Geochemical and Stable Isotopic Studies of Celebes and Sulu Sea Sediments: Sites 767 and 768, ODP Leg 124

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ABSTRACT

This paper presents the depth distributions of sediment organic carbon, total reduced sulfides, and carbon and oxygen isotopic compositions of carbonates from Sites 767 and 768, ODP Leg 124 in the Celebes and Sulu Sea Basins. Carbonate, organic carbon and sulfide profiles have probably been complicated by changes in sediment organic source input and mode of sedimentation as well as variations in carbonate content during the tectonic development of the basins. $\delta^{18}O$ and $\delta^{13}C$ of carbonates both generally become lighter with increasing depth. $\delta^{13}C$ is more variable than $\delta^{18}O$. The progressive decrease in $\delta^{18}O$ with depth may indicate diagenetic formation of carbonates at elevated temperature induced by deep burial and volcanic activity. The highly variable and negative $\delta^{13}C$ values in the carbonates indicate an organically-derived CO_2 origin of carbon in diagenetic carbonate components.

1. INTRODUCTION

The Sulu and Celebes seas in the western Pacific are two semirestricted marginal basins located between the Borneo to the west and the Philippine archipelago to the east (Figure 1; Rangin, Silver, von Breymann *et al.*, 1990). In the region, the Sulu Sea is further separated from the Celebes Sea by a major bathymetric high, the Sulu Ridge. On ODP Leg 124 two cores were drilled in the Sulu Sea (Site 768) and Celebes Sea (Site 767) at a water depth of 4395 and 4916 m, respectively. Preliminary shipboard study of the core lithology revealed that sediment accumulation in the basins was characteristic of sharp changes in environment and processes of deposition and hence, implied a very different history of tectonic development of the basins. The Celebes Sea originated in an open ocean setting in the middle Eocene (42 Ma), whereas the

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Fig. 1. Geotectonic map of the Sulu and Celebes sea basins and the location of the Site 767 and 768 drilled during ODP Leg 124 (from Rangin, Silver, von Breymann *et al.*, 1990).

Sulu Sea seems to have formed from a back-arc spreading in the late-early to early-middle Miocene (20 Ma). Furthermore, the deep water in the Sulu Sea is presently depleted of dissolved oxygen (< 1.6 ml/L) and warmer (~ 10°C) as compared to the normal oceanic water (Exon *et al.*, 1981; Linsley *et al.*, 1985).

This study analyzed sediment carbonate, organic carbon and total reduced sulfur contents, and oxygen and carbon stable isotopic compositions of carbonates of cores from Sites 767 and 768 in an attempt to test the feasibility of using sediment geochemical and stable isotopic characteristics to temporally and spatially constrain the tectonic history of these two basins.

2. MATERIAL AND METHODS

Samples used for carbonate, organic carbon, and reduced sulfur analysis were selected from sediments that had been previously squeezed on shipboard to obtain pore water for inorganic geochemical analyses. Sediment samples were first disintegrated, washed thoroughly, then dried in the oven and ground to a powder. Sediment carbonate and total carbon concentrations were measured with a LECO carbon analyzer, and the difference between the two measurements was defined as organic carbon content (Heath *et al.*, 1977). Additionally, samples of low organic carbon content were further checked by direct digestion of sediment with 1N HCL and then measured with a LECO carbon analyzer as usual. Comparison on the results (data are not shown) from these two techniques showed that the acid digestion method gave a better precision than the combustion method.

Sediments samples used for isotopic analysis (see below) were also measured for their carbonate content. The amount of CO_2 evolved from 100% $H_3 PO_4$ treatment was measured and used to calculate carbonate concentration using a carbonate reagent standard. The result of this determination agrees well with that previously measured on shipboard (r = 0.94; Figure 2). Concentrations of total reduced sulfide were determined after the method of Vogel (1961) by treating the sediment with aqua regia and precipitating and weighing the resulting sulfate (Sheu and Presley, 1986; Sheu, 1987). Consequently, the reported sulfide data may thus contain all reduced sulfur components which are indistinguishable from each other due to the highly oxidizing nature of aqua regia treatment.

Carbon and oxygen isotope analyses of carbonates were conducted on selected shipboard samples which had been used for carbonate determination. Sample preparation and isotopic measurement followed the standard method of McCrea (1950). Isotopic analysis was preformed with a VG Micromass 903 mass spectrometer and results were reported in the conventional delta notation (δ) relative to the PDB standard of Craig (1957).



Fig. 2. Comparison of carbonate concentrations determined on shipboard (data from Rangin, Silver, von Breymann *et al.*, 1990) with those determined onshore in this study.

Precision of the reported analyses, based on replicates of samples, are carbonate ($\pm 1 wt\%$), organic carbon ($\pm 0.02 wt\%$), reduced sulfur ($\pm 0.02 wt\%$), $\delta^{13}C$ ($\pm 0.04 o/oo$), and $\delta^{18}O$ ($\pm 0.06 o/oo$).

3. RESULTS AND DISCUSSION

3.1 Sulfate Reduction and Sulfide Formation

Results of pore water sulfate and sediment organic carbon and reduced sulfide analyses are shown in Table 1 and plotted in Figure 3. Sulfate data were previously measured aboard ship by the author and reported in Rangin, Silver, von Breymann *et al.* (1990). At both sites, dissolved sulfate concentrations in the upper section of the cores decrease with increasing depth, indicative of sulfate reduction (Berner, 1964; Goldhaber and Kaplan, 1974). Sulfate concentration approaches a minimum at about 325 mbsf in Site 767 cores and at about 220 mbsf in site 768 cores, respectively. The depth of the minimal sulfate concentration measured in the cores coincides well with the onset of the methane production (Rangin, silver, von Breymann *et al.*, 1990). The mutual exclusion of sulfate reduction from methane production has long been observed in marine sediment cores (Martens and Berner, 1977; Claypool and Kaplan, 1974; Winfrey *et al.*, 1981), and the observed depth distribution of sulfate and methane in cores from sites 767 and 768 agrees well with those observations.

Sediment organic carbon and sulfide concentrations measured in different depths from Sites 767 and 768 vary considerably throughout the cores (Figure 3). Range, Silver, von Breymann *et al.* (1990) showed that rate of deposition of cores at Sites 767 and 768 decreased with depth within the sulfate reduction zone, and that of Site 767 was greater than Site 768 (average approximately 51 m/my. vs. 37 m/my.) In addition, previous shipboard examination of core lithology has revealed intriguing volcanic ash and turbidite layers in the cores, indicating a complicated history of sediment accumulation. Study of clay mineralogy also showed that the sediment within the sulfate reduction zone was composed of a mixture of terrigenous clays with clays of submarine volcanogenic origins. As a consequence, the lack of definite trends in the observed organic carbon and sulfide profiles can be attributed to changes in depositional processes during the tectonic development of the basins.

A variation in sediment source and depositional rate could further result in a change of the quantity and nature (i.e., reactivity) of organic matter utilized by the sulfate-reducing bacteria, and thus the rate of sulfate reduction and sulfide formation (Lyons and Gaudette, 1979; Westrich and Berner, 1984). The variation in organic matter distribution of the cores may also account for the observed minor variations in dissolved sulfate profile (e.g., Lasaga and Holland, 1976). Thus the characteristic distributions of sulfate, organic carbon, and Table 1.Carbonate, organic carbon, and total reduced sulfur analyses of sediments from
Holes in Sites 767 (Celebes Sea) and 768 (Sulu Sea). Pore-water sulfate data are
from Rangin, Silver, von Breymann, et al. (1990).

Core-section	Depth	Pore-water	Reduced	Organic	
(interval in cm)	(mbsf)	sulfate	sulfur	carbon	
124-		(mM)	(wt%)	(wt%)	
767B-1H-04,145-150	5.95	28.90	0.094	0.78	
2H-04,1 45- 150	14.95	27.20	0.048	0.32	
3H-04,145-150	24.45	25.70	0.253	0.82	
4H-04,145-150	33.95	25.00	0.080	0.56	
5H-04,145-150	43.45	24.40	0.088	0.33	
7H-04,145-150	62.40	23.90	0.183	0.93	
8H-03,145-150	70.40	23.50	0.099	0.20	
9H-04,145-150	77.45	23.10	0.081	0.15	
10H-04,145-150	86.95	22.50	0.128	0.15	
13X-01,145-150	111.05	22.80	0.082	0.32	
17X-02,145-150	151.25	19.20	0.215	0.15	
23X-02,145-150	206.25	14.80	0.159	0.49	
29X-04,145-150	267.35	7.50	0.225	0.17	
35X-04,145-150	325.30	3.00	n.d.	0.14	
41X-04,145-150	383.40	3.00	0.297	0.20	
47X-02,145-150	438.30	3.30	0.556	0.78	
53X-04,145-150	498.90	4.30	0.300	0.85	
59X-03,145-150	554.60	2.70	0.685	0.83	
71 X -04,145-150	671.50	4.60	0.008	0.04	
75X-04,145-150	710.10	5.70	0.010	0.10	
768 B- 1H-02,145-150	2.95	27.12	0.033	0.28	
2H-05,145-150	11.45	27.33	0.020	0.11	
3H-05,145-150	20.95	26.16	n.d.	0.09	
4H-05,145-150	30.45	24.90	0.020	0.14	
5H-04,145-150	38.45	22.79	0.083	0.10	
6H-05,145-150	49.45	23.88	0.127	0.19	
7H-05,145-150	58.95	22.72	n.d.	0.07	
8H-05,145-150	68.45	21.08	0.072	0.27	
9H-05,145-150	77.95	19.90	0.014	0.09	
10H-05,145-150	87.45	19.13	0.019	0.06	
13H-05,145-150	115.95	12.97	0.083	0.04	
16H-05,145-150	144.45	7.21	0.105	0.23	
19H-05,145-150	172.90	0.77	0.011	0.11	
26X-02,145-150	222.20	0.00	0.414	0.39	
34X-02,145-150	299.30	0.00	0.100	0.39	
768C-1R-03,145-150	357.60	6.46	0.392	0.47	
10R-03,145-150	444.30	1.20	0.059	0.45	
16R-01,145-150	499.30	5.30	0.131	0.25	
22R-02,145-150	558.70	4.12	0.235	0.49	
28R-04,145-150	619.80	0.32	0.129	0.34	
31R-03,145-150	647.20	6.39	0.151	0.37	
37R-04,145-150	706.70	1.76	0.245	0.28	
40R-03,145-150	734.30	6.88	0.059	0.20	
43R-02,145-150	761.40	5.23	0.033	0.15	

n.d.: Not determined

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Fig. 3. Depth distribution of pore-water sulfate, organic carbon, and total reduced sulfur in cores from Site 767 (Celebes Sea) and Site 768 (Sulu Sea). Pore-water sulfate data are from Rangin, Silver, von Breymann *et al.* (1990).

sulfide concentrations observed in cores at Site 767 and Site 768 represent a combined consequence of sulfate reduction with organic and detrital source input changes during sediment deposition in the basins.

3.2 Oxygen Isotopic Analysis

Results of oxygen isotope analyses of carbonates from Holes at Sites 767 and 768 are given in Table 2 and their depth distributions are displayed in Figure 4. The $\delta^{18}O$ varies between +0.11 and -6.40 o/oo in the Celebes Sea and ranges from +1.84 to -6.00 o/oo in Sulu Sea with a similar spread of approximately 7 o/oo. Furthermore, a close examination of $\delta^{18}O$ data at the top of cores reveals there is a 1 to 2 o/oo difference between the Celebes and Sulu basins.

As shown in Figure 4, $\delta^{18}O$ values in both basins become progressively lighter with increasing sediment depth. Such a tendency toward lighter $\delta^{18}O$ has been observed in many Deep Sea Drilling Project (DSDP) cores in the world ocean, and was attributed to diagenetic formation of carbonates in association with thermal alteration caused by the elevated burial temperature and/or volcanic activity (Coplen and Schlanger, 1973; Eade and Anderson, 1975). For instance, Lawrence et al. (1975) reported a systematic decrease of 1 to 3 o/oo of $\delta^{18}O$ with depth in the pore water of several DSDP cores as a consequence of the diagenetic alteration of basalt in Layer II of the ocean floor and/or of volcanic ash in the sediments. A similar depletion of pore water $\delta^{18}O$ with depth in the Hole 149 of DSDP was also observed by Perry et al. (1976) and was attributed to the submarine weathering of volcanics to form diagenetic smectites. Although these observed decreases in pore water $\delta^{18}O$ are primarily a result of increasing temperature, subsequent exchange of oxygen in the carbonates with the pore water $\delta^{18}O$ could result in a negative $\delta^{18}O$ of carbonates observed in the cores. In addition, study of the CO_2 derived from various organic degradation processes revealed a persistent decrease in $\delta^{18}O$ up to about 7 o/oo (Irwin et al., 1977).

3.3 Carbon Isotopic Analysis

The $\delta^{13}C$ distribution of carbonates from cores 767 and 768 also generally exhibits an overall decrease with depth and has a considerable range of variation (Table 2 and Figure 4). In the Celebes Sea (core 767) $\delta^{13}C$ varies between 0.43 and -29.33 o/oo, and in the Sulu Sea (core 768) ranges from 0.44 to -15.77 o/oo.

The highly negative $\delta^{13} C$ values measured in the cores are clearly not derived from a marine carbonate source, instead, the data indicate an organicallyderived CO_2 origin of carbon. The role of organic matter in contributing isotopically light carbon for the formation of diagenetic carbonates has long been recognized. Hodgson (1966) postulated the enzymic decarboxylation of organic compounds at low temperature as a mechanism for the source of light $\delta^{13}C$ of carbonates in marine sediments. Hein *et al.* (1979) reported the light $\delta^{13}C$

Table 2. Results of carbon and oxygen isotopic analysis of carbonate in sediments from Holes in Site 767 (Celebes Sea) and Site 768 (Sulu Sea).

Core-section	Depth	Carbonat	e (wt%)	δ ¹⁸ Ο	$\delta^{13} C$
(interval in cm)	(mbsf)	onshore	shipboard	(per	mil)
124-				- r	
767B-5H-06,124-127	46.24	28.13	24.2	0.11	0,14
17X-03,76-79	152.06	88.02	87.1	0.03	0.19
21X-01,115-118	184.95	59.53	68.0	-1.76	0.43
22X-05,60-61	200.10	90.59	88.5	-0.41	-0.74
27X-03,27-29	245.47	85.64	85.9	-2.02	-3.06
29 X- 02,8-9	262.98	80.70	81.5	-1.41	-7.76
30X-04,128-129	267.78	81.28	81.6	-2.83	0.21
35X-04,51-53	324.41	69.18	69.9	-2.77	-17.21
38X-01,73-75	349.23	67.94	76.9	-0.59	-18.39
45X-03,96-98	420.16	79.18	75.8	-4.08	-29.33
50X-05,100-102	471.10	61.78	60.1	-3.42	-4.50
59 X- cc	557.66	74.44	72.4	-6.40	-9.14
64X-04,58-60	603.58	75.66	73.5	-5.26	-11.94
768A-1H-01,66-68	0.66	45.48	40.0	-1.52	0.44
768B-3H-04,69-72	18.69	62.68	49.7	-0.88	-0.15
7H-02,45-47	53.45	75.56	61.5	-1.20	0.09
16H-05,55 - 58	143.55	40.47	38.4	0.69	-5.51
27 X- 02,137-139	231.77	36.35	56.9	-0.33	-11.46
32X-03,10-12	280.30	12.48	18.0	-1.02	-4.82
35 X-02 ,5-6	307.65	36.33	44.6	-0.99	-3.64
39X-02,15-17	346.45	71.50	55.6	0.75	-8.01
768C-2R-01,30-32	363.20	53.86	55.5	1.49	-11.14
3R-cc	373.35	45.05	48.6	1.84	-6.07
18R-01,6-8	517.26	35.90	35.7	-4.37	-6.22
22R-01,31 - 33	556.11	35.12	36.0	-5.22	-8.02
23R-01,5-7	565.55	65.18	68.6	-2.86	-4.48
40R-02,48-50	731.88	36.88	36.6	-6.00	-15.77

source for carbonates in sediment from the deep Bering Sea to have originated from oxidation of organic matter under anaerobic conditions during bacterial reduction of sulfate, or from CO_2 produced in concert with CH_4 during degradation of organic matter. Pisciotto and Mahoney (1981) attributed the low $\delta^{13}C$ values (up to -30%) at Site 468 and 471, DSDP Leg 63 to microbial



Fig. 4. Carbon and oxygen isotopic compositions of carbonate in Site 767 holes (Celebes Sea) and Site 768 holes (Sulu Sea).

reduction of sulfate at shallow depths. According to Irwin *et al.* (1977), CO_2 formed during bacterial sulfate reduction and thermally-induced decarboxylation can result in a -20 to -25 o/oo shift in $\delta^{13}C$. In all cases, CO_2 derived from these various processes can dissolve readily in pore water to form bicarbonate, and then precipitates out as carbonate, thus giving the observed light $\delta^{13}C$ of carbonate of the sediment cores studied.

As mentioned earlier, the onset of methane production was found to coincide with the base of the sulfate reduction zone in the cores. Although the processed (e.g., bacterial vs. thermogenic) for the formation of this methane have not been thoroughly determined, the occurrence is thought to be predominantly of biogenic origin (Rangin, Silver, von Breymann *et al.*, 1990). Furthermore, it has been shown that the fermentation processes (i.e., $2CH_4O_2 \rightarrow$ $CH_4 + CO_2$) are quantitatively more important in Holocene fresh sediments, whereas methane formed by CO_2 reduction is most common in older sediments upon deep burial (Balabane *et al.*, 1987; Kuivila *et al.*, 1990; Schoell, 1988). Regardless of the exact pathway for its formation, methane of biogenic origin is known to have a very negative $\delta^{13}C$ value. As a result, methane formed by various processes and subsequently oxidized to CO_2 can provide the necessary light carbon source for the carbonate (Hathaway and Degens, 1988; Irwin *et al.*, 1977). For this and other reasons, the observed low $\delta^{13}C$ values of carbonates of core 767 and 768 can be attributed to the biogenic carbon source input during carbonate formation. Nevertheless, in addition to the various sources of carbon possibly contributed from these different processes, the mixing of terrestrial with marine organic matter in the sediments may ultimately account for the observed large variation in $\delta^{13}C$ in the cores.

4. SUMMARY

Results of this study revealed that variation in organic carbon and sulfide distributions in the cores largely reflects changes in sediment source input. Sediment sections (e.g. Unit III of core 767 and Unit II of core 768; Rangin, Silver, von Breymann *et al.*, 1990) showing relatively higher organic carbon and sulfide concentrations generally correlate to high quartz content, high continentality index, and high rate of sedimentation, and thus are deposited under environments of strong terrigenous inputs. $\delta^{18}O$ of carbonates decreases progressively with increasing sediment depth in both cores, suggesting possible formation of carbonates at elevated temperature due to deep burial and/or volcanic activity. $\delta^{13}C$ of carbonates also shows a general decrease with depth, yet the profile is more variable than $\delta^{18}O$ and minimal values of $\delta^{13}C$ are found at various depths. The negative values of $\delta^{13}C$ are attributed to the biogenic carbon source input during carbonate formation.

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海洋鑽探計畫 124 航次 767 和 768 號 岩心之地球化學與穩定同位素研究

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摘要

本研究分析了兩根來自西里伯斯和蘇魯海盆岩心樣品(海洋鑽探計畫 124 航次;岩心編號 767 和 768)之碳酸鹽,有機碳和還原硫之含量,以及 碳酸鹽中碳氧同位素之組成,並據以探討其在岩心中垂直變化和已知海盆 演育過程之地化關係。結果顯示碳酸鹽,有機碳和還原硫在岩心中不同深 度之變化主要爲沉積物來源和沉積作用之不同所致。另外,碳氧同位素在 碳酸鹽中之值均隨深度之增加而遞減,且碳同位素值之變化較氧同位素值 為大並較不規則。氧同位素之遞減趨勢顯然和海盆演育過程中之劇烈火山 活動,盆地深化和沉積物堆積之結果有關;而碳同位素呈現較大之負值和 多變則表示其碳酸鹽來源之不同,且有部分碳酸鹽中之碳在高溫變質形成 過程中或多或少含括了有機來源之碳分子。