

**Variations in organic
matter bound in fluffy
layer suspended matter
from the Pomeranian Bay
(Baltic Sea)***

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KEYWORDS

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KSENIA PAZDRO¹

ANDRZEJ STANISZEWSKI¹

JACEK BELDOWSKI¹

KAY-CHRISTIAN EMEIS²

THOMAS LEIPE²

JANUSZ PEMPKOWIAK¹

¹Institute of Oceanology,
Polish Academy of Sciences,
Powstańców Warszawy 55, PL-81-712 Sopot, Poland;
e-mail: pazdro@iopan.gda.pl

²Baltic Sea Research Institute,
Seestrasse 15, 18119 Rostock-Warnemünde, Germany

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Abstract

Fluffy layer suspended matter (FLSM) and surface sediment samples from the Pomeranian Bay were examined for fatty acid, lipid and organic matter contents. FLSM is a several-centimetre-thick layer of fairly concentrated particulate matter lying on the sea floor significantly affecting the flux of matter to depositional basins. Analyses of fatty acids were used to establish sources and decomposition rates of labile organic matter along a transect from the shallow, highly dynamic, Odra estuary to the Arkona Basin, a deep, low energy, depositional area. In FLSM and sediments respectively, the ranges of organic matter contents were 4.0–25.0% and 5.1–23.0%, those of lipids 0.1–5.4% and 0.30–1.67%, and those of fatty acids 50–991 $\mu\text{g g}^{-1}$ dry wt. and 100–546 $\mu\text{g g}^{-1}$ dry wt. In shallow waters, the contents

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of these compounds are very variable, mirroring variations in biological activity and hydrological conditions. The high percentage of polyunsaturated fatty acids in shallow areas indicates the presence of fresh, undegraded, labile organic matter of autochthonous origin. Fatty acids, the most labile components, are transported as suspended matter attached to minerals and finally accumulated in the depositional area in the form of condensed organic macromolecules. The intensity of bacterial decomposition of organic matter in this system is reflected in the high percentage of branched fatty acids. The low fatty acid content in the organic matter is attributed to the high rate of decomposition of the labile organic matter. No linear correlation was found between the contents of fatty acids and lipids.

1. Introduction

The variations in organic matter composition during transport from coastal areas of high productivity to depositional basins are determined by competing processes, such as *in situ* primary production, secondary production, alteration and decomposition during sinking and horizontal transport (Henrichs 1992). Such transformations are of major importance for establishing the contribution of organic matter in the global carbon cycle, as well as for the qualitative evaluation of the fate of organic matter (Bouloubassi et al. 1997). Sedimentary and near-bottom organic matter is a mixture of constituents derived from a number of sources and covering a wide spectrum of reactivity (Canuel & Martens 1996). Lipids are important constituents of organic matter (Harvey et al. 1986). Although not as abundant as proteins, carbohydrates and poorly-characterized macromolecular material, lipids possess specific molecular characteristics (due to the presence of fatty acids and sterols), which can be used to obtain insight into the sources and decomposition patterns of transported matter (Thoumelin et al. 1997, Wakeham et al. 1997a). Fatty acids (FAs) are useful tracers of organic matter sources and transformation in particulate matter and sediments (Lee & Wakeham 1992, Najdek 1993). Of particular interest are polyunsaturated FAs, owing to their specific occurrence in different planktonic organisms, which enables them to be traced to their planktonic origin (Marty et al. 1988, Scribe et al. 1991). Because of their high reactivity, unsaturated FAs are more prone to decomposition processes than saturated ones. Their percentage contribution may thus serve as an indicator of organic matter freshness (Reetsma et al. 1990). The detection of long-chain saturated (> 22 carbon atoms per molecule) and monounsaturated FAs (C 20:1 and C 22:1) commonly found in the cuticular waxes of higher plants is indicative of terrigenous organic matter (Matsumoto 1981, Grimalt & Albaiges 1990). The presence of branched aliphatic FAs containing an odd number of carbon atoms in the molecule is an indicator of microbiological degradation (Perry et al. 1979, Brown et al. 1996).

In the coastal zone, one of the most important means of organic matter transport from estuaries to areas of sedimentation is the suspended matter spread just above the sea bed (Christiansen et al. 1999). Often referred to as fluffy layer suspended matter (FLSM), this layer of fairly concentrated particulate matter may be several centimetres thick. Unconsolidated FLSM is composed of aggregated biogenic and lithogenic particles. During calm weather FLSM accumulates at the sediment-water interface. It is easily re-suspended when current velocities exceed 5 cm s^{-1} . Where the sea bed is covered with muddy sediment, it is scarcely distinguishable from the sediment proper (Leipe et al. 2000, Löffler et al. 2000). FLSM is of considerable interest, as it seems to be the means by which biogenic material, pollutants and riverine nutrients are transported from shallow estuarine waters to deeper sedimentary basins (Christiansen et al. 1999, Witt et al. 2001). At present, still very little is understood about the properties, composition and fluxes of FLSM.

The objective of the present study was establishing modification of organic matter transported as FLSM. The river Odra – Arkona Deep transect running from the shallow, high-energy environment near the mouth of the Odra to the final, calm depositional area, the Arkona Basin (Southern Baltic), was selected as the study area. Fatty acids were the parameter used for establishing the sources and decomposition intensity of the labile organic matter along this transect.

2. Materials and methods

Samples of FLSM and surface sediment were collected from four stations in the Odra mouth – Arkona Basin transect from October 1996 to December 1998 (Fig. 1). The characteristics of the sampling sites are given in Table 1. Samples of suspended matter were collected by divers using a vacuum pump system operating from on board ship. The water was removed by means of a flow-through centrifuge. Undisturbed sediment samples were collected by divers or with a Reineck box corer. The samples were transferred to Petri dishes and kept deep-frozen for laboratory analyses.

The freeze – dried material was extracted three times with 2:1 chloroform:methanol. The fatty acids contained in the combined extracts will be referred to as free fatty acids (F1). The solid residue was then saponified with 0.5M KOH in a 95:5 v/v methanol:water solution and filtered. The acids determined in the resulting filtrate are referred to as bound fatty acids (F2). The solid saponified residue was refluxed in 6M HCl to yield residual fatty acids (F3). After separation from other lipid components, the fatty acids were derivatized into coumaryl esters and analyzed quantitatively using high performance liquid chromatography (HPLC) with a reversed

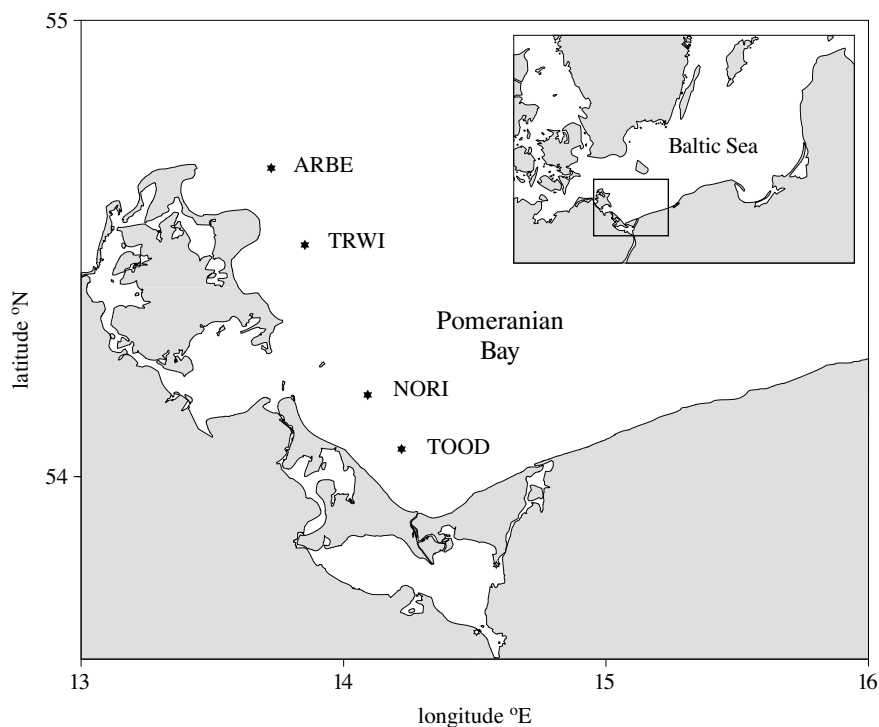


Fig. 1. Location of sampling stations

phase column, a gradient methanol:water mobile phase and fluorescence detection system. Details of the procedure were described earlier (Pazdro & Falkowski 1994). The total lipid content was determined as the dry-weight chloroform:methanol (2:1, v/v) extracts of freeze-dried material (100 mg). The precision of the results, expressed as a relative standard deviation, varied from 0.4 to 16.8% for the total fatty acid content, from 1.4 to 20% for individual compounds, and from 1.4 to 5.6% for the lipid content analysis.

The organic matter content was measured as loss on ignition (a 200 mg subsample was ignited at 450°C for 4 hours). To obtain the organic carbon content of Baltic sediments, the loss on ignition has to be multiplied by a factor of 0.47 (Pempkowiak et al. 1988, Persson & Jonsson 2000).

3. Results and discussion

Tables 2 and 3 present the data sets of fatty acid contents and composition, lipid and organic matter contents. The values measured in the samples displayed considerable variability. At ODAS TONNE, organic matter varied from 5.1% dry wt. in June 1997 to 29.3% dry wt. in June 1998, but no obvious trend in its variability could be discerned. Compared to

Table 1. Characterization of sampling sites (Emeis 1997)

Sampling site	Characterization
Odas Tonne (TOOD)	14 m water depth, situated near the mouth of the Odra river, sand ripples are occasionally overlain by thin fluff. Highest variability in conditions at the seafloor, surface sediment type, benthic communities and sedimentary conditions
Nordperd Rinne (NORI)	20 m water depth, sand overlain by 2 cm fluff during calm periods. Diverse seafloor types including sand ripples and bivalve clusters, boulder fields on erosional hardgrounds
Tromper Wiek (TRWI)	25 m water depth, transition from sand-covered areas to depositional area of southern Arkona Basin. Sandy mud, frequently inhabited by bivalves
Arkona Basin (ARBE)	46 m depth, depositional and accumulation area covered with liquid mud. Oxygen concentrations as low as 0.3 ml/l in bottom waters

deep-sea samples, the total organic matter contents at this station are relatively high (Bouloubassi et al. 1997, Wakeham et al. 1997b), and are indicative of the limited mineralisation of bulk organic matter in FLSM along the transect. Lipid contents varied from 0.33 to 2.30% dry wt. of FLSM at ARKONA BASIN in March 1997 and ODAS TONNE in June 1998 respectively. Total fatty acids ranged from 25 $\mu\text{g g}^{-1}$ dry wt. at NORPERD RINNE in March 1997 to 300 $\mu\text{g g}^{-1}$ dry wt. at ODAS TONNE and ARKONA BASIN in August 1997. The relatively high contents of August 1997 can be related to the higher biological activity due to nutrients entering the Pomeranian Bay after the Odra flood in July 1997 (Laima et al. 1999). No significant correlation was found between fatty acids, lipids and organic matter. The linear regression coefficient r^2 ranged from 0.32 to 0.54 ($n = 25$). The correlation ($r^2 = 0.54$) was highest between polyunsaturated fatty acids and lipids. Both are indicators of fresh organic matter resulting from intense biological activity (Sargent et al. 1980, Saliot et al. 1991).

Normalizing the contents of lipids and fatty acids to bulk organic matter allows the variable composition, relative to organic carbon, to be assessed (Martin et al. 1987). Converting these concentrations to lipid-organic matter

Table 2. Average free fatty acids, lipid, and organic matter contents at stations along the transect

Sampling station*	Sampling date	Total free FAs	Total lipids	Total organic matter	Total free FAs	Total free FAs	Total lipids
		[$\mu\text{g g}^{-1}$ dry wt.]	[%]	[%]	(% of OM)	(% of lipid)	(% of OM)
Odas Tonne (TOOD)	March 1997	43	***	14.55	0.03	***	***
	June 1997	52	***	5.08	0.10	***	***
	August 1997	302	1.22	18.39	0.16	2.48	6.63
	October 1997	113	***	13.70	0.08	***	***
	June 1998	302	2.30	29.29	0.10	1.31	7.85
	December 1998	195	2.02	19.27	0.10	0.97	10.48
Nordperd Rinne (NORI)	March 1997	25	***	16.02	0.02	***	***
	June 1997	55	0.28	17.93	0.03	1.96	1.56
	October 1997	105	***	9.30	0.11	***	***
	June 1998	158	1.25	23.31	0.07	1.26	5.36
	December 1998	250	1.21	17.24	0.15	2.07	7.02
	Tromper Wiek (TRWI)	March 1997	168	***	15.88	0.11	***
June 1997		96	0.41	23.14	0.04	2.34	1.77
October 1997		160	***	7.96	0.20	***	***
June 1998		300	1.3	20.54	0.15	2.31	6.33
December 1998		200	0.14	15.12	0.13	14.29	0.93
Arkona Basin (ARBE)		October 1996	910	***	11.62	0.78	***
	March 1997	101	0.33	16.29	0.06	3.06	2.03
	June 1997	300	0.78	16.30	0.18	3.85	4.79
	August 1997	191	0.49	14.90	0.13	3.90	3.29
	December 1998	87	1.14	***	***	0.76	***

* The location of sampling stations is given in Fig. 1; *** – not determined.

Table 3. Free fatty acids composition in fluffy layer samples at sampling stations along the transect

Sampling station*	Sampling date	Fatty acids composition (% of Total FAs)			
		saturated FAs C8:0–C24:0**	monounsaturated FAs C14:1–C22:1**	polyunsaturated FAs C18:2–C22:6**	branched FAs C _{ai, i} 15:0–C _{ai, i} 17:0***
Odas Tonne (TOOD)	March 1997	99.30	0.70	–**	–**
	June 1997	94.70	2.10	2.90	3.00
	August 1997	53.90	22.50	17.20	6.40
	October 1997	65.20	21.40	7.60	5.80
	June 1998	52.40	29.90	15.20	1.50
	December 1998	83.10	9.10	6.80	1.60
Nordperd Rinne (NORI)	March 1997	100.00	–**	–**	–**
	June 1997	95.30	3.40	0.00	1.40
	October 1997	83.10	9.90	6.90	3.20
	June 1998	76.50	7.70	14.20	1.60
	December 1998	64.30	18.10	16.02	1.48
Tromper Wiek (TRWI)	March 1997	100.00	–**	–**	–**
	June 1997	99.00	1.00	–**	–**
	October 1997	80.20	10.70	6.00	3.10
	June 1998	73.20	14.60	10.40	1.80
	December 1998	70.50	20.30	8.50	0.80
Arkona Basin (ARBE)	October 1996	74.10	16.50	8.50	1.10
	March 1997	94.30	4.40	–**	1.50
	June 1997	87.60	7.20	3.00	2.20
	August 1997	85.10	12.10	1.20	2.68
	December 1998	74.00	18.40	4.00	3.60

*See Fig. 1 for location of sampling stations.

**The first numerals denote the number of carbon atoms in the aliphatic chain, the second denote the number of double bounds;

*** ai – anteiso, i-iso. These abbreviations indicate the position of methyl group (branch) in branched aliphatic acids;

–** – below the detection limit.

equivalents (Table 2) showed that lipids represented from 0.9% of particulate organic matter at TROMPER WIEK in December 1998 to 10.5% at ODAS TONNE in December 1998. The calculated fatty acid/organic matter and fatty acid/lipid ratios ranged from 0.02 to 0.78% and from 0.76 to 14.26% respectively. Wakeham (1995) reported lipid-organic matter ratios of 15–30% in euphotic zone particles – characteristic of fresh organic material – and ratios of 0.05–0.5% in deep-water surface sediments; the respective ratios for fatty acids/organic matter ranged from 3.5 to 0.8–1.2%. This study showed that transfer to sediments leads to preferential loss of fatty acids, the most labile compounds, as a consequence of which fatty acid/organic matter ratios are very low (< 0.8%). The lipid/organic matter ratios showed that lipids were, in general, selectively lost in the course of transport, owing to their greater reactivity relative to the bulk organic matter. On the basis of the variability in fatty acid/lipid ratios it can be inferred that fatty acid degradation is also faster than that of lipids as a whole. This is probably due to the variable reactivity of particular classes of lipids (Canuel & Martens 1996).

The contribution of different FA classes to the total amount of FAs is given in Table 3. The fatty acid fractions found in FLSM were dominated by saturated C8–C22 even-carbon FAs. There was as much as 64 to 100% of saturated FAs in the total FA quantity. The substantial contribution, from 10% to 40%, of shorter-chain (C8–C14) saturated fatty acids in the samples should be noted: the carbon chains become shorter as they decompose (Sun & Wakeham 1997). Polyunsaturated FAs (C18:2–C22:6) made up from 3% of the total FAs at ODAS TONNE in June 1997 to 17% at the same station in August 1997. In shallow areas these compounds were highly variable as a result of changes in biological activity and hydrological conditions. The increased amounts of fresh autochthonous polyunsaturated FAs, characteristic of planktonic and benthic communities at ODAS TONNE in August 1997, were probably due to enhanced primary productivity stimulated by nutrients supplied by the masses of river water after the July 1997 flood (Humborg et al. 1998). The high percentage contribution of polyunsaturated fatty acids in the total amount at both shallow stations – ODAS TONNE and NORDPERD RINNE – indicates the presence of fresh, undegraded, labile organic matter of autochthonous origin (Marty et al. 1988). Interestingly, long-chain saturated and monounsaturated FAs – markers of terrigenous origin (Lipiatou & Saliot 1991) – were exclusively present in August in the free fatty acid fraction (F1) (at the level of 5%). This must have been related to the elevated quantities of river water entering the Pomeranian Bay during the Odra flood (Laima et al. 1999). Fatty acids specific to bacteria were clearly present in all the

samples. The contributions of iso- and anteiso- 15:0 and 17:0 fatty acids were high, varying from 0.80 to 6.40% of the total free fatty acids present in FLSM. This feature indicates intensive reworking of organic matter (Grimalt & Albaiges 1990).

The results were evaluated in order to assess the statistical significance of spatial or temporal trends in the distribution of the fatty acids in the investigated area. Fig. 2 and Table 4 illustrate the total free fatty acid contents and composition as dependent variables grouped according to station. While the average total concentrations do not reveal any significant differences, analysis of the average percentage composition does point to an increase in saturated fatty acids, the most resistant compounds,

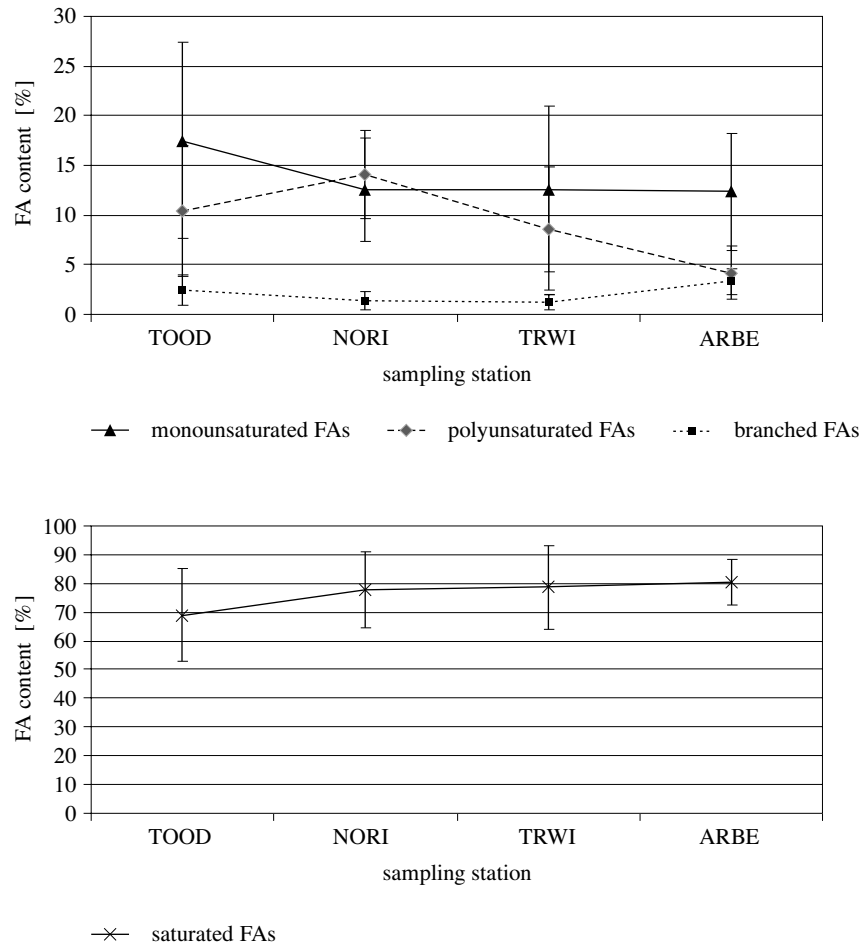


Fig. 2. Mean composition of fatty acids and standard deviation in fluffy layer samples from successive stations

Table 4. Average fatty acids concentrations and standard deviations at stations

Sampling station*	Average		Average percentage							
	conc \pm SD [μg^{-1} dry wt.]	RSD [%]	saturated FA		monounsaturated FA		polyunsaturated FA		branched FA	
			(1)	(2)	(1)	(2)	(1)	(2)	(1)	(2)
Odas Tonne (TOOD)	253 \pm 179	71	69.0 \pm 16.4	24	17.0 \pm 9.9	58	10.4 \pm 6.5	63	2.4 \pm 1.5	63
Nordperd Rinne (NORI)	369 \pm 420	114	77.7 \pm 13.3	17	12.5 \pm 5.2	42	14.1 \pm 4.4	31	1.4 \pm 0.9	64
Tromper Wiek (TRWI)	365 \pm 321	88	78.6 \pm 14.7	19	12.6 \pm 8.4	67	8.6 \pm 4.2	49	1.2 \pm 0.7	58
Arkona Basin (ARBE)	317 \pm 306	97	83.1 \pm 7.9	10	12.3 \pm 5.8	47	4.1 \pm 2.6	63	3.3 \pm 1.3	39

*See Fig. 1 for location of sampling stations.

(1) [% of total FAs] \pm SD;

(2) RSD [%]

and a decrease in polyunsaturated fatty acids, the most susceptible to degradation processes, with increasing distance from the Odra mouth. Branched fatty acids were distributed fairly uniformly, confirming that intensive reworking of organic matter was taking place along the transect. Near-bottom particles at ODAS TONNE, a sandy, non-depositional site, showed the organic matter to be relatively fresh. FLSM at this site contained relatively high proportions of polyunsaturated fatty acids, which is characteristic of organic matter of algal and benthic origin (Scribe et al. 1991). Seasonal variations in biological activity seem to cause high standard deviations at this station. The fate and quality of near-bottom particles appeared to be related to the local hydrography and depositional circumstances. On the other hand, the particulate organic material in the Arkona Basin depositional area was depleted of fresh fatty acids. The trend was reversed where saturated FAs, the most stable ones, were present. Low standard deviations, demonstrating the low variability of these components in the sedimentation area, confirm their relatively high resistance to degradation.

The physicochemical association between organic compounds and mineral particles can influence the decomposition of organic matter (Boon et al. 1996, Canuel & Martens 1996). Fig. 3 illustrates the speciation of

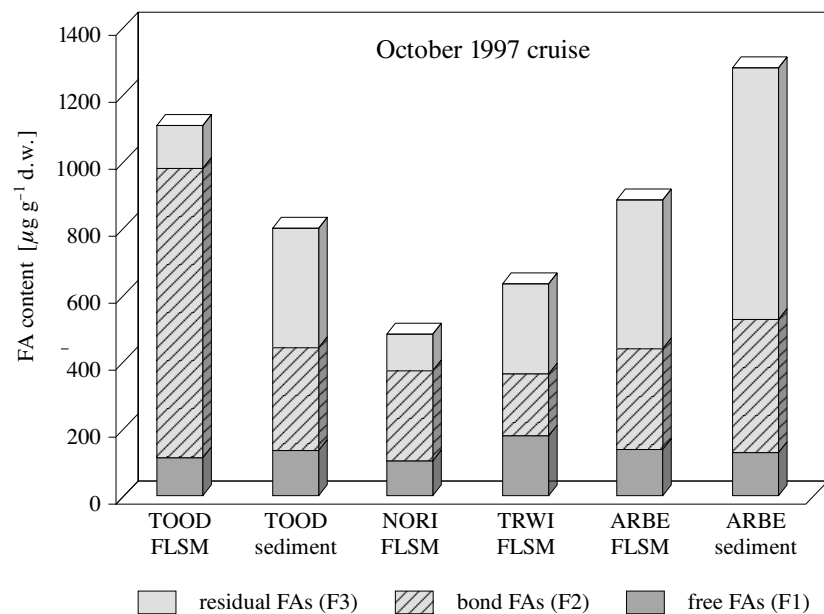


Fig. 3. Fatty acid content in different fractions of fluffy layer suspended matter and selected surface sediments along the transect in October 1997

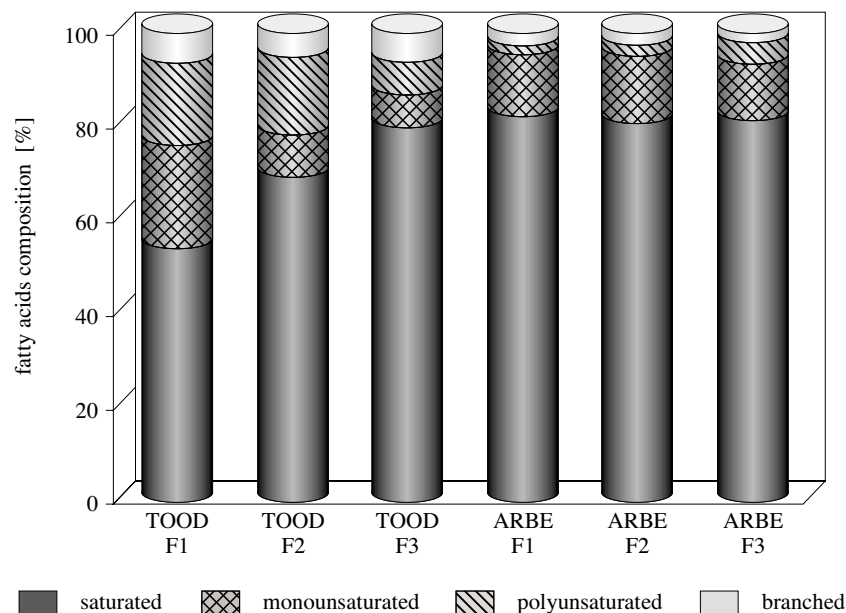


Fig. 4. Distribution of fatty acid types in successive fractions extracted from fluff layer suspended matter collected at the TOOD and ARBE stations in August 1997

fatty acids in FLSM along the transect in October 1997. Free fatty acids ranged from $110 \mu\text{g g}^{-1}$ dry wt. at ODAS TONNE to $190 \mu\text{g g}^{-1}$ dry wt. at TROMPER WIEK. Fatty acids released after saponification of the solid residue (F2) changed from $200 \mu\text{g g}^{-1}$ dry wt. at the latter station to $850 \mu\text{g g}^{-1}$ dry wt. at the former, while fatty acids present in the fraction obtained after hydrolysis of the solid residue (F3) varied from $110 \mu\text{g g}^{-1}$ dry wt. at ODAS TONNE to $450 \mu\text{g g}^{-1}$ dry wt. at ARKONA BASIN. At the shallow station, near the Odra river mouth, FLSM contains a high amount of fraction F2 (about nine times that of F1 and F3), representing fatty acids incorporated into biopolymers, characteristic also of living organisms (Sun et al. 1997). This feature reiterates the ‘freshness’ of organic matter in shallow, coastal areas, already shown up by the presence of relatively high proportions of polyunsaturated fatty acids. The importance of fraction F3, representing the fraction included in macromolecules and attached to mineral particles, increased with distance from the land, reaching peak values in the depositional area. Fig. 4 demonstrates the contribution of different fatty acid groups to the fractions described above. Fatty acids show a rather uniform distribution with branched iso 15 and 17 acids indicating the intensive reworking of organic matter along the transect. The macromolecule fraction at ARKONA BASIN comprises

a low, but measurable amount (0.8% of total FAs) of long-chain fatty acids characteristic of terrigenous material and also a considerable amount of polyunsaturated fatty acids (at the 15% level). This indicates that allochthonous FAs from the land and autochthonous FAs produced in shallow, coastal areas may be transported as biopolymers, possibly attached to minerals, thus contributing to the preservation of even the most reactive constituents over several months.

The contributions of different fatty acid fractions to FLSM and surface sediments at ODAS TONNE and ARKONA BASIN in October 1997 are compared in Fig. 3. The shallow water surface sediments consisted of $150 \mu\text{g g}^{-1}$ dry wt. of free FAs (F1), $310 \mu\text{g g}^{-1}$ dry wt. of FAs contained in biopolymers (F2) and $360 \mu\text{g g}^{-1}$ dry wt. of FAs associated with minerals, while sediments in the depositional area made up 110, 360, and $750 \mu\text{g g}^{-1}$ dry wt. of the respective FA fractions. At ODAS TONNE, speciation of sedimentary fatty acids was markedly different from that of FLSM. The particulate organic material in the Arkona Basin differs to a much smaller extent. The results may suggest that in a shallow area of high biological activity the composition of labile components is derived not only from fresh material from benthic communities, but also from material sinking across the overlying water column. In deeper depositional areas, the composition of the FLSM labile fraction seems to be influenced rather by the resuspension of sedimentary organic particles.

4. Conclusions

Qualitative and quantitative changes over time and space measured along the investigated transect in the Pomeranian Bay demonstrate the dynamic range of reactivity of the target compounds, i.e. the several fatty acids and lipids, and thus of organic matter as a whole. The contents of these compounds, especially fatty acids and lipids, the most reactive ones, are highly variable in the shallow areas near the Odra mouth, where biological productivity is high. Variations in biological activity and hydrological conditions are the factors responsible for this. The high percentage of polyunsaturated fatty acids and the fraction related to biopolymers along the transect indicate the presence of fresh labile organic matter of autochthonous origin. Fatty acids, the most labile components of the organic matter, are transported mainly from the shallow area to the Arkona Basin sedimentation area as biopolymer components attached to minerals (F2 and F3). They are transported and accumulated in the depositional area as fragments of condensed organic macromolecules (F3). Markers characteristic of terrigenous organic matter (long-chain fatty acids) were found in the residual fraction (F3) in sediments from the Arkona Basin.

The important bacterial contribution to the decomposition of organic matter in this system is reflected in the large amounts of branched fatty acids in sediments and FLSM along the transect. The low percentage of fatty acids in the organic matter and lipids indicates that fatty acids decompose faster than bulk organic matter or lipids.

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