Diagenetic Screening in Porites Fossil Corals from South Pagai, Kendari, and Banten Bay, Indonesia

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Abstract. Fossil corals are commonly used in paleoclimate studies to get records of climate parameters throughout the Holocene and beyond. Diagenesis is known as an important error source in paleoclimate reconstruction. The aim of this research was to provide a comprehensive diagenetic investigation involving 2D-XRD, petrographic analysis, and scanning electron microscopy (SEM) of *Porites spp* fossil samples from South Pagai, Kendari and Banten Bay, Indonesia as a starting point for further climate studies using coral proxies. This research focused on samples with around 1% calcite content, a level that can create misinterpretation of geochemical proxies. The results indicate that the samples from Banten Bay and South Pagai are well preserved and reliable for paleoclimate study. Only Sample BG1 is not recommended for further use in geochemical proxy analysis due to intensive diagenesis. 2D-XRD allows calcite screening without destroying the coral sample and assists in defining alternative sampling transects. Secondary aragonite and dissolution cannot be identified with 2D-XRD, therefore diagenetic screening should be combined with petrographic and SEM analysis in any areas presumed to have diagenetic textures.

Keywords: *2D XRD; aragonite; calcite; coral; diagenesis; SEM; thin section.*

1 Introduction

Scleractinian corals incorporate a range of isotopic (e.g. $\delta^{18}O$) and elemental (e.g. Sr/Ca, U/Ca, Mg/Ca) tracers that can provide seasonally resolved reconstructions of sea surface temperature and sea surface salinity to increase understanding of paleo-ocean thermohaline and climate variability [1-3]. However, dealing with fossil corals also means dealing with possible diagenetic effects on the samples [4,5].

Before any detailed climatic information can be derived from the geochemical content of a fossil coral, it is necessary to study the preservation state (i.e. diagenesis) of the coral. The presence of secondary aragonite, secondary calcite, and dissolution is well known as an error source in paleoclimate reconstruction [6,7]. The presence of ≤ 1% secondary calcite can lead to warm SST artifacts

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[8-10]. XRD is one of the most convenient and widespread methods for mineralogy characteristics determination [11] and is used routinely in combination with other techniques to detect diagenesis in corals [8,10,12]. Nevertheless, conventional XRD preparation is known to have the disadvantage of destroying the coral samples, as it requires a small proportion of the sample in powder form. To avoid this problem, 2D-XRD can be used directly on coral slabs to detect calcite rapidly and nondestructively [13].

Primary coral aragonite cannot be distinguished from secondary aragonite using XRD, therefore it is still problematic to detect occurrences of secondary aragonite [6,14]. Dissolution, another essential diagenetic texture that may cause cool SST artifacts [14-16], also cannot be detected by XRD. In contrast, petrography and SEM analysis allow to screen for secondary aragonite and dissolution better. The purpose of this research was to provide a comprehensive diagenetic investigation, involving 2D-XRD, petrographic and SEM analysis, focusing on samples with around 1% calcite content, as a starting point for further climate studies using coral geochemical proxies.

2 Material and Method

Porites spp fossil corals from three different sites, i.e. Kendari, Banten Bay and South Pagai, were used in this study (Figure 1). Five fossil cores (approximately 50-120 cm in length) were drilled from Quaternary reef terraces around Banten Bay, West Java (Panjang Island). Five fossil corals (approximately 10-30 cm in length) were collected from South East Sulawesi (Kendari). Fossil coral samples from Kendari were used in [17] to screen for diagenetic features using conventional XRD and petrographic analysis. One fossil coral (approximately 50 cm in length) was collected from a micro atoll in South Pagai, Mentawai. The core samples were collected using a hand pneumatic drill. Each core location and length information can be seen in Table 1. The cores were cut into 0.5 cm thick slabs and cleaned in an ultrasonic bath filled with distilled water for 15 minutes and afterward dried in a drying cabinet at 70 °C. Samples were then prepared for 2D XRD analysis, petrographic sectioning and scanning electron microscopy (SEM).

The calcite content in all coral samples was quantified along the growth axis using 2D-XRD, following the methods discussed in [13]. The coral growth axis was recognized from an X-radiograph (Figure 2). A Bruker AXS D8 Discover with GADDS (General Area Detector Diffraction Solution) 2D-XRD system in $\theta - \theta$ configuration equipped with area detector and Goebel mirrors was used to identify the calcite directly on the coral slabs. The 2D-XRD instrument was first calibrated with gravimetric standard powders of high-magnesium calcite and low-magnesium calcite. The diffractometer settings were adjusted to achieve the best diffractogram separation between calcite (HMC/LMC) and aragonite. HMC and LMC therefore could be identified with a diffractometer setup based on the calcite reflection peak area [13].

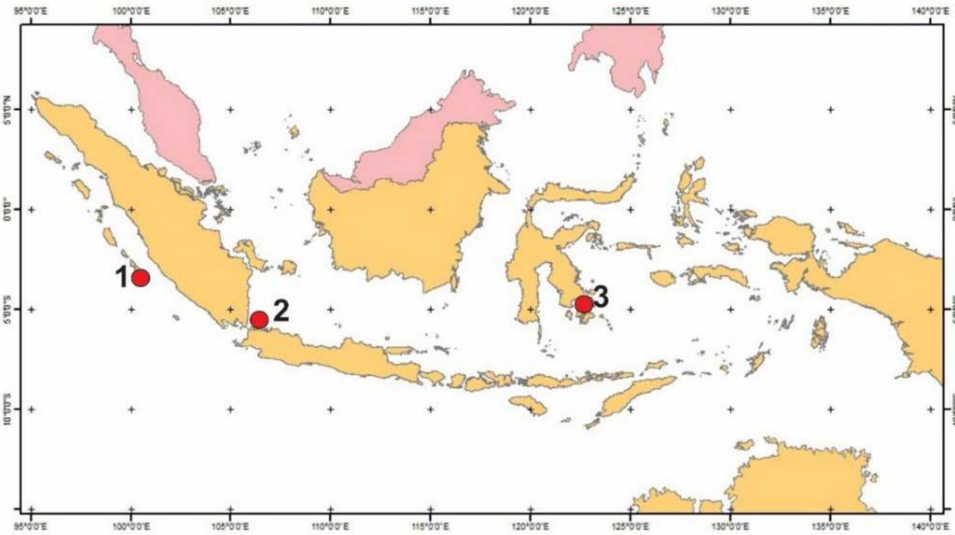

Figure 1 Map showing *Porites spp* fossil coral sampling locations. (1) South Pagai, Mentawai Island, (2) Panjang Island, Banten Bay, and (3) Kendari, South East Sulawesi.

The samples for which the 2D-XRD point test detected a calcite content of around 1% were selected for petrographic thin section and SEM. Thin-section preparation was done on 6 cores samples, i.e. 1/1 (core 1 slab 1), 2-2/4 (core 2 slab 2 of 4), PP 5.1 (core 3 slab 1), PP 5-6 (core 3 slab 6), 5-4 (core 5 slab 4), BG3B (core BG3B). The thin sections were used to evaluate the presence of secondary aragonite, calcite, and dissolution for areas equivalent to the X-ray beam spot of the 2D XRD system. Thin-section blocks were set in epoxy resin and then standard, 0.03 mm thick, 60 x 20 mm petrographic thin sections were prepared of each sample. Pictures of the coral thin sections were taken using an optical microscope in plane polarized light.

The mineralogy of the cement seen on each thin section was determined using SEM in backscattered electron (BSE) mode combined with energy dispersive X-ray (EDX) analysis performed on a Zeiss SEM (FESEM SUPRA^{TM} 55; Apollo 10 SDD, EDAX) as well as transmitted light microscopy. In areas where the 2D-XRD point test detected around 1% calcite content, 13 chipped coral samples of ~3-6 mm length were carbon-coated and prepared for SEM following standard procedures, i.e. 1-1/5 (core 1 slab 1 of 5), 1-4/5 (core 1 slab 4 of 5), 2-2/4 (core 2 slab 2 of 4), PP 5-1 (core 3 slab 1), PP 5-2 (core 3 slab 2), PP 5-6 (core 3 slab 6), 4-1/7 (core 4 slab 1 of 7), 5-2 (core 5 slab 2), 5-6 (core 5 slab 6), BG3B-1, BG3D, BLSa, and BLSb.

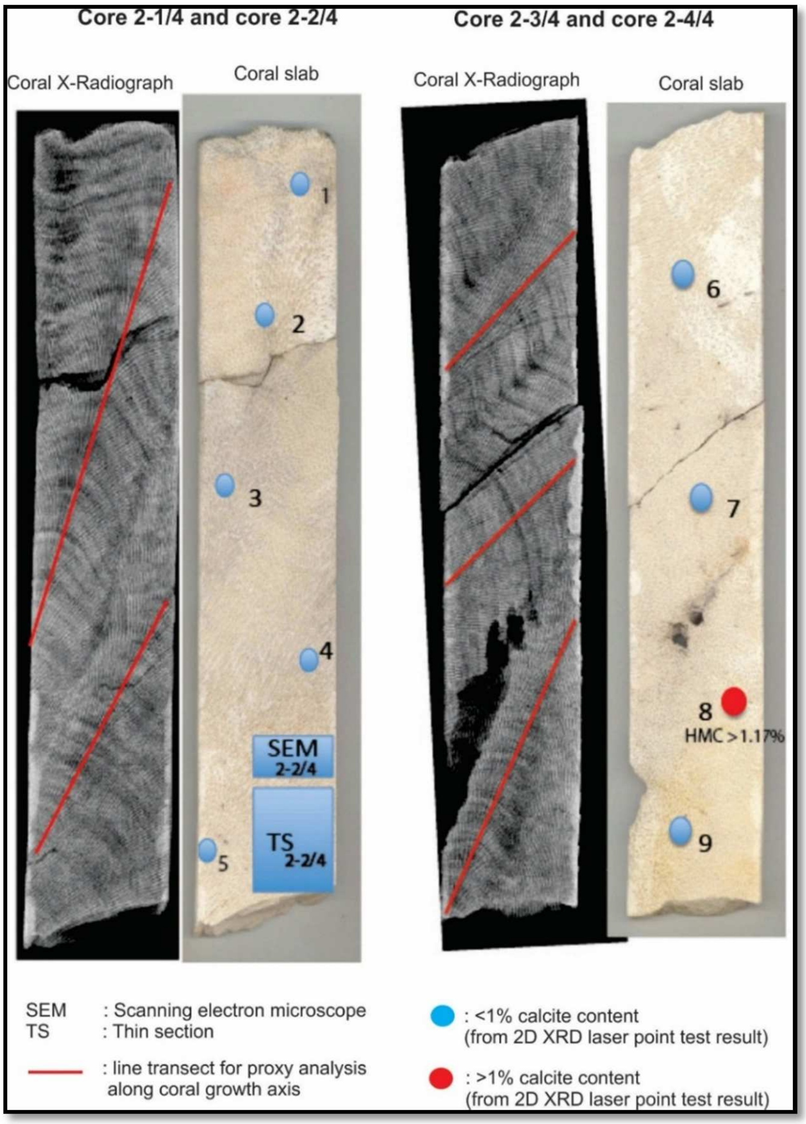

Figure 2 Sample core 2-1/4, core 2-2/4, core 2-3/4 and core 2-4/4 from Panjang Island, Banten Bay. 2D-XRD point test, SEM and petrographic analysis were applied along the coral growth axis, which was identified from an X-radiograph. It can be inferred from the 2D-XRD laser-point test that calcite usually occurs locally or as patches (for example point 8). Due to its higher spatial resolution compared to conventional XRD, 2D-XRD allows to define alternative sampling transects to avoid calcite patches.

Table 1 *Porites* core locations and lengths.

Sample code	Location	Length
1-1/5 to 1-5/5 (5 slabs)	Panjang Island	~136 cm
2-1/4 to 2-4/4 (4 slabs)		~65 cm
PP 5-1 to PP 5-6 (6 slabs)		~160 cm
4-1/7 to 4-7/7 (7 slabs)		~159 cm
5-1 to 5-6 (6 slabs)		~121 cm
BG3B_1 and BGB_2 (B1 & B2 is different colony)	Kendari	~20 cm
BG3C		~30 cm
BG3D		~20 cm
BG1		~10 cm
BLSa and BLSb (a & b samee colony)	South Pagai	~10cm, ~35 cm

3 Result and Discussion

3.1 2D-XRD Analysis

Based on the 2D-XRD result, most samples were well-preserved, except for core sample BG1, of which the calcite content was > 46% (Table 2). XRD is the most convenient and widespread method for mineralogy assessment [11,13].

Table 2 Calcite cement content in *porites spp* fossil coral from Kendari quaternary carbonate*.

2D XRD point test ID	% Calcite cement HMC	% Calcite cement LMC
BG3B1_1	0.265	0.394
BG3B1_2	0.930	0.604
BG3B2_1	0.911	0.598
BG3B2_2	0.315	0.409
BG3C_1	0.555	0.484
BG3C_2	0.596	0.497
BG3C_3	0.418	0.441
BG3C_4	0.718	0.536
BG1_01	46.942	32.274

Note *: The calcite cement percentage was calculated using the Smodej, *et al.* [13] method.

However, screening for diagenesis cannot rely on XRD alone since it is still problematic to detect the occurrence of secondary aragonite [6,14,18,19,20]. Another diagenetic texture that is essential to be screened for is dissolution since it may cause cool SST artifacts [14]. However, it cannot be detected by 2D-XRD. For this reason, all coral materials intended for coral proxy study need to be screened through a combination of methods.

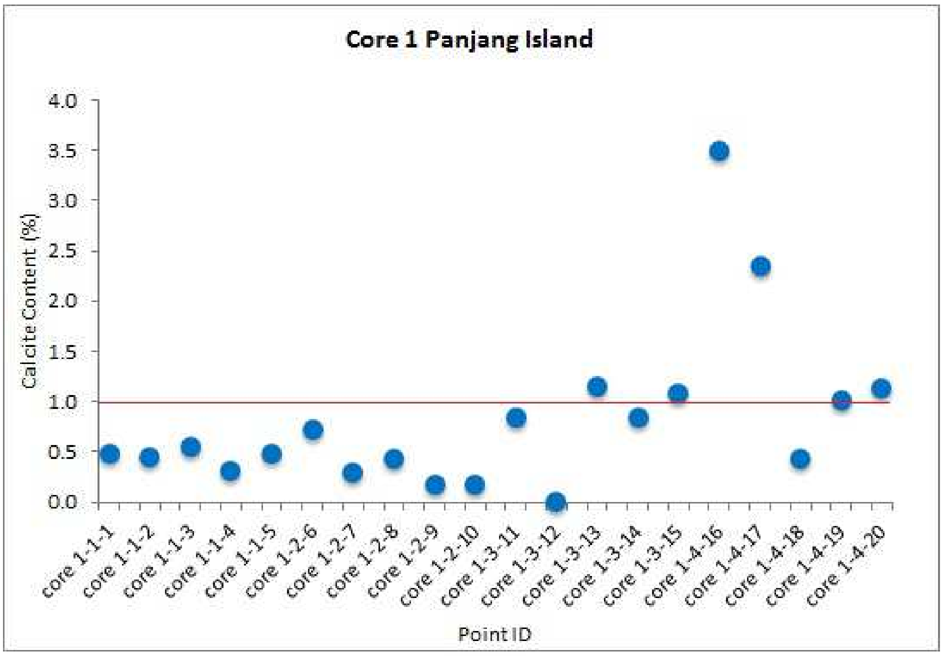

Figure 3 2D-XRD result for core 1 from Panjang Island. Note that the red line is the 1% calcite level as the focus of this study. Even ≤ 1% calcite content may cause alteration of paleoclimate signals [6,8,10]. In the area with ≥ 1% calcite content (above the red line) it is only present localized and as patches. For coral proxy study, the artifacts effect can be avoided by defining alternate sampling transects in an area with ≤ 1% calcite content.

From 2D-XRD point measurement for calcite screening of core 1 (Figure 3), it can be inferred that calcite usually occurs localized or as patches. < 1% calcite content was found in the top and middle part of core 1, while > 1% calcite content occurred in patches in core 1 slab 4 point 16 and core 1 slab 4 point 17 (ID 1-4-16 and 1-4-17). Avoiding misinterpretation due to diagenetic effects is crucial in paleoclimate studies. Defining an alternate sampling transect in the coral samples can be the solution.

3.2 Petrography Analysis

Coral skeletal elements can be seen in unaltered coral thin sections and SEM images (Figure 4). Dark centers of calcification (COC) are visible in the middle of a trabeculae (Figure 4A). COC consist of ≤ 1 micron-sized, randomly oriented, equant crystals that appear dark to opaque in thin sections and that can possibly be surrounded by a brown zone that is a mixture of micron-sized crystals and elongated aragonite needles [21]. In Figure 4B, COC are visible along with daily accretion/daily growth bands (arrow pointing at "COC"). Note the fine growth bands within and perpendicular to the growth direction of the fiber bundles. The width of each daily growth band in coral is 2-3 µm, possibly indicating the size of the calcifying space and representing the extension of the osmotic pressure that builds up between the skeleton and the calicoblastic ectoderm in which crystal extends and creates space during daytime [22].

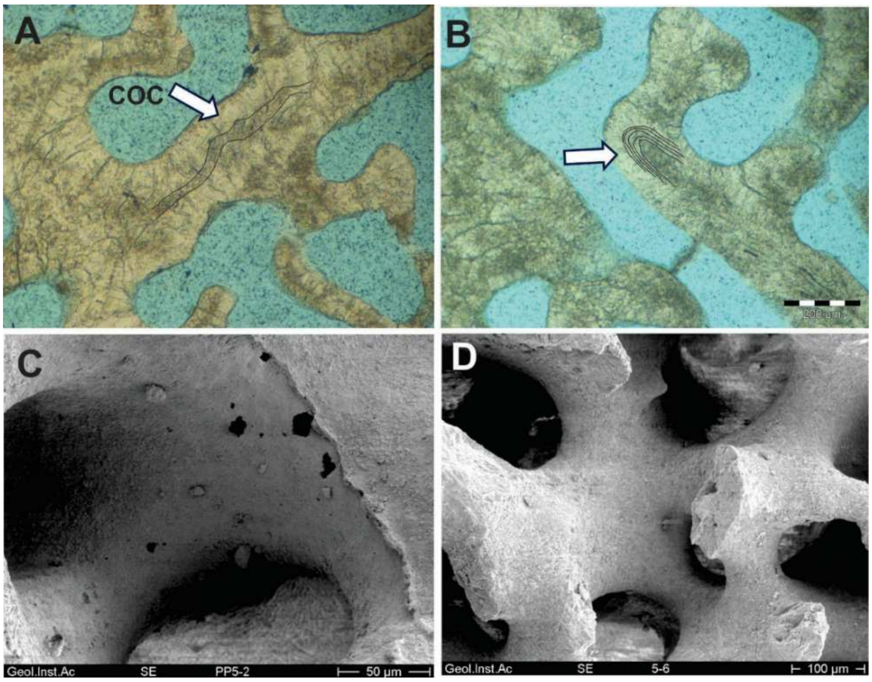

Figure 4 Images of well-preserved coral samples from Panjang Island. (A) Sample PP 5-6 (core 3 slab 6) and (B) sample 5-4 (core 5 slab 4) in plane polarized light (thin sections). (A) The arrow with "COC" points at the center of calcification. (B) The arrow points at daily growth bands. (C) SEM image from sample PP 5-6 (core 3 slab 6), and (D) SEM image from sample 5-6 (core 5 slab 6). The SEM analysis in Figures 4C and 4D show excellent preservation of the fossil coral with no borings, no sediment infillings, or cement present and minimal leaching.

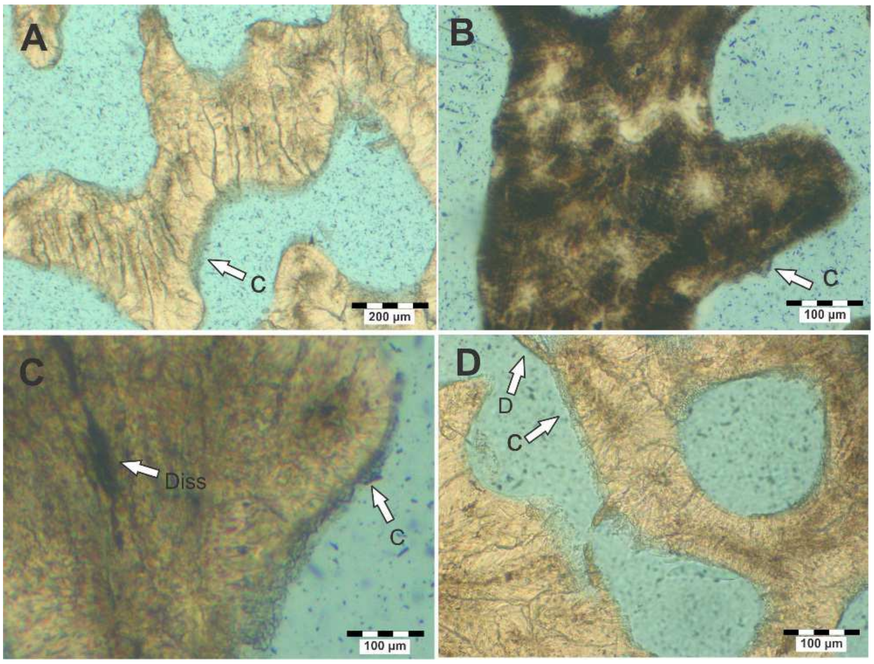

Figure 5 Thin-section images of diagenetic textures associated with ≤ 1% calcite content. A. Sample 1-1/5 (core 1 slab 1) from Panjang Island with ~0.4% calcite content, arrow with "C" pointing at secondary calcite rim growing along the edge of the coral skeleton. B. Sample 2-2/4 (core 2 slab 2 of 4) from Panjang Island with ~0.4% calcite content, arrow with "C" pointing at calcite spar. Calcite often forms dogtooth spar crystal found in vugs. C. Sample BG3B-1 from Kendari with ~0.9% calcite content, showing dissolution in COC (arrow with "Diss") and calcite rim (arrow with "C"). D. Sample BG3B-2 from Kendari with ~0.9% calcite content, showing thin rim of secondary calcite and dissepiment in the coral skeleton (arrow with D); thin (10-40 μm) horizontal layers spaced approximately every 1-2 mm [23]. All images were taken in plane polarized light.

Thin sections, both aragonite and calcite, have similar optical properties in plane polarized and cross-polarized light [24,25]. Inorganic aragonite appears as a fibrous or needle-like structure, while inorganic calcite is likely to have anhedral crystal form [6]. Both calcite and secondary aragonite appear patchy in thin-section images [10, 26]. Secondary calcite can be seen in the thin sections of samples 1-1/5, 2-2/4, and BG3B-2 (Figures 5A, 5B, 5D) as minor, equant-grained calcite crystal overgrowth along the edge of the primary coral material. Sample BG3B-1 (Figure 5C) shows a secondary calcite rim and dissolution. Dissolution is most pronounced along the center of calcification and can be recognized in the thin section by the darkening in the center of calcification and/or by a brown hue surrounding the calcification center [6]. The dark color of COC dissolution visible in the thin sections is due to the internal reflection of the light in minute voids between the dissolved primary aragonite [27]. Dissolution is a diagenetic texture that can cause cool SST artifacts [14,16]. One percent secondary calcite is enough to cause 1°C warming artifacts in absolute Sr/Ca SST reconstruction, although seasonal variability in Sr/Ca SST may still be preserved [7,8,10,16,28,29]. For this reason, in geochemical studies it is highly recommended to scan for aragonite, calcite cement, and dissolution with petrographic and SEM analysis in any areas that are suspected to have diagenetic textures and to use only corals confirmed to be pristine.

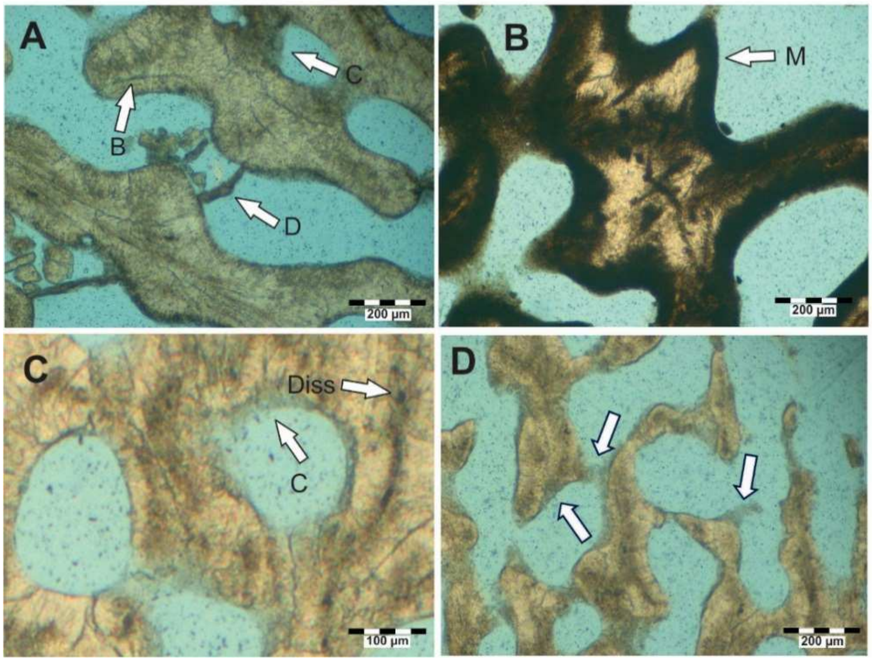

Figure 6 Thin-section images of diagenetic textures associated with > 1% calcite content. (A) Sample 5-4 (core 5 slab 4) from Panjang Island with calcite content ~1.42%. Bioerosion is visible in this image (arrow with "B") as well as thick dissepiment (arrow with "D"), and thickening related to secondary calcite (arrow with "C") growing into a void. (B) Sample PP5-1 (core 3 slab 1) from Panjang Island with ~3.0% calcite content showing the thick dark brown of a micritic calcite rim (arrow with "M"). (C) Sample BG3B-1 from Kendari with ~1.44% showing dissolution in the center of calcification (arrow with "Diss") and secondary calcite rim (arrow with "C"). (D) Sample BG3B-2 from Kendari with ~1.3% calcite content showing thickening rim of secondary calcite (arrow). All images were taken in plane polarized light.

In the thin-section image for the samples with > 1% calcite content, the thickening of secondary calcite rims growing into a skeletal void is clearly visible (Figures 6A, 6C, 6D). Dissepiments and bioerosion also appear in the thin-section images (Figure 6A). In some cases, secondary aragonite precipitation or calcite fillings are found in microbores [7]. In Figure 6A, the secondary mineral fillings in microbores cannot be seen clearly. Sample PP5-1 (Figure 6B) with ~3.0% calcite content shows evidence of micritic calcite rim coating the coral skeleton. Micritization takes place whereby bioclasts are altered while on the seafloor [30], indicating the first marine diagenetic phase [31-33]. Evidence of dissolution in the form of a dark brown color in the center of calcification is possibly due to mineral leaching (Figure 6C). Figure 6D shows more calcite cement rim growing along the skeletal void.

3.3 SEM Analysis

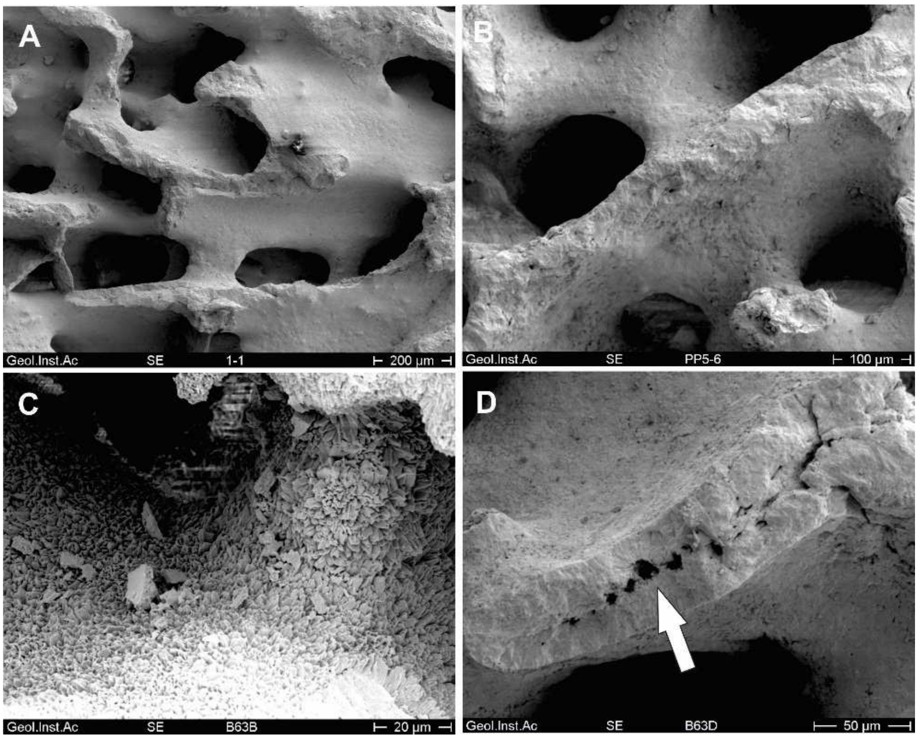

Figure 7 SEM images of diagenetic textures associated with ≤ 1% calcite content. (A) Sample 1-1_2 and (B) Sample PP5-6_3 both from Panjang Island, showing a relatively well-preserved coral skeleton. (C) Sample BG3D_4 from Kendari, showing bladed-type Mg calcite cement distributed on the skeletal wall. (D) Sample BG3B_3 from Kendari, arrow pointing at dissolution of the calcification center.

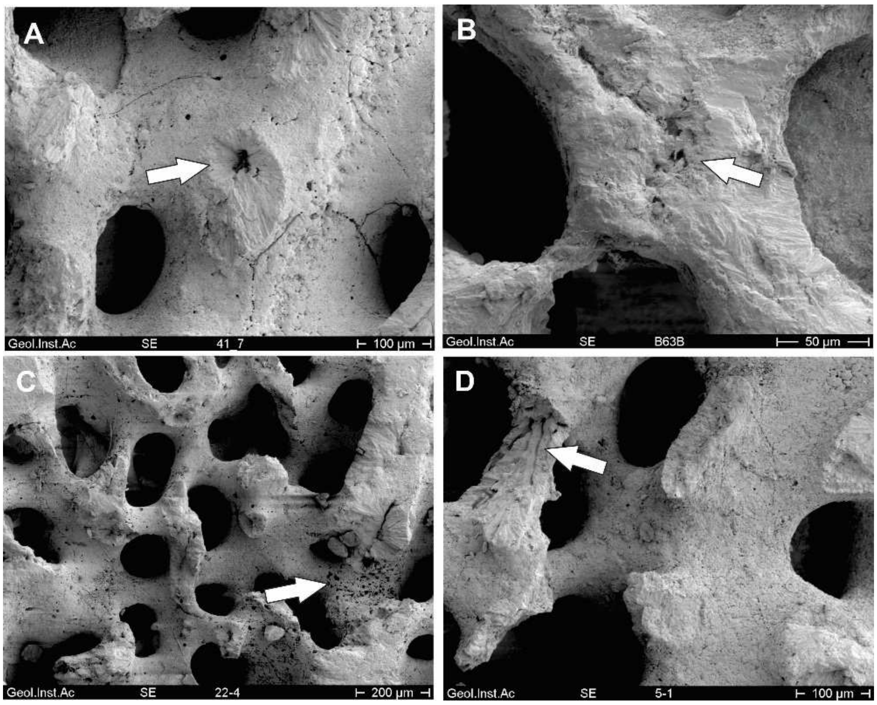

Figure 8 SEM images of diagenetic textures associated with ≥ 1% calcite content. (A) Sample 4-1_7 from Panjang Island, and (B) Sample BG3B_2 from Kendari, white arrows pointing at dissolution as irregular black rounded shapes in the center of calcification. (C) Sample 2-2_4 from Panjang Island, white arrow pointing at dissolution as black dots scattered along the coral skeleton. (D) Sample 5-1_2 from Panjang Island, white arrow pointing at evidence of microbores in center of calcification.

In well-preserved coral samples, SEM observations shows a smooth surface of the fossil coral skeleton with no micro borings, no sediment fillings or cement present and minimal leaching, as can be seen from samples 1-1_2 (Figure 7A) and PP5-6_3 (Figure 7B), both from Panjang Island. Calcite cement has broad variations in abundance and types; it may occur as flat, homogenous and compact-like areas [7], thin, curved, hexagonal plate-like cement to large rhombohedral-scalenohedral block-like cement [16], and as micrite and steep sided rhombs of which dog tooth crystal is produced [8]. The presence of high-Mg calcite cement is common in many corals; usually bladed, they show a gradual increase in width along their length and have an obtuse pyramid termination [8,30,33,34], as can be seen in sample BG3D_7 (Figure 7C). Secondary aragonite and high-Mg calcite are typical shallow marine reef cement [30,35]. Dissolution is observed as irregular black rounded shapes in the center of calcification (Figures 7D and 8A). Dissolution may also appear as black scattered dots on the coral skeleton, as can be seen in Figures 8B and 8C. Bioerosion can be observed in sample 5-1_2 (Figure 8D) in the form of a micron-size boring produced by a micro-organism in the center of calcification.

4 Conclusion

A series of 2D-XRD, petrographic thin sections and SEM observations were demonstrated here as a thorough diagenetic texture screening of coral core samples from Panjang Island, Kendari, and South Pagai (Indonesia) as a starting point for further geochemical studies. All samples presented here were in well-preserved condition and recommended for further climate study, except for sample BG1 of which the calcite content is > 46%. 2D-XRD point measurement indicated that calcite often occurs locally or as patches. Avoiding calcite patches is crucial to prevent misinterpretation in a coral paleoclimate study. 2D-XRD allows calcite screening without destroying the coral sample. If calcite is present in the coral slab then 2D-XRD can help defining alternative sampling transects. Secondary aragonite and dissolution are not recognized by XRD measurement, therefore the screening method of any areas presumed to have diagenetic textures should be combined with other techniques, such as petrographic thin section and SEM.

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