



Integrated Geochemical Investigations on Fe-Mn Nodules, Polymetallic Sulfides and Fe-Mn Oxides Recovered from Marine Sediments of Carlsberg Ridge, Northwest Indian Ocean

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Abstract

The research and exploration of submarine minerals have increased exponentially due to the requirement for rare and critical metals (cobalt, silver, platinum) as additional supplements to the gradually depleting terrestrial metal source. Manganese nodules, Fe-Mn oxides and polymetallic sulfides represent the most important source of these aforementioned critical metals. This paper highlight the results of the integrated studies on recovered minerals grains (e.g., Fe-Mn nodules, Fe-Mn oxide crusts and polymetallic sulfides) from marine sediments of the Carlsberg Ridge (CR), Northwest Indian Ocean. Geochemical and mineralogical techniques (e.g., X-Ray Diffraction studies (XRD); Standard Emission Microscope equipped with Energy Dispersive Spectrometer (SEM-EDS) and Electron Probe Micro-Analysis (EPMA) were used in conjunction with binocular and stereographic microscopes on the separated mineral grains. The representative marine nodules from the sediment core collected at ~300 km to the ridge segment are dark and botryoidal to spherical with average Cu, Co, Ni and Zn concentrations of 0.17 wt%, 0.19 wt%, 0.22 wt%, and 0.15 wt%. The sulfides of the near-vent sediments of CR showed euhedral texture, with average Cu and Co concentrations of up to 0.08 wt% and 0.05 wt%. The Fe-Mn oxide crust was yellowish to reddish-brown, poorly crystallized, with goethite minerals. The average Ni and Cu concentrations are 0.04 wt% and 0.60 wt%. These studies further highlight the need to integrate all specializations and expertise for a thorough understanding of the growth, trophic interactions and resilience of the marine ecosystems in the Carlsberg Ridge and other vent fields in the Indian Ocean.

Keywords

Submarine minerals, Near-vent sediments, Carlsberg Ridge, Indian Ocean, Marine ecosystems

Introduction

The oceans cover more than 70% of the planet and represent a potentially promising new frontier for the research and exploration of minerals. The research and exploration of submarine minerals have greatly increased due to the requirement for critical metals and the recent drive for a green economy. Fe-Mn nodules, polymetallic sulfide minerals, and Fe-Mn crusts have been identified as the source of these critical metals such as Rare Earth Elements (REEs), Silver (Ag), Gold (Au), Zinc (Zn), antimony (Sb), Selenium (Se), Cobalt (Co), Tellurium (Te), Neodymium (Nd), copper (Cu), Manganese (Mn), platinum (Pt) and others [1].

Two typical micronodules have been identified in mid Ocean ridges and environments including the diagenetic micronodules and the micronodules that are precipitated from

plume fallout of oxyhydroxide matter. Whereas, with distance away from the Mid Ocean ridge segments, hydrogenous and diagenetic processes dominate due to masked hydrothermal

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signatures [2]. It is noteworthy that these micronodules are not easily observed as pure end members [2-5].

Polymetallic sulfides are one of the manifestations of hydrothermal activities with other diverse mineral assemblages such as pyrite/marcasite, chalcopyrite, bornite, covellite, digenite, isocubanite, pyrrhotite, barite, anhydrite, amorphous silica and Fe-oxide and hydroxides [6]. Ferromanganese oxides and crusts are widespread on the ocean floor and are formed through the precipitation of suspended iron and manganese oxyhydroxides owing to the hydrothermal-hydrogenetic supply. However, the hydrogenetic supply tends to affect the proportions of goethite (FeOOH), vernadite, the Mn/Fe ratio, cerium anomaly (Ce*), and the REEs, Cu and Co concentrations therein. These diverse marine mineral deposits (Fe-Mn nodules, polymetallic sulfides and Fe-Mn crust) are supplied to the marine sediments by slumping or fall-out of nearby buoyant plume [2,7,8], therefore sediments can act as a sink to the three aforementioned mineral deposits.

Oceanographic factors such as global contouritic bottom-currents or upwelling undercurrents and plate tectonics have a great influence on the formation of these mineral resources in submarine environments like abyssal plains, seamounts and continental margins [1]. The essential techniques on the aforementioned oceanographic factors include a wide range of methodologies, such as multi beam bathymetry, remote-operated vehicles (ROVs), autonomous underwater vehicles (AUVs), magnetometers [1], and detailed micro and nano-scale mineralogical and geochemical *in-situ* investigations of marine mineral grains recovered from rocks and sediments.

This paper aims to integrate the results of micro-scale mineralogical and geochemical *in-situ* investigations of min-

eral grains (e.g., Fe-Mn oxides, polymetallic sulphides) with the Fe-Mn nodules recovered from sediment core approximately 500 km East to Tianxu Hydrothermal field (THF) and 1100 km North-East to Wocan Hydrothermal field (WHF), Carlsberg Ridge (CR), Northwest Indian Ocean (Figure 1).

Geological settings of Carlsberg Ridge (Northwest Indian Ocean)

The Carlsberg Ridge (C.R, Figure 2) is part of the Indian Ocean Ridge Systems (IORS) which is a long chain of active submarine volcanoes [9-13]. It defines the divergent plate boundary between the Indian and Somalian plates, which opened during the late Paleocene (~55 Ma) when the Seychelles bank separated from the Indian plate [12,13-18]. The spreading rate along the Carlsberg Ridge gradually increases from North to South: with a half-spreading rate of ~11-16 mm/yr [11,18-20].

Previous research had been conducted on the geophysical investigations involving, the multi-beam swath bathymetry maps along with a few segments of the Carlsberg Ridge. Few of the previous reports on the morphological characteristics of the Carlsberg Ridge showed rugged topography and steep inner valley walls, similar to the ridge morphology of northern Mid Atlantic Ridge, and other slow-spreading ridges [11,12,21,22]. Three distinctive segment morphotypes have been identified in the C.R: 1) Symmetric segments with a shallow axial valley and smooth axial plateau; 2) symmetric segments with steep axial valley walls and a hummocky axial volcanic ridge (AVR), and 3) segments with deep axial valley walls, often with an asymmetric flank massif [23]. Subsequent exploration and scientific studies have led to the discovery of three active hydrothermal fields (i.e. mafic hosted Wocan-1

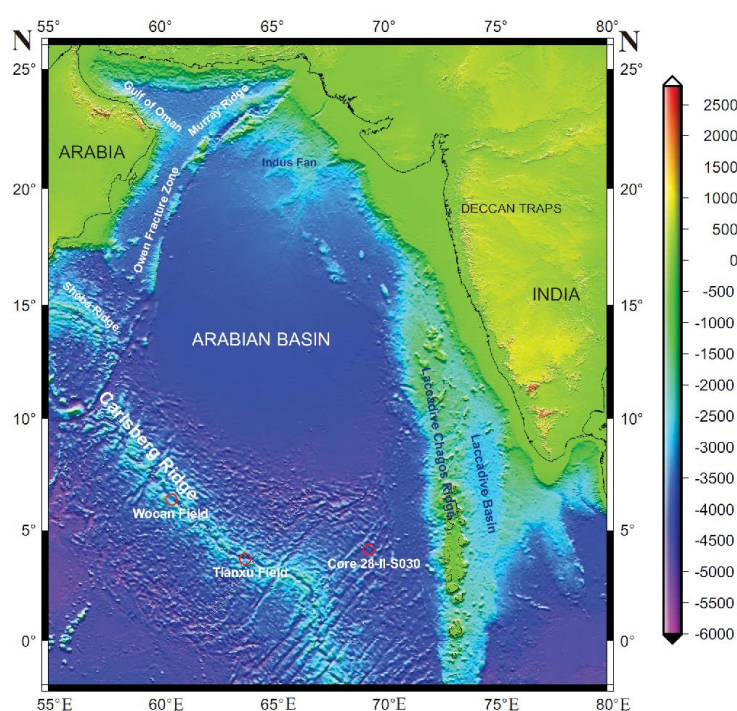


Figure 1: Bathymetric map of subsurface core samples where Fe-Mn nodules were isolated off Tianxiu and Wocan field.

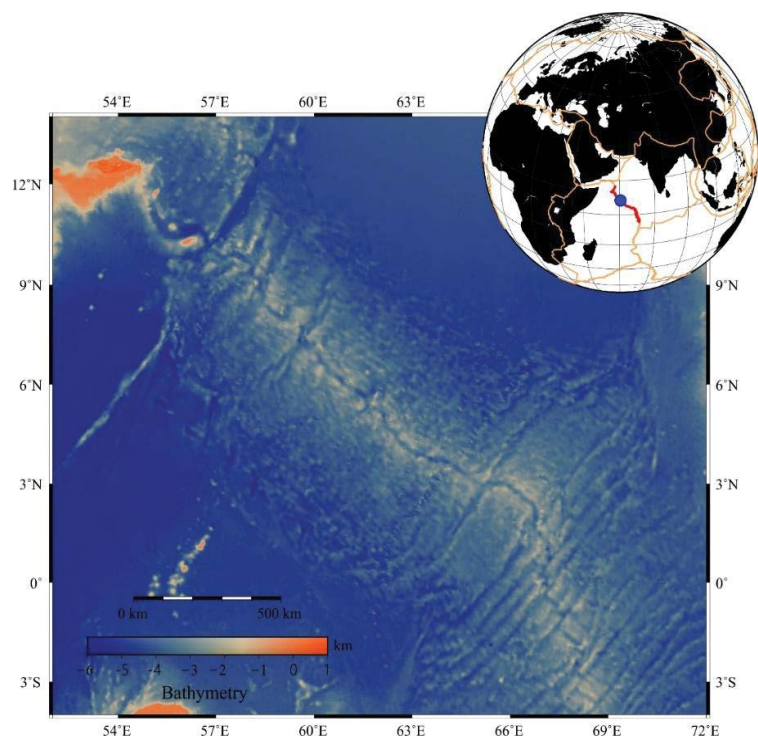


Figure 2: Bathymetric map of Carlsberg Ridge.

Table 1: Coordinates of sampling stations and type of sediment samples collected from the Northwest Indian Ocean.

Site	Station	Longitude (E)	Latitude (N)	Depth (m)	Type of samples
Wocan-1	28I-TVGO7	60° 31.534'	6° 21.796'	2989	Metalliferous sediment
	28I-TVGO8	60° 31.635'	6° 21.756'	2973	Metalliferous sediment
Wocan-2	28I-TVG10	60° 30.372'	6° 21.866'	3104	Metalliferous sediment
	28II-TVGO5	60° 30.226'	6° 22.534'	3105	Metalliferous sediments
Ridge flank	28I-TVG13	60° 13.190'	6° 35.675'	3254	Pelagic sediment
	28I-TVG12	60° 13.550'	6° 30.462'	2009	Pelagic sediment
Core sediments	28II-TVMC01-S030	69° 20.201'	4° 07.52'	4228	Pelagic sediment

(Figure 1) and ultramafic hosted Tianxiu (Figure 1), and Daxi hydrothermal field on the Non-transform offset along the Carlsberg Ridge [5,6,24-31].

Methodology and Approach

Polymetallic sulfides and Fe-Mn oxides

Bulk representative samples of marine sediments from 6 stations in Carlsberg Ridge (Table 1) were washed with ultra-pure water to remove interstitial salts and dried at 60 °C. Approximately 60 g of the sub-samples were wet-sieved to separate into > 63 µm (sand size fraction) and < 63 µm (silt and clay size). Polymetallic sulfide mineral grains and Fe-Mn oxy-hydroxides and crust in the > 63 µm fractions were examined, and hand-picked under the stereographic microscope, binocular microscope and standard Electron Microscope (SEM) equipped with an EDX analytical X-Ray system operated at an accelerating voltage of 20-30 kV. The isolated mineral grains were ultrasonically cleaned in ultra-pure alcohol to remove any seawater influence, then impregnated with epoxy resin and hardener (ratio 2:1), [5,6,26]. Representative grains from

the minerals were analyzed using an Electron probe micro-analyses (EPMA, JEOL JXA-8100) with an acceleration voltage of 24 kV, beam current of 1 nA and a beam diameter of 5 µm at the State Key Laboratory of Submarine Geosciences (KLSG), Hangzhou China. The analytical precision and minimum detection limits (MDL) are > 0.1 wt. % and better than ± 10%.

Fe-Mn nodules

The selected working half of the subsurface sediment core collected at about 300 km from the Ridge Segment (Figure 3) was sliced into 4 cm interval. Fe and Mn concentrations were analysed from one-half of the core sediment samples by inductively coupled Plasma Mass Spectrometer (ICPMS) using Agilent 7700 ICPMS, and method ME-MS61 at the Australian Laboratory Services (ALS) Chemex Guangzhou Limited Company, China.

About 5 g of representative samples at 4 cm interval (e.g., 2 cm, 6 cm, 10 cm.....38 cm, 42 cm) were soaked in de-ionized water for 24 hours, and wet-sieved to separate > 63 µm (sand size) fractions. Micro nodules were observed, and further

Photo	Litho-Log	Description	XRD investigation					
			Qz	Cln	M	V	P	C
		Biogenic carbonates with traces of altered Mg-aluminosilicates and basaltic fragments	XXX	X	XXX	X	-	-
			XXX	X	XX	-	XX	XXX
		Biogenic carbonates with traces of Mn-oxide minerals	-	-	-	-	-	XXX
		Biogenic carbonates with minor Cu-rich Fe-Mn-oxyhydroxides and Ca-Mg aluminosilicates minerals	X	XXX	XX	-	-	XX
			X	XXX	XXX	-	-	XX

Figure 3: Photograph and mineralogical description of sediment core retrieved off Tianxiu and Wocan field XXX: Abundant, XX: Minor, X: Trace.
Abbreviations: Qz: Quartz, Cln: Clinocllore, M: Muscovite, V: Vermiculite, P: Phlogopite; C: Calcite.

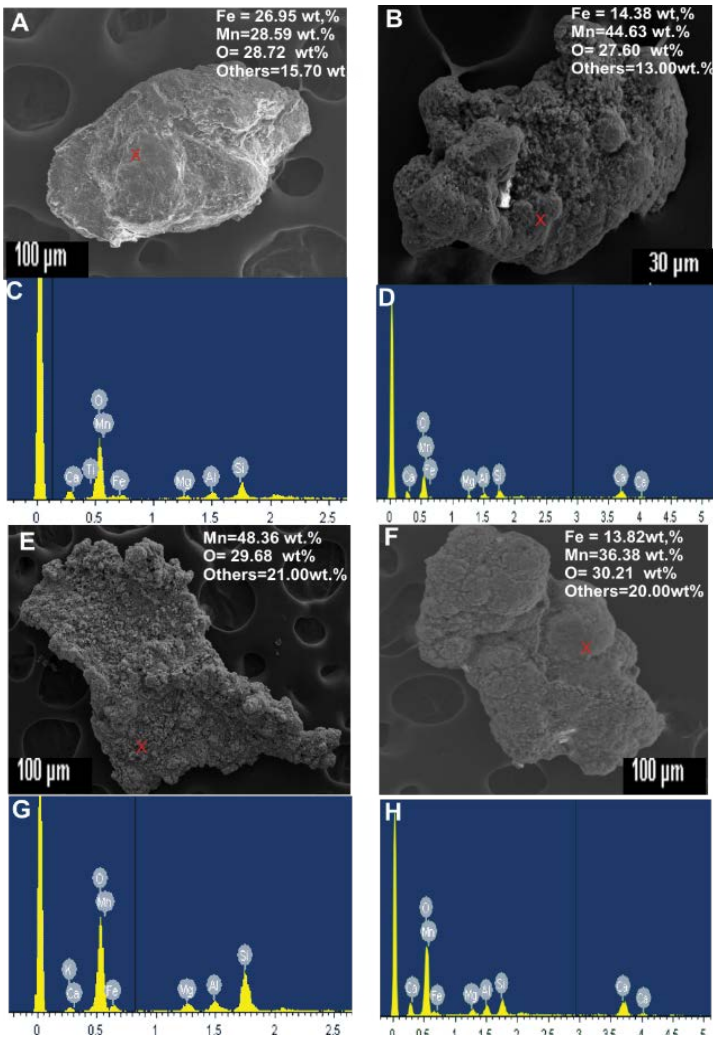


Figure 4: SEM morphology and EDS spectra of representative Fe-Mn nodules from the Carlsberg Ridge, NW Indian Ocean. The X indicate the analyse spot.

hand-picked, temporarily at the third layer (unit III, 30-42.5 cm). The grains were mounted on stud attached with carbon tapes from the sand-size sediment fractions using a binocular and stereographic microscope. X-ray diffraction studies (XRD) was conducted on the < 63 μm (silt + clay size) fractions. The SEM-EDX, EPMA and Xrd investigations were operated at an accelerating voltage similar to the approach adopted on the sulfides and Fe-Mn crust at the State Key Laboratory of Submarine Geosciences (KLSG), Hangzhou China.

Results

Geochemical and mineralogical investigations of isolated Fe-Mn micro nodules

Based on the microscopic studies, the sediment core was divided into three units. The combinations of optical and SEM investigations on the isolated mineral grains revealed that unit I (0-19 cm) are majorly biogenic with minimal siliceous carbonates, traces of altered magnesium alumina silicates minerals, and altered basaltic fragments. Unit II (20-29 cm) are majorly biogenic with minimal siliceous carbonates and traces of Mn oxide minerals. Unit III (30-42.5 cm) majorly contains a mixture of biogenic carbonates, minor Cu-rich-Fe-Mn oxides, Fe-oxyhydroxides and calcium magnesium alumina silicates minerals (Figure 3).

The XRD investigations of the core sediments reveal quartz, clinocllore, muscovite, vermiculite, phlogopite and calcite (Figure 3). The micronodules were abundant in Unit III. The microscopic observation of representative marine nodules from the sediment core revealed dark brown and black coloured nodule. The micro-nodules ranges from botryoidal to spherical in shape and the SEM-EDS investigation showed Mn concentration of up to 44.63 wt%, with Fe values of up to 26.95 wt% (Figure 4). The (EPMA) further revealed average Cu, Co, Ni, Zn concentrations of 0.17 wt%, 0.19 wt%, 0.23 wt%, and 0.15 wt% respectively (Table 2).

The enriched major elements Fe_2O_3 and MnO at unit III (Figure 5) further supports the abundant micronodules and Fe-oxyhydroxides from the third layer.

Geochemical and mineralogical investigations of isolated polymetallic sulfide grains from CR sediment

The stereographic microscopic observation, SEM, and EDS investigations of Wocan-1 and Wocan-2 stations (Table 3) revealed that the hydrothermal sulfide components separated from the metalliferous sediments of Carlsberg Ridge were mainly composed of pyrite, chalcopyrite, sphalerite and secondary Cu-sulfide minerals (Figure 6).

Table 2: Average values of Micro-chemical (EPMA) composition of micro modules separated from Carlsberg Ridge sediments.

	Fe	Mn	Co	Ni	Cu	Zn
1	30.32	19.46	0.24	0.33	0.19	0.11
2	18.47	23.86	0.12	0.19	0.15	0.14
3	16.86	24.64	0.21	0.22	0.16	0.04
4	22.84	22.33	0.21	0.17	0.18	0.18
Average	19.39	22.14	0.19	0.22	0.17	0.15

All values are in wt%. Source

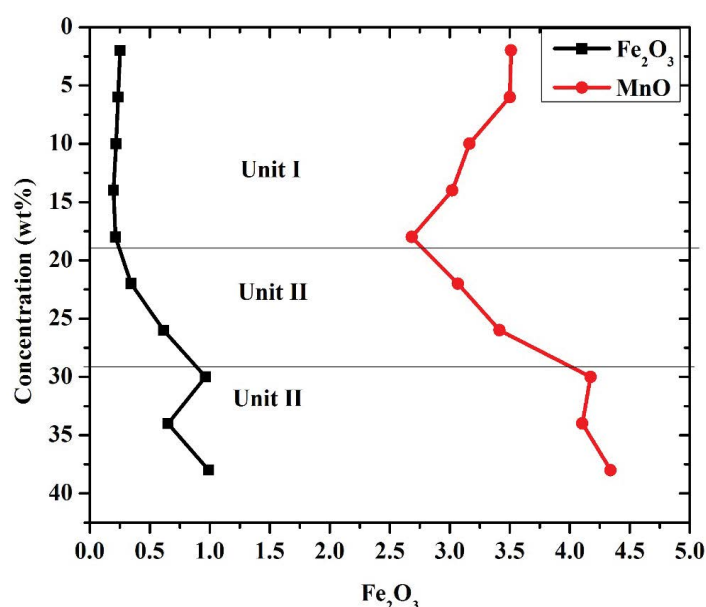


Figure 5: The Fe-Mn concentration of the sediment core samples recovered off Wocan and Tianxiu vent field.

Table 3: Average values of Micro-chemical (EPMA) compositions of Chalcopyrite (Cpy), pyrite (py) and sphalerite (sph) recovered from CR sediment.

	S	Fe	As	Se	Cu	Co	Zn
Cpy	35.43	30.33	Bld	0.02	34.46	0.03	Bld
Py	53.15	46.36	0.11	0.03	0.08	0.05	0.01
Sph	33.685	2.26	Bld	Bld	0.065	Bld	63.18
	Sb	Ag	Pb	Au	Cd	Mo	Total
Cpy	0.02	0.01	0.08	Bld	Bld	Bld	100.38
Py	0.01	0.01	0.15	0.01	Bld	Bld	99.97
Sph	Bld	Bld	0.04	Bld	0.375	0.025	99.63

All values are in wt%

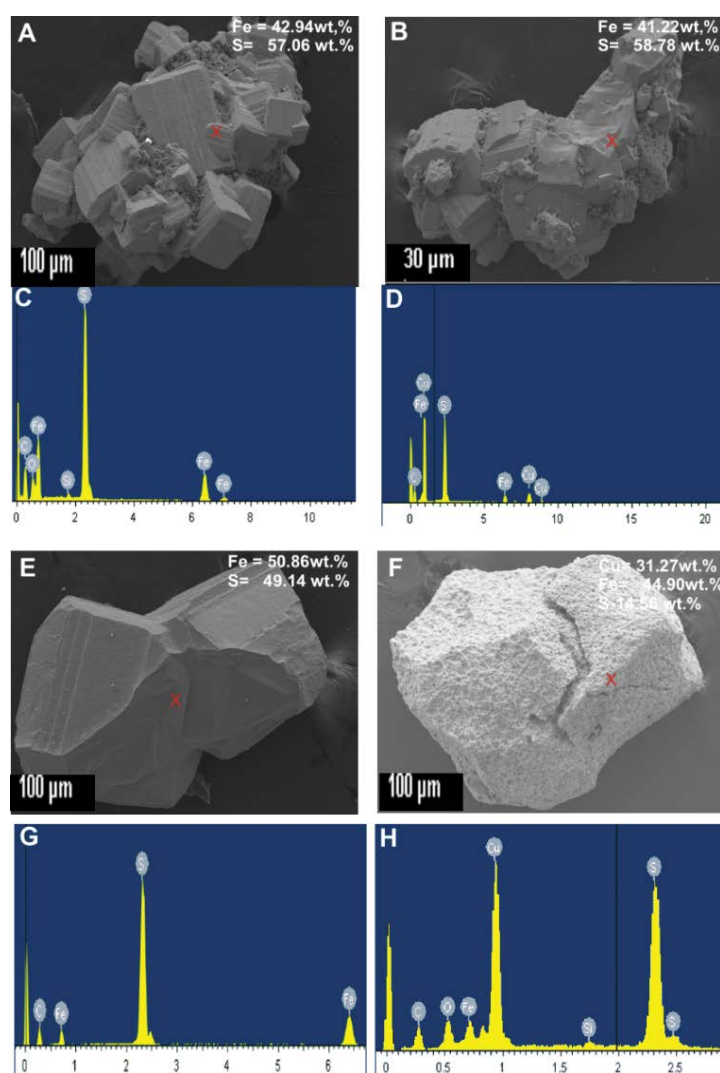


Figure 6: SEM morphology and EDS spectrum and values (wt. %) of representative sulfide mineral grains from the Carlsberg Ridge, NW Indian Ocean. The X spot indicates the analysed spot. A-B are representative pyrite grains, C-D is the respective spectrum. E-F are representative pyrite and chalcopyrite grains G-H are the respective spectrum.

The sulfides from the active (Wocan-1) stations showed euhedral texture. The mineral chemistry further revealed Cu and Co enrichment of up to 0.08 and 0.05 wt% (Table 3).

Geochemical and mineralogical investigations of isolated Fe-Mn oxide grains from C.R sediment

XRD investigations revealed goethite in association with

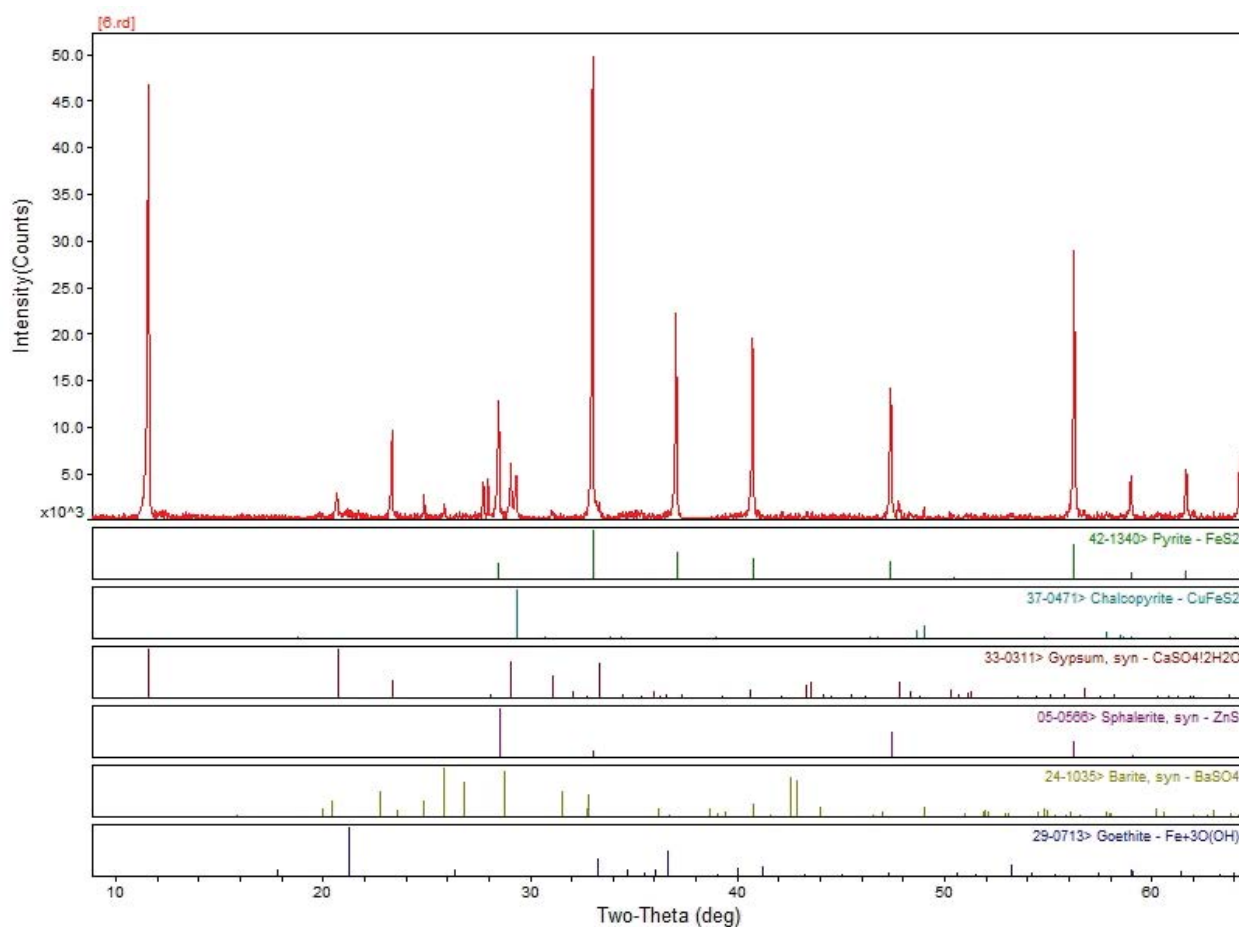


Figure 7: XRD patterns of the bulk sediment from Carlsberg Ridge.

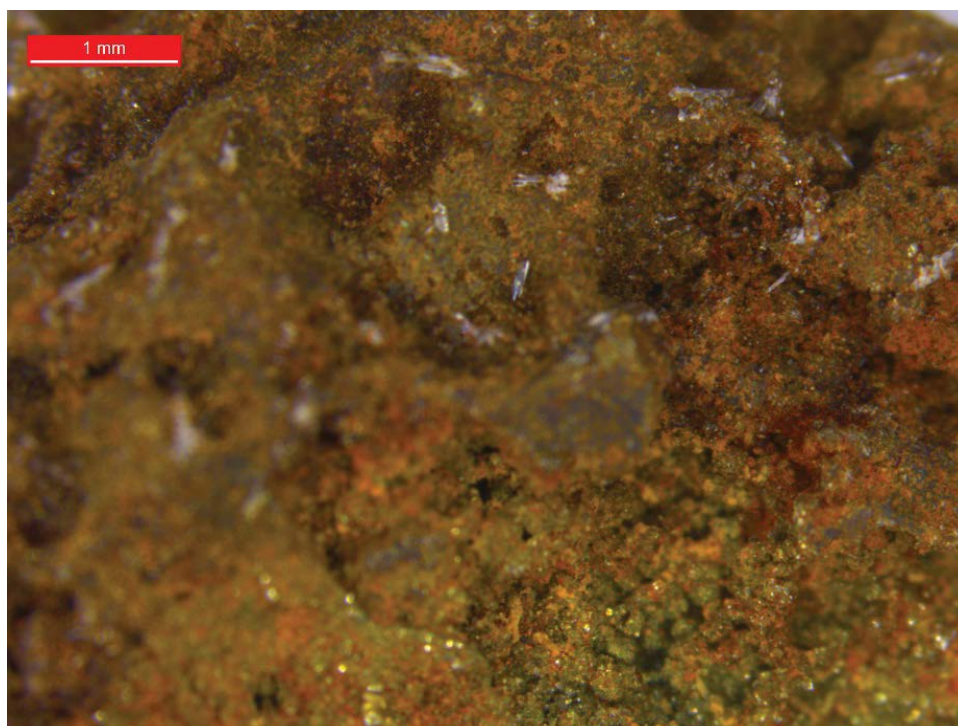


Figure 8: Photomicrographs of separated grains of Fe-Mn Oxide grains separated from the sediments of Carlsberg Ridge.

sulphide and sulphate minerals (Figure 7 and Figure 8). The mineral chemistry (EPMA) of the isolated Fe-Mn oxides further reveal Ni, Cu, as and Zn of up to 0.04 wt%, 0.60 wt%, 0.06 wt% and 0.51 wt% respectively (Table 4).

Discussions

The research gap

Recent studies on CR were concentrated on the mineralogy and geochemistry of hydrothermal precipitates and metalliferous sediments [5,6,25]. The studies confirmed the occurrence of seafloor weathering and mixing of hydrothermal fluid with seawater (during the waning stages of hydrothermal process) as the controlling factor in sulphur isotopic concentrations, minor and trace element re-distributions. Ref [29] suggested Cu-Zn-Fe sulphides as the dominant minerals in the proximal sediments of the Tianxiu hydrothermal field (0-0.22 km). While fine-grained Fe-oxides and hydroxides and calcite minerals dominated the distal sediments (1.84-6.05 km). This is an indication of particle size characteristics as a factor of a hydrothermal indicator. Ref [31] worked on 3 sediment push cores collected via submersible *jiaolong* to determine the degree of evolution of hydrothermal activity in the WHF. They further conducted Pb-Sr isotopes on the push core samples. They confirmed that the Wocan-1 site exhibit a pervasive hydrothermal activity that spans 1069 years, with Fe fluxes of 9483-10834 mg cm⁻² kyr⁻¹, while the Wocan-2 site had been inactive since 4864 ± 114-years before present (yr BP). Ref [32] worked on the Fe and S isotope fractionation and suggest a $\delta^{56}\text{Fe}$ ranges of -2.05‰-0.10‰. The $\delta^{34}\text{S}$ concentrations of the Wocan-1 and Wocan-2 sediments fall within the range reported by [6]. Ref [30] worked on the Daxi vent field (an active mafic hosted hydrothermal systems at a non-transform offset) in the CR and suggested the occurrence of a silica-rich chimney, and sulfide chimney dominated by sphalerite, pyrrhotite (with enriched Sn, Co and Ag).

The right direction in the CR's studies is the identification of seven mega fauna species (including allvelliid worms) in the Daxi vent field. However, there is a dearth of information on the lifestyles, adaptation and trophic requirements of endemic/chemosynthetic vent fauna in Wocan vent field; Tianxiu vent field; Daxi vent field and other recently discovered hydrothermal fields in the Indian Ocean. This call for thorough research into the endemic/chemosynthetic vent fauna of the recently discovered vent field in the Central Indian Ridge (MESO, [33]; Kairei, [34]; Edmond, [35]; Dodo and Solitaire [36]; Yokoniwa [37]. A similar approach is required in Longqi-1, Longqi-2 (49° 39'E; [38,39]; Yuhuang 49° 16'E, [40]; Xilongjing 49° 40'E, and Zhanqiao 50° 59'E [41]; Duanqiao 50°

24'E, [42] and Mount Jourdanne [43] along the South West Indian Ridge (SWIR).

At present, the public and private institutions have rediscovered the zeal in deep-sea mining exploration and exploitation. However, further research on the relationships between Nodules, Sulfides and Fe-Mn crusts with marine sediments and marine ecosystems requires an interdisciplinary approach and collaboration among marine geologists, marine geophysicists, marine geochemists, scientists, macro/microbiologists, marine biogeochemists, environmental management, social scientists, engineers, contractors/investors, legal scholars, and economists.

Conclusions

The geochemical and mineralogical investigation of proximal and distal sediments from Wocan hydrothermal sites, CR, Indian Ocean revealed diverse marine mineral deposits (Fe-Mn nodules, polymetallic sulfides and Fe-Mn crust). The recovered micro-nodules at ~300 km to the CR ridge segment ranges from botryoidal to spherical, with Mn concentration of up to 44.63 wt%, and Fe vales of up to 26.95 wt%. The EPMA investigations revealed average Cu, Co, Ni, Zn concentrations of 0.17 wt%, 0.19 wt%, 0.23 wt%, and 0.15 wt%. The hydrothermal sulfide components separated from the metalliferous sediments of Carlsberg Ridge were mainly composed of pyrite, chalcopyrite, sphalerite and secondary Cu-sulfide minerals. The sulfides of the Wocan-1 stations showed more euhedral texture, with Cu and Co enrichment of up to 0.08 and 0.05 wt%. The recovered Fe-Mn oxides are angular to sub-angular, yellowish to reddish-brown with goethite minerals, and Ni, Cu, As and Zn concentrations of up to 0.04 wt%, 0.60 wt%, 0.06 wt% and 0.51 wt%.

This study suggests a combination of science and policy (e.g., micro/macro biologists, ecologists, marine geoscientists, contractors, environmental managers, civil society, ecotoxicologists) on the marine mineral deposits of Carlsberg Ridge for a thorough understanding of the species, dynamics, structure, distributions, lifestyles and adaptation, trophic requirements and reproductive characteristics of the associated sediment faunas from the effect of anthropogenic disturbance arising from deep-sea mining of these mineral deposits from CR and other vent fields along the Indian Ocean.

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Table 4: Electron probe microanalysis results (wt%) for Fe-Mn oxides from Carlsberg Ridge.

Element	SiO ₂	SO ₃	FeO	MnO	TiO ₂	Al ₂ O ₃	Al/Al + Fe + Mn	Fe/Mn			
Av	11.82	0.10	67.5	0.06	0.06	0.31	0.003	2,486			
	Cu	Co	Ni	Ba	Sr	As	Zn	Pb	Total	H ₂ O ⁺ (calc)	Cu + Co + Zn + Ni
Av	0.66	Bdl	0.04	0.04	0.02	0.06	0.51	0.10	81.7	18.28	1.19

Av: Average, n = 11.

sampling of the Wocan Hydrothermal Field, Carlsberg Ridge, Northwest Indian Ocean, and the four anonymous reviewers for the helpful comments that have greatly improved the manuscript.

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