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Assessment of Sediment Organic Matter Transportation Using Stable Isotope Analysis

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Abstract. Sedimentation is one of the major potential stressors to coral reef ecosystems. Our study was conducted in the Berau, East Kalimantan, Indonesia which is known as a part of the Coral Triangle in West Pacific, in order to trace the transportation of sediment organic matter to the coral reef areas using stable isotopes. The study area divided into three Transects: Transect 1, 2, and 3, representing the northern, middle, and southern parts of the Berau River mouth. Field work was carried out during all seasons and a total of 126 sediment and 129 particulate organic matter samples were collected. Results are that the $\delta^{13}C$ during the rainy and shifting season had lower values than the dry season, probably because of increased oil erosion during the rainy and shifting seasons with heavy rainfall. About 10% of the total sediment samples had $\delta^{13}C$ values higher than -23‰, indicating that most of sediments sampled were of terrestrial origin. Spatially, terrestrial sediments had reached around 60 km, 80 km and 70 km from the river zero point along Transects 1-3 respectively. $\delta^{15}N$ values of Particulate Organic Matters in the Transect 1 during the rainy season were variable and higher (around +10‰ to +20‰) in some points around coal mining and power plant sites, and some villages. The nitrogenous organic matter is interpreted as allochthonous and sourced from areas of anthropogenic impact and agricultural activities, which produce isotopically higher organic nitrogen.

Key words: Coral reef, Sedimentation, Carbon and Nitrogen Isotopes, Allochthonous.

Introduction

Coral reefs are one of the most productive ecosystems in the world, rivaling rain forest in their richness of life (Birkeland 1997). It is home 4000 different fish, 700 species of corals, and thousands of other plants and animals (Hinrichsen 1997). Coral reefs also provide environmental and economic benefits to millions of people by protecting shorelines, providing foods, building materials, pharmaceuticals, creating employment, recreational, and tourism opportunities.

However, coral reefs are declining in many areas of the world due to steadily increasing threats from direct human pressures (Pastorok and Bilyard 1985; Fabricius 2005) and indirect effects of global climate change (Hoegh-Guldberg *et al*. 2007). Sedimentation is a major cause of mortality in the initial life stages of hard corals (Fabricius *et al.* 2003). High levels of sedimentation due to terrestrial erosion have severely degraded many coastal reefs around the world. Fabricius (2005) stated that most of sediments were imported into coastal marine systems via rivers, with more than 95% of the coarser grain fractions being deposited within a few kilometers of the river mouth, while fine grains can be transported up to longer distances.

Our study aimed to trace transport of sediment organic matter from the river to estuary area and to assess how far terrestrial sediments reach the coastal area using $\delta^{13}C$ and $\delta^{15}N$ analysis in the Berau Regency, East Kalimantan, Indonesia.

The Berau River starts at Tanjung Redeb, the capital city of Berau Regency, and converges in the estuary to the Sulu Sulawesi Seas. Buschman (2007) estimated that the average sediment concentration of the river water was 50 mg/l and estimated that every year about 2,000,000 tons of sediment exported to the coastal area. The Berau estuary is dynamic systems like other estuaries, and the physical or chemical conditions had sometimes with extreme changes. The estuary has semidiurnal type of tide with tidal range was 2.5 m (Susanto 2009).

Even though it is difficult to trace transportation of the organic matter in an estuary area because of multiple sources, Hoffman and Bronk (2006) traced the sources of the organic matter through a watershed or an entire season using carbon and nitrogen isotope composition of suspended particulate organic matter (POM) in the brackish and tidal water regions of the Mattaponi River, Virginia. They observed that fresh water POM had a high C:N ratio (>12), depleted carbon isotopic composition ($\delta^{13}C_{POC}$, -26‰ to -30‰), and depleted nitrogen isotopic composition ($\delta^{15}N_{PN}$, 2‰ to 10‰). Brackish water POM had lower C:N ratio and enriched $\delta^{13}C_{POC}$ (-24‰ to -27‰) and $\delta^{15}N_{PN}$ (7‰ to 15‰).

Material and Methods

POM samples were collected from 46 points of transects 1, 2 and 3, representing northern, middle, and southern parts of the Berau River mouth, East Kalimantan Province of Indonesia (Figure 1). Transect 1 (19 sampling points) is utilized as the main route for transportation. Some villages, agricultural activities, logging port, power plant, coal processing and loading, as well as shrimp pond development existed in this transect. Transect 2 (17 points) is dominated by shrimp/fish ponds; mangroves are being converted for industrial activities and few people lived in the surrounding area. Transect 3 (10 points) is characterized by mangrove forest, few fishing activities, and many channels at the outskirts of the estuary.

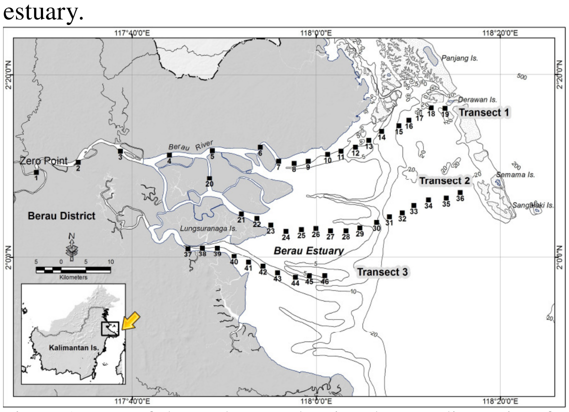

Figure 1: Map of the study area showing the sampling points for sediment and POM along Transect 1, Transect 2, and Transect 3.

A total of 129 POM samples during the dry season 2006 (45 samples), the rainy season 2007 (29 samples), and the shifting season 2007 (35 samples) were collected in the estuary area and marine water. Mean rainfall rate during dry, rainy, and shifting seasons were 83.5 mm, 170.8 mm, and 190.2 mm, respectively.

POMs were collected using a plankton net of 100 µm mesh size which was towed near the water surface. All samples were kept in the plastic bottle with absolute ethanol before transportation to Kyushu University for analysis.

A representative sub-sample of each POM sample were photographed using a microscope. The POM sample then was centrifuged at 3000 rpm for ten minutes to remove supernatant. The precipitate sample was decalcified with 0.05N HCl to remove any inorganic carbonate, and then rinsed twice using Deionized Distilled Water. The sample was prefrozen in a deep freeze for minimum ten minutes and then were freeze-dried overnight by using an Eyela freeze dryer (Tokyo-Rika, FDU 506). 0.80 ± 0.05 mg (two analyses for each POM sample) was measured and placed into tin capsule for the isotope measurements. $\delta^{13}C$ and $\delta^{15}N$ values were measured using a continues-flow stable isotope ratio mass-spectrometer (ANCA-mass 20-20, Europe Scientific Instruments, UK) with glycine and citric acid as running standards. Measurement errors are within 0.1‰ for $\delta^{13}C$ and 0.3‰ for $\delta^{15}N$. Where values between two analyses were larger than the measurement errors, the samples were re-measured.

Sediment samples were collected using an Ekman grab sampler. A total of 102 sediment samples were collected during the dry season (41 samples), the rainy season (29 samples), and the shifting season (32 samples) from the same points with POM, except for a few locations where the sampler returned empty. Sediments were kept in plastic bottles with ethanol and transported to the laboratory for further analysis.

First, visible and non-sedimentary inclusions (e.g. shell fragments) were removed manually. Sediment samples were decalcified using 0.02 N HCl for the river and estuary samples, and using 0.1 N HCl for the marine samples in order to remove any inorganic carbonates in the samples (Salomons and Mook, 1981). Then samples were freeze-dried overnight and powdered with a porcelain mortar and pestle. The samples were passed through a 0.7 mm mesh sieve, measured, and placed into tin capsule. Results from the first measurement was calculated for minimum volume of 100 µg / N and measured into tin capsule for the second measurement.

Results

Carbon contents of POMs during the dry season (mean 22.9±2.9%) were always higher compared to those of the rainy (mean 11.9±5.0%) ($F_{32,21} = 3.63$, $p < 0.0014$) and shifting seasons (mean 6.9±3.0%) ($F_{32,24} = 2.51$, $p < 0.0112$). Mean nitrogen content in the POMs collected during the dry season was 3.9±0.8% higher than those collected during the rainy season (mean 1.9±1.0%) ($F_{32,21} = 4.26$, $p < 0.0005$) and the shifting season (mean 0.9±0.5%) ($F_{32,24} = 3.02$, $p < 0.0033$). However, mean C:N ratio during the dry season (6.3±0.8) was not statistically different than those of the rainy (6.7±1.2) and shifting (8.2±1.4) seasons (ANOVA, p>0.05).

Table 1 shows C:N, $\delta^{13}C$ and $\delta^{15}N$ of POMs and sediments during dry, rainy, and shifting seasons. Mean $\delta^{13}C$ value of POM samples during the dry season was slightly lower than those of the rainy and the shifting seasons, although these differences were not significant (ANOVA, $p > 0.05$). $\delta^{13}C$ values of POMs had a close relationship with salinity during all seasons average r^2=0.83 in dry season, r^2=0.69 for rainy, and r^2=0.95 for shifting seasons. Moreover, $\delta^{13}C$ values of sediments had also a close relationship with salinity during all seasons but with lower correlation coefficients (r^2=0.38 in dry season, r^2=0.46 in rainy season, and r^2=0.38 in shifting season).

	Dry season			Rainy season			Shifting season		
	C/N	$\delta^{13}C$	$\delta^{15}N$	C/N	$\delta^{13}C$	$\delta^{15}N$	C/N	$\delta^{13}C$	$\delta^{15}N$
POMs									
minimum	4.1	-30.0	1.0	5.6	-27.2	1.1	5.1	-29.9	-2.9
maximum	16.0	-14.5	7.7	10.0	-18.0	17.3	23.3	-18.3	7.0
average	6.3	-22.4	5.0	6.7	-20.2	5.4	8.2	-21.6	2.6
STDev	0.8	2.4	1.4	1.2	1.8	0.5	1.4	0.8	1.0
SEDIMENTS									
minimum	7.7	-28.9	-3.5	8.4	-29.3	-4.0	10.1	-28.5	-1.4
maximum	17.5	-21.3	1.9	21.8	-21.1	3.0	29.8	-14.9	2.7
average	13.9	-26.7	0.8	15.2	-26.9	-0.4	14.2	-26.4	0.1
STDev	2.0	2.1	1.3	3.5	2.4	1.9	3.9	2.7	0.8

Table 1. C/N, $\delta^{13}C$ (‰) and $\delta^{15}N$ (‰) of POMs and sediments during the dry, rainy, and shifting seasons

$\delta^{15}N$ values of POMs during the shifting season were lower than those of the dry and rainy seasons. Mean $\delta^{15}N$ values of POMs were 5.0±1.4‰ during the dry season, 5.4±0.5‰ during the rainy season (sampling point 5, 7, 9, 11 were excluded in the mean calculation), and 2.6±1.0‰ during the shifting season (Figure 2). Wide ranges of POM $\delta^{15}N$ during the rainy season were caused by $\delta^{15}N$ values of point 5, 7, 9, 11 in the Tract 1 which were 12,0‰, 17.3‰, 12.2‰, and 13.1‰, respectively. These points were near the location of coal processing and loading, logging port, agricultural field, shrimp pond development, and the villages, which may contribute to the high $\delta^{15}N$ values.

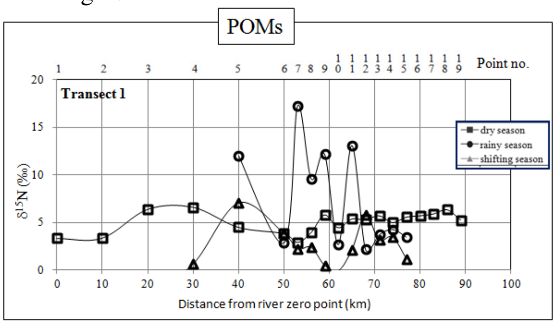

Figure 2: $\delta^{15}N$ values of POMs in the Transect 1 during the dry (□), rainy (○), and shifting (△) seasons.

Sediments contained much less organic matter than POMs with carbon contents ranged between 0.4% and 3.3% along Transect 1, 0.3% and 10.1% in Transect 2, and 0.8% and 4.7% in Transect 3. The trend of organic carbon content decreased seaward, suggesting that carbon content in the terrestrial sediments were higher than marine sediments.

The mean value of nitrogen content among transects was 0.2±0.1%; 0.4% or less in Transect 1, 0.5% or less in Transect 2, and between 0.1% and 0.3% in Transect 3. Consequently, C-N ratios of Transect 2 (14.8±3.1) and Transect 3 (14.9±3.9) were slightly higher than Transect 1 (13.6±2.9). C-N ratio during rainy season (15.2±3.5) was higher than those of shifting (14.2±3.9) and dry (13.9±2.0) seasons. However, these spatial and seasonal differences were not statistically significant (ANOVA, p>0.05).

Figure 3 shows $\delta^{13}C$ and $\delta^{15}N$ values of sediments in three transects. In Transect 1, $\delta^{13}C$ values of sediments ranged from -28.9‰ to -14.9‰. Starting from point 11 in Transect 1 (66 km from river zero point) seaward, it seems that the origin of sediments is not terrestrial anymore. Sediments were mostly mixed with marine organic matter, resulting in $\delta^{13}C$ values of these points to be higher than -23.0‰. For Transect 2 and Transect 3, most $\delta^{13}C$ values showed terrestrial in origins with mean values of -26.8±2.0‰ and 27.1±1.2‰, respectively.

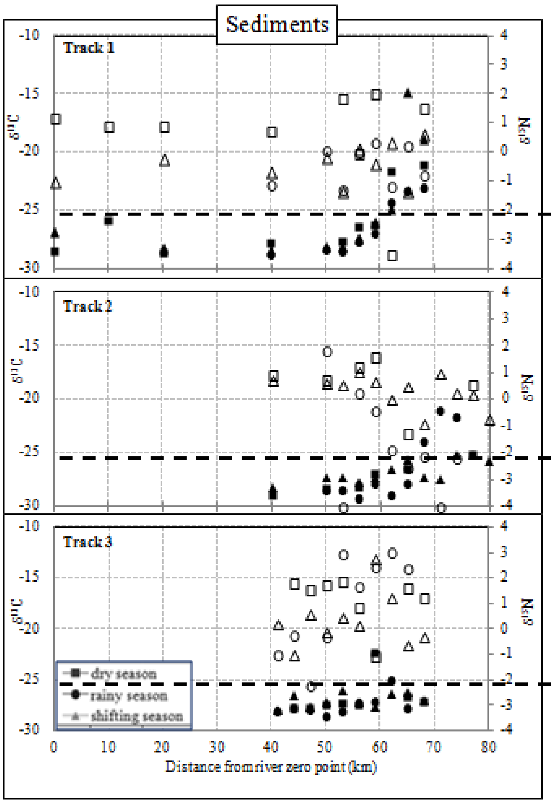

Figure 3: $\delta^{13}C$ (‰, filled) and $\delta^{15}N$ (‰, open) values of POMs in the Transect 1, Transect 2, and Transect 3 during the dry, rainy, and shifting seasons.

$\delta^{15}N$ values of sediments ranged from -3.5‰ to +2.0‰ in Transect 1, from -4 to +1.8 in Transect 2, and from -2.2 to +3.0 in Transect 3. Standard $\delta^{15}N$ value of sediment is 0‰. Heavy $\delta^{15}N$ values than the standard must be an indication of high nitrification process, while $\delta^{15}N$ values which were lower than the standard value may indicate de-nitrification process by organic matter.

There is no different on seasonal changes of organic carbon sediments. Mean carbon contents in sediment during the dry, rainy, and shifting seasons were 2.3±0.9%, 2.6±2.0%, and 2.6±0.5%, respectively. Moreover, $\delta^{13}C$ values of sediments varied from -28.9‰ to -21.3‰ during the dry season, from -29.3‰ to -21.1‰ during the rainy season, and from -28.5‰ to -14.9‰ during the shifting season. Although mean $\delta^{13}C$ value of sediments during the rainy season was lower (-26.9±2.4‰) than those during the dry (-26.7±2.1‰) and shifting (-26.4±2.7‰) seasons, there were no significant differences of sediment condition during the dry, rainy, and shifting seasons (ANOVA $p>0.05$).

Based on three-time separate surveys, $\delta^{13}C$ values of sediments showed that the terrestrial sediments have reached around 60 km, 80 km and 70 km from the river zero point along Transects 1-3 respectively. In the Transect 2 and Transect 3, majority of the sediment samples had lower $\delta^{13}C$ values than -23‰.

Discussion

Daily tidal range and river flow are two main factors that influenced variation of the Berau estuary. During high tide, the estuary area was dominated by marine organic matter, while during low tide the estuary area indicated mixing with terrestrial and marine organic matter.

$\delta^{13}C$ values of POMs gradually increased towards offshore and C-N ratio also decreased. Most POMs exhibited mixing of organic matter, whose $\delta^{13}C$ values were in the range of -19.5‰ to -26.0‰. POMs from points 1 to 5 of Transect 1 and point 20 of Transect 2 indicated terrestrial organic matter with $\delta^{13}C$ < -26.0‰. $\delta^{13}C$ values of fresh waters vary widely depending on the source of dissolved CO_2 in the waters; where respiration inputs are strong in the limited environments, $\delta^{13}C$ may reach -20‰ (Peterson and Fry, 1987). $\delta^{13}C$ values of POMs were between -18‰ and -24‰ with a mean -21‰. During algal blooming POMs showed higher values -12.8‰ to -16.8‰. POMs of fresh and brackish waters have lower $\delta^{13}C$ than marine POMs, ranging from -24‰ to -30‰. $\delta^{13}C$ values for terrestrial C3 plant detritus (-26‰) may contribute to these lower values (Peterson and Fry, 1987). Based on these results, $\delta^{13}C$ of POMs in the estuary contributed from mangrove detritus, during low tide in particular.

In Transect 1, $\delta^{13}C$ value of POMs had close relationship with salinity during the dry and shifting seasons, while in Transect 2 and Transect 3, $\delta^{13}C$ values of POMs had close relationship with salinity during the dry and shifting seasons. It indicates that $\delta^{13}C$ values of POMs are more stable during the dry and shifting seasons than the rainy season. Mixing of the terrestrial and marine organic matter was relatively high during the rainy season which might be due to heavy rainfall, high runoff and river discharge. In aquatic ecosystems, $\delta^{13}C$ values of plants are determined by three factors: the isotopic composition of the dissolved inorganic carbon (DIC) pool, the isotopic discrimination of the enzyme responsible for carbon fixation, and the intracellular concentration of CO_2 or HCO_3^- that is the active species fixed by the carboxylating enzyme (Fry and Sherr, 1984). Our study confirmed that $\delta^{13}C$ values of POMs mostly affected by the three factors.

The C:N ratio of POMs during the dry season was slightly but not significantly lower than those during the rainy and shifting seasons as a result of increase in the organic nitrogen fraction binding to proteins and decrease in the $\delta^{13}C$ values. This indicates that the dry season had higher primary productivity than the other seasons. The rainy and shifting seasons with high rainfall generate high soil erosion, resulting in an increased supply of terrestrial organic matter, indicated by lower $\delta^{13}C$ values (Junge *et al.*, 2005).

$\delta^{15}N$ values of POMs along Transect 1 during the rainy season were more variable and were higher at some locations around coal mining, power plant sites and some villages, indicating an effect of anthropogenic impact and agricultural activities, which produce isotopically high residual organic nitrogen; $\delta^{15}N$ around +10‰ to +20‰ (McClelland *et al.*, 1997; Peters *et al.*, 1978).

There is a gradient from generally lower $\delta^{15}N$ value of terrestrial organic matter to higher of marine origin. The highest values are at 20-30 km from the zero point. This tendency showed only during the dry season rather than other seasons, which may be explained by the mixing of terrestrial organic matter and the marine POMs (Kumar *et al.*, 2005). This mixing of organic matter occurred during the rainy and shifting seasons must be influenced by large volume of rainfall and river water discharge.

Generally, $\delta^{15}N$ values of POMs decrease toward offshore, however, biogeochemical processes influence $\delta^{15}N$ value of POM distributions, in the estuary in particular, where has greater physical mixing between terrestrial and marine inputs (Cifuentes *et al.*, 1988). Ground water may be influenced only by atmospheric deposition typically ranging from 2‰ to 8‰, while $\delta^{15}N$ value is higher when affected by human and animal wastes (+10‰ to +20‰), and nitrate from synthetic fertilizer is more depleted in δ^{15}N value (-3‰ to +3‰) (McClelland *et al.,* 1997).

The lower POM δ^{15}N values during the shifting season may be attributed to the contribution of terrestrial sources of particulate organic nitrogen (Kumar *et al.*, 2005), which might be connected to the higher rainfall rate during the shifting season. Rainfall rate during the shifting season was higher (mean 190.2 mm) than those during the rainy (mean 170.8 mm) and the dry (mean 83.5 mm) seasons. Although there were trend that organic carbon contents decreased and δ^{13}C values of sediments became higher toward offshore, results of this study showed little variation in δ^{13}C values of sediments either within or among Transects. From 80 sediment samples analyzed from river to estuary area, only 11% (9 samples) have higher δ^{13}C value than -23‰ which indicate a border separating terrestrial sources from marine origin. Newman *et al.* (1973) measured δ^{13}C of mud samples which were terrestrial in origin (δ^{13}C=-23‰ to -25‰), and oozes which were marine in origin (δ^{13}C=-17‰ to -21‰). These results indicate that about 90% of the sediments sampled in all Transects were terrestrial in origin. The rest of the samples were not pure sediments but rather mixed with sand and rubble coral. However, based on Wada *et al.* (1987), -23‰ of the δ^{13}C of value would indicate that about 50% of the sedimentary organic carbon is terrestrial in origin. By using an equation developed by Wada *et al.* (1987), the value of -26.5‰ reflects that 100% of sedimentary organic carbon is terrestrial in origin. In this case, sediment organic matter showed that 80% of samples were terrestrial in origin.

δ^{15}N values of sediments during the dry season were higher than other two seasons. During the rainy and shifting seasons, δ^{15}N values of sediments tend to be lower than standard value (0‰), which may affected by increased de-nitrification. Montoya (2007) noted that the de-nitrification can reduce the δ^{15}N value by up to 2-3‰. Furthermore, this study shows that even POMs in the estuary indicated mixing area with terrestrial and marine waters, sedimentary organic matter was terrestrial in origin.

Most sediment accumulated in the Berau River estuary comes from soil erosion which is generated by forest clearing and coal mining in the upper land, mangrove conversion to ponds, agriculture activities around villages, sewages, and few from *in-situ* aquatic production.

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