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EVIDENCE OF TERRESTRIAL ORGANIC CARBON INPUTS ON NIGER DELTA SEA SHELF.

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ABSTRACT

The objective of this research is to determine the actual source of organic matter in the Niger Delta Sea Shelf (NDSS). Organic carbon (OC) and n-alkanes were investigated in the suspended matter (SM) and surfcial sediments of the Niger and Imo Rivers and the opposite Niger Delta Sea Shelf. Organic nitrogen and carbon isotope ($\delta^{13}C$) values were determined as additional parameters in the sediments. OC and n-alkane concentrations were highly variable. This variability was interpreted as the result of sources of materials from the inland basins and a complex sedimentation pattern involving the discharges of these two rivers. Using the constant ratio of high molecular weight odd n-alkanes to OC in both rivers, the estimated value of more than 70% of the OC preserved in shelf sediments were terrestrially-derived. This result was substantiated by the overall dominance of land-derived n-alkanes. A second approach using $\delta^{13}C$ values and assuming binary dilution of riverine and marine OC led to the determined source of organic matter.

Keywords: Organic carbon; $\delta^{13}C$; n-Alkanes; Niger River; Imo River; Niger Delta Sea Shelf.

INTRODUCTION

The Niger Delta is situated in the Gulf of Guinea (Fig. 1) and formed the Niger Delta Basin as defined by Klett *et al*. (1997). From the Eocene to the present, the delta has prograded southwestward, forming depobelts that represent the most active portion of the delta at each stage of its development (Doust & Omatsola, 1990). These depobelts form one of the largest regressive deltas in the world with an area of some 300,000 km^2 (Kulke, 1995); a sediment volume of 500,000 km^3 (Hospers, 1965); and a sediment thickness of over 10 km in the basin depocenter (Kaplan *et al*., 1994).

Rivers are the prime sources of fresh water and sediments to the ocean, representing 70% of the world's solid inputs to the marine realm (Milliman, 1991; Szczepanska *et al*., 2012; Kandasamy & Nath, 2016; Wu *et al*., 2019). However, river discharges can vary substantially with changes in climate and human land-use. In the twentieth century, the worldwide development of industrial and urban centers has motivated the construction of dams for hydroelectric plants and irrigation conduits inducing major alterations of river flow and discharge of suspended solids reaching coastal margins (e.g. River Nile).

It is estimated that 90% of riverine particulate inputs remain trapped in coastal areas (Mantoura *et al*., 1991). The extent to which these areas can retain or export fluvial material can affect the global carbon mass balance. The understanding of the transfer of fluvial organic carbon (OC) to the marine environment on a global scale requires an extended geochemical database. Comparing with other regions in the World such as rivers discharging into temperate North-American, European and Eurasian shelves of the Arctic Ocean, the Niger-Delta shelf are poorly documented (e.g. Dmitriev & Pivovarov, 1983; Fernandes, 1996; Yunker *et al*., 1996; Broyelle, 1997; Fernandes & Sicre, 2000; Zaghden *et al*., 2017).

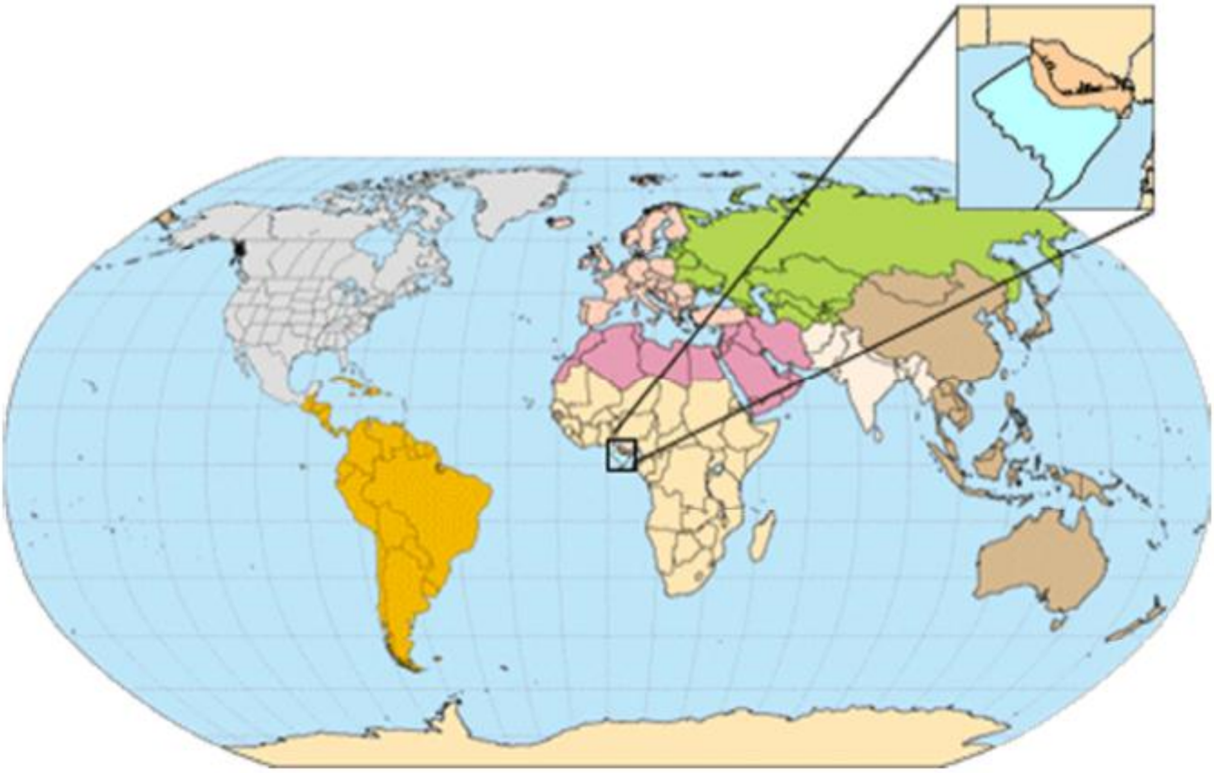

Figure 1: Niger delta in the Gulf of Guinea West Africa (modified after Michele *et al*., 1999).

The present study aims to partly to determine and to document the source of organic matter in the Niger Delta Sea Shelf (NDSS).

SYNOPSIS OF THE GEOLOGY

The onshore portion of the Niger Delta Basin is delineated by the geology of southern Nigeria and southwestern Cameroon (Fig. 1). The northern boundary is the Benin flank--an east-northeast trending hinge line south of the West Africa basement massif. The northeastern boundary is defined by outcrops of the Cretaceous on the Abakaliki High and further east-south-east by the Calabar flank a hinge line bordering the adjacent Precambrian Basement. The offshore boundary of the province is defined by the Cameroon volcanic line to the east, the eastern boundary of the Dahomey basin (the eastern-most West African transform-fault passive margin) to the west. The basin covers 300,000 km^2 and includes the stratigraphic extent of the Tertiary Niger Delta (Akata-Agbada) Petroleum System.

Several workers (Short & Stauble, 1967; Weber, 1971; Weber & Daukoru, 1975;

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Weber *et al*., 1978; Evamy *et al*., 1978; Doust & Omatsola, 1990) have given detailed reports on the geology of this province. The Niger delta is composed of regressive sequence of clastic sediments developed in a series of offlap cycles demonstrating a tripartite lithostratigraphic succession (Fig. 2) of a massive monotonous marine shale (Akata Formation) below a paralic sequence of alternation of sand and shale in the middle (Agbada Formation) and capped by thick sequence of fresh water sand (Benin formation).

The Akata Formation likely extends to the basement rock. These three sedimentary formations are typical of most deltaic environments, extend across the whole delta and ranges in age from early Tertiary to Recent.

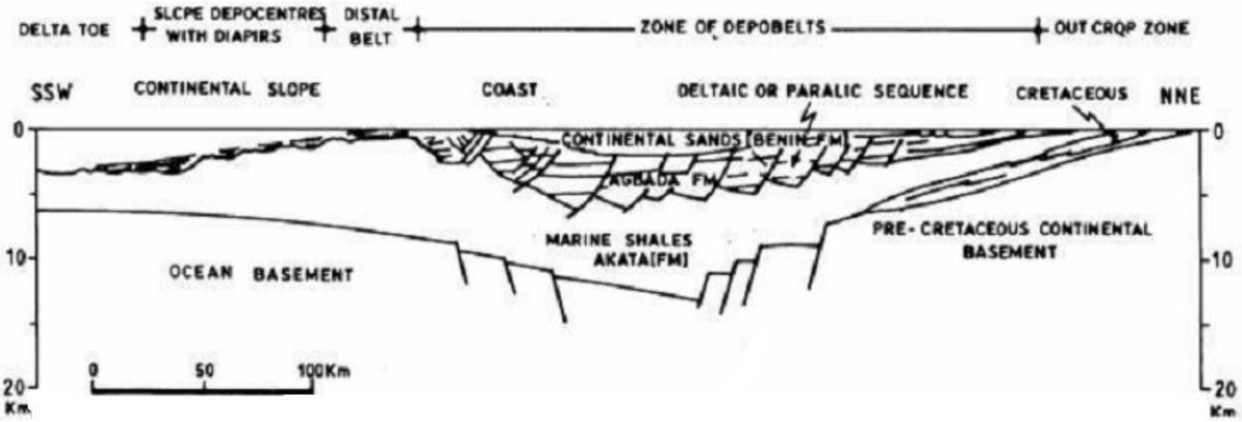

Figure 2: Schematic section through the axial portion of the Niger Delta showing the relationships of the tripartite division of the tertiary sequence to basement (Doust & Omatsola, 1990).

Petroleum in the Niger Delta is produced from sandstone and unconsolidated sands predominantly in the Agbada Formation. The most striking structural features of the Cenozoic Niger Delta complex are the synsedimentary structures which deform the delta largely beneath the Benin Formation. These structures, regarded as the product of gravity sliding during the course of deltaic sedimentation, are polygenic in origin and their complexity increases generally in down delta direction (Merki, 1972). The synsedimentary structures, called growth faults, are predominantly trending northeast to southwest and northwest to southeast (Hosper, 1971). Associated with these growth faults are rollover anticlines, shale ridges and shale diapers which are caused by shale upheaval ridges. Mud diapers are the most common and occur on the landward side of the growth faults restricting sedimentation on the up-thrown side of the faults and enhancing sedimentation on the down-thrown side. Oil and gas are predominantly trapped by roll over anticlines and fault closures. Stratigraphic traps of paleo-channel fills, regional sand pinch-outs and truncations, crestal accumulations below unconformity surfaces, canyon-fill accumulations above unconformity surfaces, incised valley and low-stand fans have been recognized (Orife & Avbovbo, 1982; Kruise & Idiagbor, 1994).

MATERIALS AND METHODS

Samples were collected between February and April 2018 during the time water level has reduced. The representative samples were subjected further to organic geochemical analyses. Elemental organic carbon and nitrogen (OC and N), carbon stable isotope ($\delta^{13}C$), and n-alkanes (ALKs) were determined in the suspended matter (SM) and surface sediments of the Niger (NGR) and Imo (IMR) estuaries and Niger Delta Sea Shelf (NDSS) to estimate the amount of terrestrially-derived OC buried in shelf sediments.

The area of investigation covers between latitude 4°20' and 5°59'N and longitude 6°42' and 7°35'E, along the Niger and Imo rivers and in the opposite NDSS. Three longitudinal sections of the estuary (riverine zone, mixing zone and marine zone) were distinguished based on the locations and salt content (Fernandes & Sicre, 1999). Salinity values ranged from 0 to 32 throughout the study area.

Surface water samples were collected above the halocline, between 2 and 7 m. 3 to 5 litres (L) of water were pumped into glass containers and then filtered on-board boat on pre-extracted Whatman GF/F glass fiber filters (147 or 290 mm diameter, 0.7µm porosity). The fiber filters and surficial sediments (0 - 3 cm) were wrapped in aluminum foil and kept frozen at -18°C before extraction from the samples. All surfaces in contact with the samples were of Teflon or stainless steel.

Filtrates for hydrocarbon analysis were extracted three times from the fiber filters by ultrasonication in 200 mL of a mixture of $CH_3OH:CH_2Cl_2$ (1:3 v/v) for 15 min. The three extracts (600 mL) were combined in a round bottom flask and dried with $MgSO_4$ overnight. The total lipid extract was filtered through glass wool, concentrated by rotary evaporation and then transferred to a 4 mL vial. Sediments were freeze-dried and homogenized. An aliquot was extracted in a reflux system with 200 mL of a mixture of CH_3OH: CH_2Cl_2 (1:3 v/v) for 2 hours followed by ultrasonication for 15 min. The total lipid extract was filtered, concentrated by rotary evaporation and dried with $MgSO_4$ overnight. These extracts were then treated in the same way as those of the particulate phase. Particulate and sedimentary non-aromatic hydrocarbons were isolated from the lipid mixture by elution with hexane through a pipette filled with silica gel (Fernandes *et al.*, 1997).

Quantification of individual n-alkanes was based on comparison of peak areas with those of perdeuterated tetracosane added prior to extraction. The quantification limit for individual compounds was estimated at around 1-3 ng (signal/noise $\geq$ 10) and the reproducibility of the method was estimated at ±10%. Blanks showed no detectable contamination. Hydrocarbons were analyzed using a CP-Sil-8 CB Chrompack capillary column (50 m x 0.32 mm i.d. x 0.25 µm film thickness) mounted in a Girdel 3000 gas chromatograph (GC) equipped with a glass needle injector heated at 250°C and a Flame Ionization Detector (FID). The GC oven was programmed from 80 to 300 °C at 2 C/min. Helium was used as carrier gas. Data acquisition and processing was performed on a Varian Vista CDS 402 integrator.

The identification of individual n-alkanes was performed by comparison of their retention times with those of authentic standards. Structural assignment was

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confirmed by gas chromatography/mass spectrometry (GC/MS) on selected samples. GC/MS analyses were carried out on a Varian 3400 GC coupled to an Ion Trap Varian Saturn mass spectrometer. Samples were injected using an on-column, Septum, Programmable Injector (SPI) on a DB5 J & W Scientific capillary column (30 m x 0.25 mm i.d. x 0.25 µm film thickness) programmed from 80 to 300 °C at 3 °C/min. Operating conditions were: ion source temperature 140 °C, electron energy 70 eV, scanning from 40 to 600 amu, 0.6 scan/s.

Particulate organic carbon (POC) and SM data were obtained by Dai (1995) during the same cruise. The analytical reproducibility of POC measurements was estimated at ± 2%. Sedimentary organic carbon (OC), organic nitrogen (N) and $\delta^{13}C$ were analyzed following the experimental procedure by Girardin and Mariotti (1991). Briefly, samples were decarbonated in 25 mL of HCl (0.001 M), followed by the addition of HCl (0.5 M) till a constant pH of 3 was reached. They were centrifuged, washed with deionized water and dried for 12 hours at 50 °C. After being homogenized and weighed, aliquots were combusted in an elemental analyzer (Carlo Erba, NA 1500 NC, Fisons). The CO_2 and N_2 produced were chromatographically separated. OC and N were then measured by thermal conductivity. The analytical reproducibility of these measurements was estimated at ±3%. Combustion gases were then cryogenically purified through several temperature traps and introduced into the isotopic ratio mass spectrometer fitted for rapid switching (VG Sira 10 Spectrometer). Samples were compared to Pee Dee Belemnite (PDB) as reference to determine their $\delta^{13}C$ values. The experimental error was estimated to be better than ±0.1%o based on successive injections of the tyrosine standard.

Two different methods were employed (Prahl *et al*., 1994; Mayer *et al*., 2007; Zaghden *et al*., 2017; Wu *et al*., 2019). The first method is based on the correlation earlier evidenced between sedimentary n-alkanes and OC. Accordingly, the %TERR coastal sediment of the NDSS can be estimated by the following proportion:

$$\%TERR = \frac{(\Sigma ALK_{25\text{-}31}/OC)_{\text{sample}}}{\text{Slope}} \times 100$$

The second approach uses $\delta^{13}C$ values and assumes that OM in coastal sediments derives from the binary mixing of riverine and marine OC. The %TERR in this case is calculated by the following equation:

$$\%TERR = \frac{(\delta^{13}C)_{\text{sample}} - (\delta^{13}C)_{\text{marine}}}{(\delta^{13}C)_{\text{riverine}} - (\delta^{13}C)_{\text{marine}}} \times 100$$

RESULTS

Suspended matter (SM) concentrations range from 4 to 143 mg/L (average of 30 mg/L, n=7) in the Niger River, and from 1 to 23 mg/L (average of 5 mg/L, n=11) in the Imo River, while < 1 mg/L in the NDSS. OC percentages vary from 4 to 11% in the Niger River (average of 4%, n=7), and from 3 to 5% in the Imo (average of 7%, n=11). They are higher in the NDSS, ranging from 3 to 19% (average of 12%, n=5) as shown in Table 1.

Total n-alkane concentrations are on the average ~11 µg/g in both Niger and Imo sediments. The value in the NDSS is 4 µg/g (Table 2). The ratios of $\delta^{13}C$ (from - 29.0 to - 24.4) and C/N (from 7 to 13) cover the same ranges as those found for sediments in the same area ($\delta^{13}C$ from - 28.5 to - 24.4, C/N from 5 to 14), Table 3.

DISCUSSION

Suspended matter

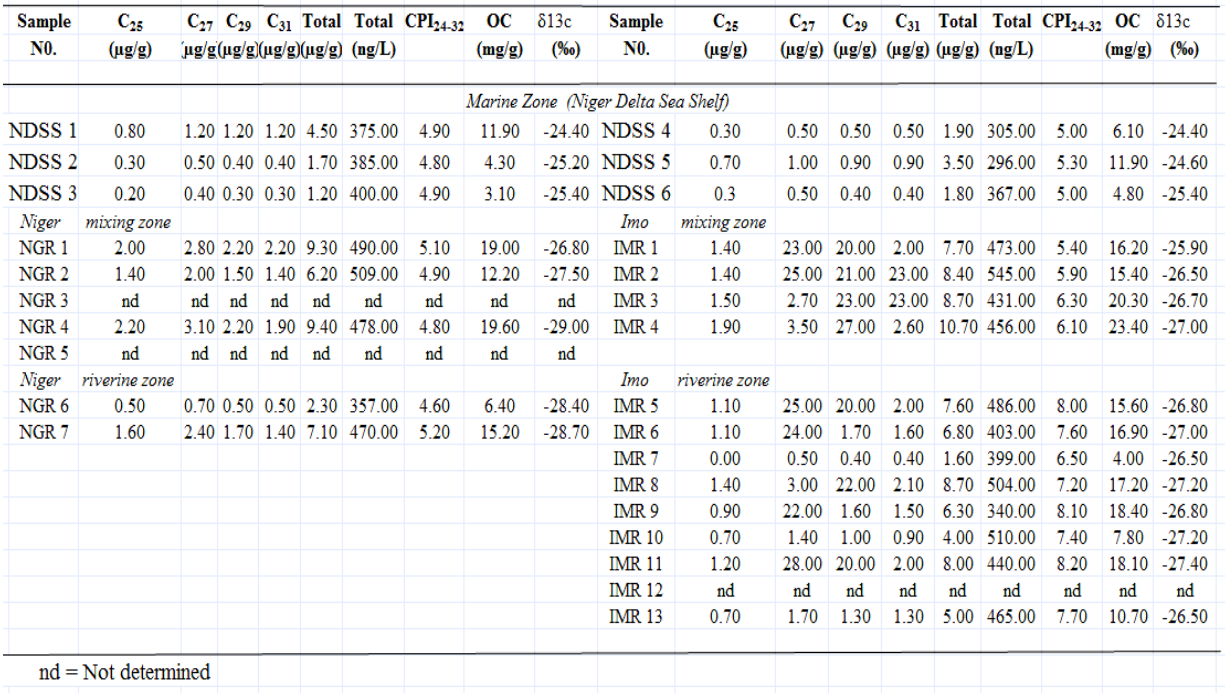
Suspended matter (SM) concentrations (Dai, 1995) are in the lower range as reported for World Rivers (Meybeck, 1982; Ittekkott, 1988). Total particulate odd carbon number n-alkanes from C_{25} to C_{31} (ΣALK_{25-31}) and OC concentrations (in ng/L and mg/L, respectively) are higher in the

Table 1: C_{25}, C_{27}, C_{29} and C_{31} individual and total n-alkane concentrations (in µg/g and ng/L) in the suspended matter along the Niger and Imo estuaries and Niger Delta Sea shelf; CPI_{24-32} values inclusive.

Sample N0.	**C_{25} (µg/g)**	**C_{27} (µg/g)**	**C_{29} (µg/g**	**C_{31} µg/g**	**Total (µg/g)**	**Total (ng/L)**	**CPI_{24-32}**	**Sample N0.**	**C_{25} (µg/g)**	**C_{27} (µg/g)**	**C_{29} (µg/g)**	**C_{31} µg/g**	**Total (µg/g)**	**Total (ng/L)**	**PI_{24-32}**
							Marine Zone	*(Niger Delta Sea Shelf)*							
NDSS 1	2.10	3.60	2.40	2.30	10.40	9.40	3.00	NDSS 4	6.00	11.60	7.00	6.00	30.50	13.70	3.30
NDSS 2	5.90	12.00	7.20	7.40	32.50	18.20	4.20	NDSS 5	4.20	8.30	4.80	4.30	21.60	12.50	3.90
NDSS 3	2.40	4.70	3.40	3.10	13.60	7.40	2.50	NDSS 6	nd	nd	nd	nd	nd	nd	nd
Niger	*mixing zone*							*Imo*	*mixing zone*						
NGR 1	1.10	1.70	1.20	1.10	5.20	20.70	3.70	IMR 1	2.70	4.60	3.10	2.80	13.30	14.10	3.70
NGR 2	1.30	1.60	1.30	1.10	5.30	31.10	3.20	IMR 2	2.30	4.00	2.80	2.60	11.60	11.90	3.60
NGR 3	3.60	4.60	3.40	2.90	14.40	128.50	4.00	IMR 3	3.30	6.20	4.90	4.60	18.80	23.20	4.00
NGR 4	2.40	3.40	2.20	1.90	9.80	92.40	4.20	IMR 4	7.80	13.70	9.70	9.30	40.50	86.70	5.40
NGR 5	3.00	4.10	2.60	2.30	12.00	191.70	4.30								
Niger	*riverine zone*							*Imo*	*riverine zone*						
NGR 6	3.10	4.40	2.90	2.50	12.90	259.10	4.40	IMR 5	2.10	3.20	2.70	2.30	10.30	51.30	3.80
NGR 7	2.00	2.90	2.00	1.70	8.50	1220.40	4.50	IMR 6	nd	nd	nd	nd	nd	37.30	3.20
								IMR 7	3.20	4.90	3.80	3.10	15.00	49.50	3.70
								IMR 8	2.10	3.60	2.70	2.60	11.00	76.80	5.60
								IMR 9	2.60	4.20	3.50	2.80	13.00	59.20	4.40
								IMR 10	2.50	4.10	3.00	2.80	12.30	278.30	5.50
								IMR 11	nd	nd	nd	nd	nd	nd	nd
								IMR 12	1.90	3.30	2.90	2.40	10.50	56.40	4.80
								IMR 13	2.00	3.40	2.80	2.50	10.70	61.60	5.10

nd = Not determined

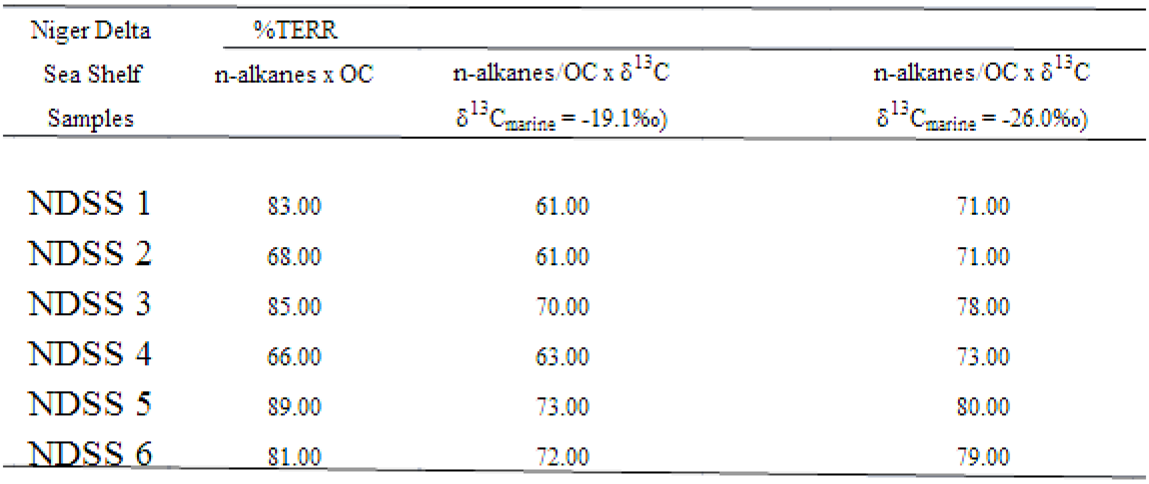
Table 2: C_{25}, C_{27}, C_{29} and C_{31} individual and total n-alkane concentrations (in µg/g dry weight and µg/g OC), CPI_{24-32} values, OC concentrations (in mg/g) and $\delta^{13}C$ values (in ‰ PDB) in sediments along the Niger and Imo estuaries and Niger Delta Sea shelf.



Sample N0.	**C_{25} (µg/g)**	**C_{27} (µg/g)**	**C_{29} (µg/g)**	**C_{31} (µg/g)**	**Total (µg/g)**	**Total (ng/L)**	**CPI_{24-32}**	**OC (mg/g)**	δ13c **(‰)**	**Sample N0.**	**C_{25} (µg/g)**	**C_{27} (µg/g)**	**C_{29} (µg/g)**	**C_{31} (µg/g)**	**Total (µg/g)**	**Total (ng/L)**	**CPI_{24-32}**	**OC (mg/g)**	δ13c **(‰)**
								Marine Zone (Niger Delta Sea Shelf)											
NDSS 1	0.80	1.20	1.20	1.20	4.50	375.00	4.90	11.90	-24.40	NDSS 4	0.30	0.50	0.50	0.50	1.90	305.00	5.00	6.10	-24.40
NDSS 2	0.30	0.50	0.40	0.40	1.70	385.00	4.80	4.30	-25.20	NDSS 5	0.70	1.00	0.90	0.90	3.50	296.00	5.30	11.90	-24.60
NDSS 3	0.20	0.40	0.30	0.30	1.20	400.00	4.90	3.10	-25.40	NDSS 6	0.3	0.50	0.40	0.40	1.80	367.00	5.00	4.80	-25.40
Niger	*mixing zone*									*Imo*	*mixing zone*								
NGR 1	2.00	2.80	2.20	2.20	9.30	490.00	5.10	19.00	-26.80	IMR 1	1.40	23.00	20.00	2.00	7.70	473.00	5.40	16.20	-25.90
NGR 2	1.40	2.00	1.50	1.40	6.20	509.00	4.90	12.20	-27.50	IMR 2	1.40	25.00	21.00	23.00	8.40	545.00	5.90	15.40	-26.50
NGR 3	nd	nd	nd	nd	nd	nd	nd	nd	nd	IMR 3	1.50	2.70	23.00	23.00	8.70	431.00	6.30	20.30	-26.70
NGR 4	2.20	3.10	2.20	1.90	9.40	478.00	4.80	19.60	-29.00	IMR 4	1.90	3.50	27.00	2.60	10.70	456.00	6.10	23.40	-27.00
NGR 5	nd	nd	nd	nd	nd	nd	nd	nd	nd										
Niger	*riverine zone*									*Imo*	*riverine zone*								
NGR 6	0.50	0.70	0.50	0.50	2.30	357.00	4.60	6.40	-28.40	IMR 5	1.10	25.00	20.00	2.00	7.60	486.00	8.00	15.60	-26.80
NGR 7	1.60	2.40	1.70	1.40	7.10	470.00	5.20	15.20	-28.70	IMR 6	1.10	24.00	1.70	1.60	6.80	403.00	7.60	16.90	-27.00
										IMR 7	0.00	0.50	0.40	0.40	1.60	399.00	6.50	4.00	-26.50
										IMR 8	1.40	3.00	22.00	2.10	8.70	504.00	7.20	17.20	-27.20
										IMR 9	0.90	22.00	1.60	1.50	6.30	340.00	8.10	18.40	-26.80
										IMR 10	0.70	1.40	1.00	0.90	4.00	510.00	7.40	7.80	-27.20
										IMR 11	1.20	28.00	20.00	2.00	8.00	440.00	8.20	18.10	-27.40
										IMR 12	nd	nd	nd	nd	nd	nd	nd	nd	nd
										IMR 13	0.70	1.70	1.30	1.30	5.00	465.00	7.70	10.70	-26.50

nd = Not determined

Table 3: The terrestrial contribution to organic carbon (%TERR) in sediments of the Atlantic Sea using two different approaches: n-alkanes x OC, and n-alkanes/OC x $\delta^{13}C$ (with two different marine END-MEMBERS).



Niger Delta Sea Shelf Samples	%TERR n-alkanes x OC	n-alkanes/OC x $\delta^{13}C$ $\delta^{13}C_{marine}$ = -19.1‰)	n-alkanes/OC x $\delta^{13}C$ $\delta^{13}C_{marine}$ = -26.0‰)
NDSS 1	83.00	61.00	71.00
NDSS 2	68.00	61.00	71.00
NDSS 3	85.00	70.00	78.00
NDSS 4	66.00	63.00	73.00
NDSS 5	89.00	73.00	80.00
NDSS 6	81.00	72.00	79.00

Niger River than in the Imo River. Total n-alkanes (in mg/L) decrease seawards in both estuaries (Fig. 3). Except for some stations of the eastern NDSS toward the Imo River, n-alkane particle loads (in mg/g) are relatively constant, generally around 10 mg/g (Fig. 3), indicating the rather homogeneous composition of riverine SM. This translates into a high correlation between n-alkane concentrations and OC or SM values (r>0.95, P<0.001, for both transects) and suggests that soil erosion and riverine hydrodynamics are the major factors controlling the aquatic distribution of n-alkanes. Enhanced concentrations (in mg/g) are found in the eastern NDSS toward the Imo River, mainly at stations NDSS 2, NDSS 4 and NDSS 5 (> 20mg/g).

Prevailing odd and high molecular weight ($\geq C_{25}$) n-alkanes indicate major vascular plant wax inputs. n-C_{27} is dominant and carbon preference index values (CPI_{24-32}) are generally > 3 (Table 1). The occurrence of alkylated polycyclic aromatic hydrocarbons, such as octahydrochrysenes and tetrahydropicenes, generated from the aromatization of 3-hydroxy triterpenoids and other constituents of higher plants, further supports the hypothesis of a dominant terrestrial origin (Fernandes, 1996; Chairi, 2018). Ekweozor & Telnǽs (1990) also reported presence of oleananes on sediments from Niger delta indicating that OM is terrestrially derived. Omontese *et al*. (2019) used Odd- Over- Even preference (0.23 -2.10), CPI values (1.09 - 1.61) and Oxygen index (53.33mgHC/gTOC) to designate the contribution of organic matter is terrestrially derived. The identification polyaromatic hydrocarbon on terrestrial organic matter and its absence in marine is an indication that most of the organic matters on the shelf are controlled by land derived source (Uzoegbu *et al*., 2018).

Sediments

Only a small portion of the OC transported by the Niger and Imo rivers SM were preserved in the sediments. OC percentages in both rivers are relatively constant at around 1.5% (n=17) (Fig. 4). OC percentages in sediments of the NDSS are significantly lower (average of 0.7%, n=6), but close to the average OC concentrations of fine-grained shelf and delta sediments (0.75%) calculated for world rivers by Berner (1982). Besides direct inputs from soil erosion, the OC content of shelf sediments may also reflect better preservation of OM in fine fluvial particles which bear the highest mineral surface areas (Prahl & Carpenter, 1983; Prahl, 1985; Mayer, 1994a, 1994b; Keil *et al*., 1998; Kandasamy & Nath, 2016).

$\delta^{13}C$ values determined from sediments of both rivers (Table 3) are close to the isotopic signature of continental C_3 vegetation (e.g. GonÄ i *et al*., 1997, 1998; Ruttenberg and GonÄ i, 1997). High C/N ratios suggest that the contribution of plankton (C/N<6; McConnaughey & McRoy, 1979a; Meybeck, 1982; Wu *et al*., 2019) is minor (Fig. 4). The results of these ratios $\delta^{13}C$ (from - 29.0 to - 24.4) and C/N (from 7 to 13) values for sediments in the same area ($\delta^{13}C$ from - 28.5 to - 24.4, C/N from 5 to 14) suggest that a major portion of the OC preserved in the sediments is of terrestrial origin.

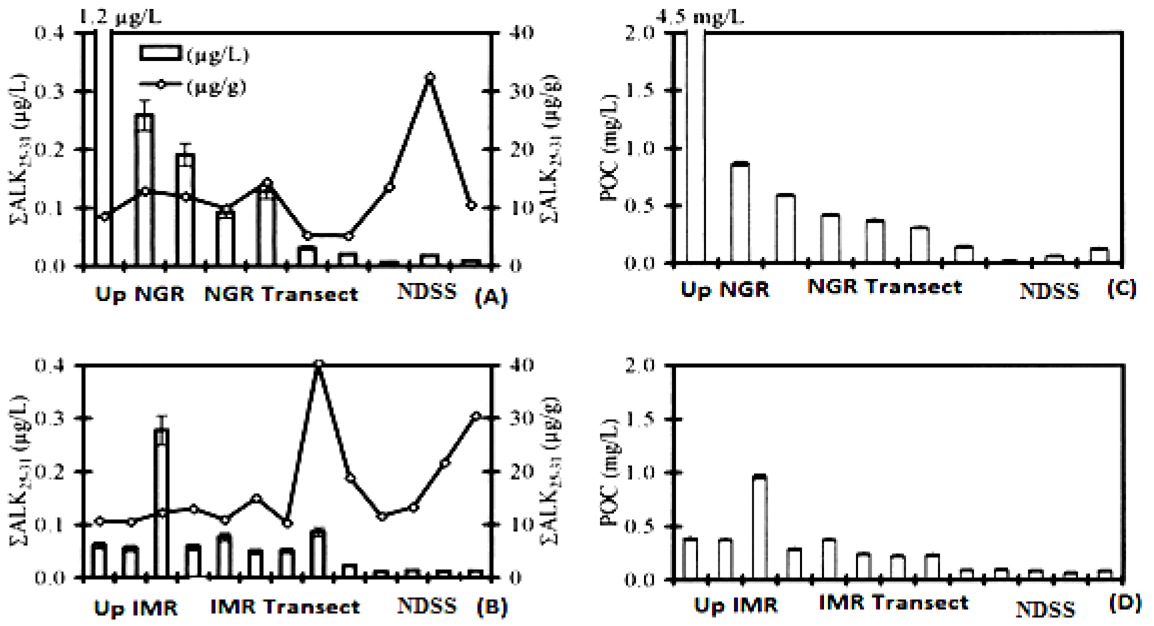


Figure 3: Total particulate concentrations of odd n-alkanes from C_{25} to C_{31} ($\Sigma ALK_{25\text{-}31}$ in mg/L and mg/g) along the Niger (a) and Imo (b) transects. POC data (in mg/L) also showed from upriver the Niger (c) and Imo (d) rivers to the Niger Delta Sea Shelf.

Values in the NDSS is around 4 µg/g, i.e. lower than those recorded in the Canadian Mackenzie Shelf and in the Eurasian Lena Delta, but higher than in Alaskan Arctic shelves where river inputs are smaller (Fernandes and Sicre, 2000). As for SM, the ratio of odd n-alkanes from C_{25} to C_{31} to OC is relatively constant in the estuary.

However, concentrations are quite variable in space (Fig. 4). High values, mainly in the mixing zone, indicate an area of preferential deposition (Fig. 4). River runoff and estuarine mixing are, in general, the dominant factors controlling particle deposition/resuspension episodes (Fernandes & Sicre, 2000; Szczepanska *et al*., 2012).

n-C_{27} is the dominant sedimentary n-alkane, as also observed in other Eurasian shelves such as the Barents Sea (Yunker *et al*., 1996) and the Laptev Sea (Broyelle, 1997). At the easternmost marine stations (NDSS 1 and NDSS 4), n-C_{31} prevails. Yunker *et al.* (1991, 1993) reported the dominance of n-C_{31} in peat samples of the Mackenzie Basin and explained the shift from n-C_{27} to n-C_{29}/n-C_{31} in the outer estuarine sediments of the Mackenzie River by selective deposition of peat particles towards the outer shelf. Transport by nepheloid brines (Pfirman *et al*., 1995; Bao et al., 2018) could also account for the presence of fine low density peat inputs offshore.

Total n-alkane particle content in the eastern Eurasian Lena Delta and Laptev Sea are substantially higher than values found in this work (Table 2).

Compositional features indicate significant planktonic inputs (Broyelle, 1997) that are not observed in the NDSS, at least not during this cruise. Such inputs may account partially for the observed differences in concentrations with respect to our data. At the freshet of the Mackenzie River, with a similar basin cover (vegetation and soil), values are lower than ours (Table 2), but n-alkane and SM levels are also significantly correlated (Yunker *et al*., 1991; Mayer *et al*., 2007; Chairi, 2018).

The dominance of n-C_{31} in sediments of the Arctic Ocean has been attributed to similar mechanisms by Schubert & Stein (1996). According to Yunker *et al*. (1991, 1993) peat inputs would be a seasonal phenomena thus, more easily observed in the sediments than in the SM.

Sedimentary CPI_{24-32} and $\delta^{13}C$ values (Fig. 5) clearly discriminate the OM in both rivers. Sediments from the mixing zone have CPI_{24-32} values intermediate between those of the Niger and Imo while the $\delta^{13}C$ values tend to be heavier than they should be by mixing only. Further offshore, OC is even more enriched in ^{13}C (Table 2). Admixtures of marine constituents likely account for the carbon isotope composition changes, as also indicated by decreasing C/N ratios seawards (Fig. 4).

These features suggest the production and settling of OM enriched with N-containing compounds and in ^{13}C, most probably autochthonous. However, the CPI_{24-32} and $\delta^{13}C$ values indicate that the OM deposited in shelf sediments mainly originates from land erosion (Chairi, 2018).

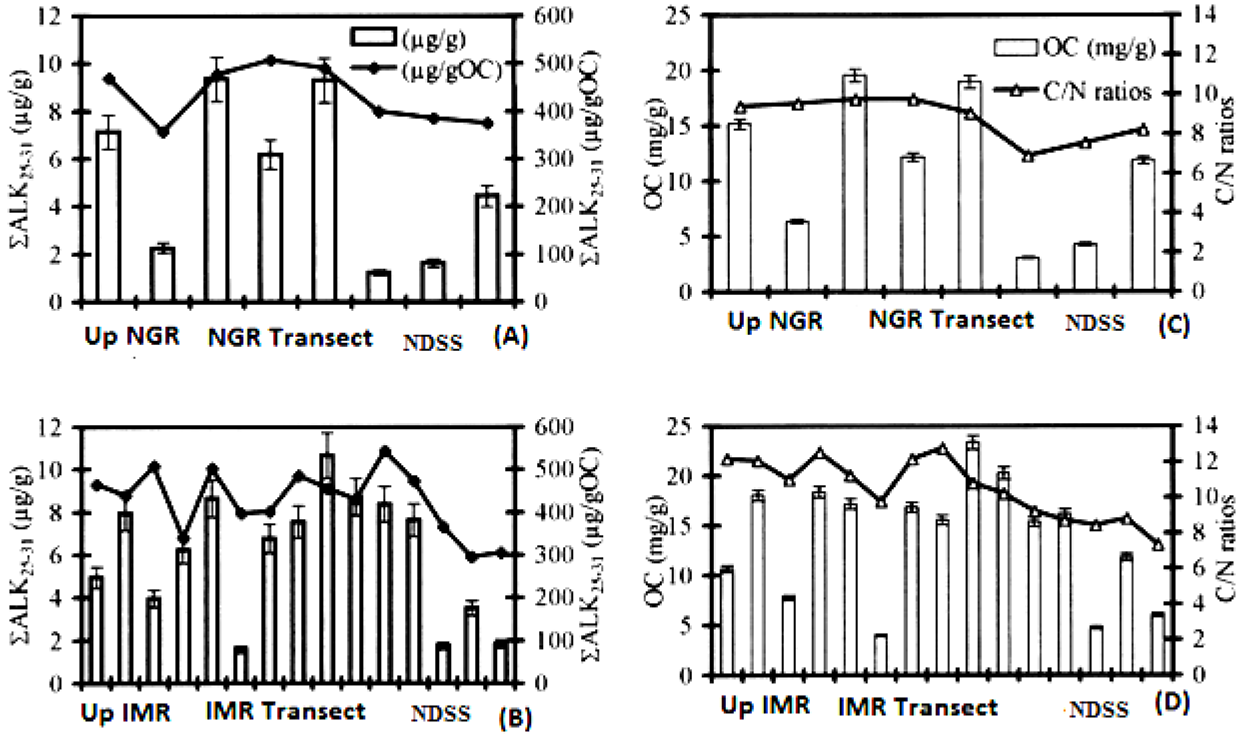


Figure 4: Total sedimentary levels of odd n-alkanes from C_{25} to C_{31} (ΣALK_{25-31} in mg/g and mg/gOC) along the Niger (a) and Imo (b) transects. OC levels (in mg/g) and C/N ratios in sediments collected along the estuaries of the Niger (c) and Imo (d) rivers are also plotted from upriver to the Niger Delta Sea Shelf.

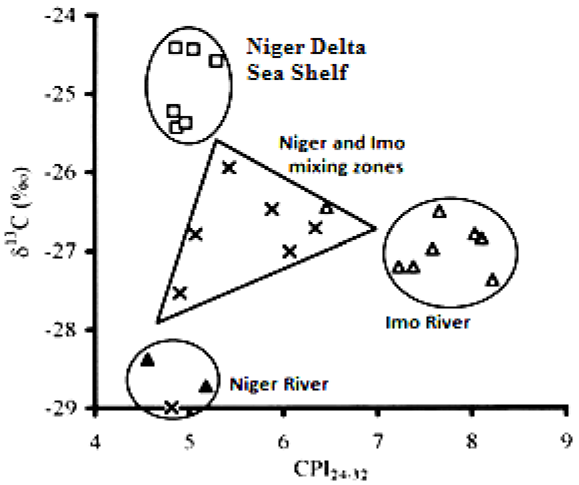


Figure 5: δ^{13}C vs. CPI$_{24-32}$ values in sediments along the Niger and Imo transects into the Niger Delta Sea Shelf.

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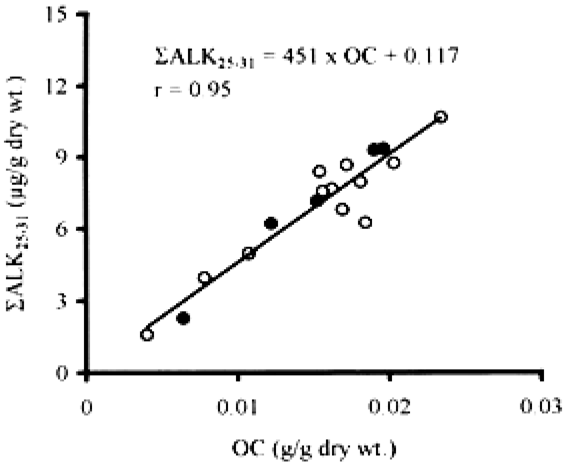

Figure 6: Terrigenous odd n-alkanes from C$_{25}$ to C$_{31}$ (ΣALK$_{25-31}$) vs. OC in Niger (black circles) and Imo (white circles) river sediments.

Terrestrial deposition

All the parameters used for this study emphasize the accumulation of continentally-derived material over the NDSS. In order to quantify the percentage of terrestrial OC sequestered in the sediments of the Atlantic Sea (%TERR). The equations of the linear fit between OC (in g/g dry wt) and terrigenous odd n-alkanes from C$_{25}$ to C$_{31}$ (ΣALK$_{25-31}$ in mg/g dry wt) for both rivers is given below and shown in Fig. 6:

$\Sigma ALK_{25-31} = 451 \text{ x } OC + 0{:}117 (r = 0{:}95, P < 0{:}001)$

This equation indicates that in the absence of terrigenous n-alkanes, OC concentrations tend to zero in the river, which implies that sedimentary fluvial OC is almost entirely terrigenous (Fig. 6). It also suggests that plant wax n-alkanes and terrestrial OC are exported from the mouth of both rivers in a fixed ratio, which equals the slope of this linear regression. The change of this ratio in shelf sediments would then reflect the dilution with marine OC that does not contain plant wax n-alkanes.

The slope defined by the Niger and Imo dataset (~450) is steeper than for the temperate Columbia River discharging along the Washington coast, around 280 (Prahl *et al*., 1994; Mayer et al., 2017). %TERR in the NDSS calculated from this method are comprised in the range 65-90% (Table 3).

The δ^{13}C riverine END-MEMBER is estimated taking into consideration the solid discharges of each river, 130 kg/s for the Imo and 504 kg/s for the Niger (Dai, 1995; Chairi, 2018):

$$\delta^{13}C_{riverine} = \frac{130}{(130 + 504)} \text{ x } \delta^{13}C_{Imo} + \frac{504}{(130 + 504)} \text{ x } \delta^{13}C_{Niger}$$

where $\delta^{13}C_{Imo}$ is the mean value for Imo sediments from stations IMR 13 to IMR 1 (-26.8‰) and $\delta^{13}C_{Niger}$ is the mean value for Niger sediments from stations NGR 7 to NGR 1 (-28.1‰). The riverine end-member calculated from this equation is -27.8‰. The drawback of this estimation is the lack of knowledge on the isotopic composition of the river-borne suspended particles that are actually being deposited on the shelf on a year-round basis. The absence of odd high molecular weight n-alkanes from C_{25} to C_{31} in the marine OM is a crucial assumption in the estimation of $\delta^{13}C_{marine}$. The absence of OC terrestrial origin for these compounds in the NDSS is supported by sedimentary alkane, alkanol and fatty acid fingerprints reported by Belyaeva *et al*. (1995) & Uzoegbu et al. (2019). The marine $\delta^{13}C$ END-MEMBER can be tentatively predicted from the x-intercept (for y = 0) of the linear regression of $\Sigma ALK_{25\text{-}31}/OC$ vs. $\delta^{13}C$ values in the marine sediments (Fig. 6). The equation obtained from the linear fit of the six Atlantic Sea sediments is:

$\Sigma ALK_{25\text{-}31}/OC = -61{:}4 \text{ x } \delta^{13}C - 1175$ ($r = -0.68$, $P < 0{:}5$)

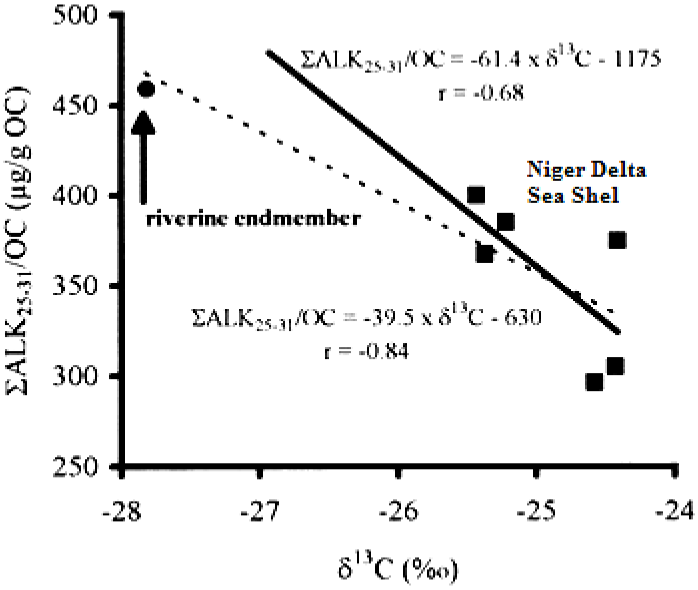

Figure 6: Terrigenous odd n-alkanes from C_{25} to C_{31} ($\_ALK_{25\text{-}31}/$ OC) vs. $\delta^{13}C$ values in Niger Delta Sea sediments. The bold line indicates the linear regression obtained from the six Niger Delta Sea Shelf sediments. The dashed line indicates the linear regression obtained from the six Niger Delta Sea Shelf sediments and the mean riverine END-MEMBER (after Fernandes and Sicre, 2000).

The marine $\delta^{13}C$ END-MEMBER derived from this equation is -19.1%o. However, this regression line passes well over the riverine END-MEMBER (Fig. 7) and the correlation is poor. If we include the mean riverine END-MEMBER in the data set ($\delta^{13}C$ = -27.8‰), the correlation coefficient rises to -0.84 ($P<0.05$), but the regression line ($SALK_{25\text{-}31}/OC= -39.5 \text{ x } \delta^{13}C - 630$) leads to a heavier marine END-MEMBER (-16.0‰).

For comparison, average values previously reported for Bering Sea are -24.4%o for phytoplankton, -22.1‰ for zooplankton and ~-22‰ in sediments (McConnaughey & McRoy, 1979a, 1979b). However, planktonic $\delta^{13}C$ values are known to span a wide range (-10 to -31‰) depending on species and environmental conditions (Wong & Sackett, 1978; Showers & Angle, 1986; Zhange *et al*., 2017).

At high latitudes the $\delta^{13}C$ of phytoplankton is expected to be more depleted in ^{13}C than found from our calculations due to a higher solubility of dissolved CO_2 in cold waters and thus enhanced isotopic fractionation (Sackett *et al*., 1965; Fontugne & Duplessy, 1981; Bao *et al*., 2018; Wu *et al*., 2019). However, in coastal environments temperature may not be the only or dominant factor affecting the carbon isotopic ratio of autochthonous matter.

The isotopic composition of dissolved inorganic carbon (DIC) may also play a part. Cifuentes *et al*. (1988) reported values as high as -16.6‰ in the SM of the lower Delaware estuary in spring, where biomass and primary production rates were high. This result was attributed in part to the $\delta^{13}C$ value of the DIC, but mostly to decreasing isotopic fractionation as diffusion of CO_2 across the cell becomes the rate-limiting step of photosynthesis. These factors possibly explain the heavy $\delta^{13}C$ values of the marine END-MEMBER calculated here.

However, inventories of $\delta^{13}C$ signatures of autochthonous organisms growing in coastal environments would be necessary to better constrain binary dilution models.

Being aware of the difficulties associated with accurate definition of END-MEMBERS, we calculated the %TERR in coastal sediments using the estimated riverine END-MEMBER (-27.8‰) and the two marine end-members (-19.1 and -16.0%o) derived from our calculations (Table 3). Estimates of the %TERR are only about 10% different depending on the marine END-MEMBER used. Those derived from $\delta^{13}C_{marine}$ = -16.0‰ fall in the same range as the values calculated by the first method. They indicate that approximately 70-80% of the OC present in NDSS sediments is of terrestrial origin. Fernandes and Sicre (2000) calculated %TERR >50 for the Kara Sea based on sedimentary isotopic signatures. Recent work has also shown that OM accumulating in other Eurasian shelves (e.g. Kuptsov and Lisitsin, 1996; Yunker *et al*., 1996; Broyelle, 1997; Fahl and Stein, 1997; Zhange *et al*., 2017) and on the west Beaufort Sea (Shaw *et al*., 1979; Steinhauer and Boehm, 1992) is mainly terrigenous although this has not been quantified. The %TERR values calculated for the Columbia shelf using terrigenous n-alkane and OC data average 59%, while those calculated from $\delta^{13}C$ values average 67% (Prahl *et al*., 1994). On the tropical Amazon shelf, %TERR estimates based on sediment accumulation rates, OC and $\delta^{13}C$ values are around 70 % (Showers and Angle, 1986; Mayer *et al*., 2017).

CONCLUSIONS

Geochemical data provided evidence of the strong influence of the Niger and Imo rivers discharges more than 50km offshore. These rivers supply terrigenous n-alkanes to the NDSS in a relatively constant ratio to OC. n-Alkane/OC values are not statistically different for the Niger and Imo, but values previously reported for the temperate Columbia River are notably lower. These results indicated the geographical specificity of this ratio, which need to be empirically evaluated for each coastal system.

Based on n-alkane, OC and $\delta^{13}C$ values, the contribution of terrestrial OC in shelf sediments was estimated to be around 70% or more of the total sedimentary OC. The values from this research fall within the range calculated for other tropical or temperate continental margins such as River Nile and Bering Sea under the influence of large rivers, despite differences in the composition of river-borne OM (e.g. different n-alkane/OC ratios).

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