replenishment of nitrate-rich Atlantic Water in the euphotic zone leading to a low relative nutrient utilization and thus to lowest $\delta^{15}N_{org}$ values in the underlying sediments (Fig. 8).

5. Conclusions

A comprehensive set of continental margin sediments off Spitsbergen was analysed for the concentrations and isotopic composition of organic and inorganic nitrogen. The results show that up to 70% of the bulk nitrogen can be composed of bound inorganic nitrogen. The variation in the inorganic contribution to the total nitrogen is potentially the result of varying supplies of terrestrial organic matter (TOM), mainly eroded soil material from the adjacent hinterlands, and is not strictly related to specific clay mineral assemblages as previously assumed. The spatial heterogeneity of inorganic nitrogen isotope ratios, which represent a land-derived signal from various sources, suggests a strong relationship to various climate-dependent ecosystem zones and may be a useful proxy for reconstructing changes in the catchment's vegetation. The $\delta^{15}N_{org}$ signal in surface sediments shows a relationship to the various oceanic front systems in the Atlantic–Arctic gateway and is appropriate for tracing relative nutrient utilization by phytoplankton. A positive correlation between the $\delta^{15}N_{org}$ value and marine organic carbon input suggests that the signal is potentially useful for reconstructing primary productivity changes in the sedimentary record.

This re-assessment of nitrogen measurements in continental margin sediments leads us to conclude that, in this and probably other environments with significant TOM supply, analysis of bulk nitrogen alone is not sufficient to obtain a comprehensive characterization of the nitrogen cycle. Especially in palaeoceanographic and -climatic studies it seems necessary to include measurements of the concentration and isotopic composition of all nitrogen fractions (organic, inorganic, exchangeable).

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.epsl.2006.11.008.

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