THE APPLICATION OF SEM-EDS FOR THE SURFACE TEXTURE OF FORAMINIFERA: IDENTIFICATION OF EARLY DIAGENESIS OF REWORKED L. DIMIDIATUS WITHIN MODERN COASTAL DEPOSITS

PENERAPAN SEM-EDS UNTUK TEKSTUR PERMUKAAN FORAMINIFERA: IDENTIFIKASI DIAGENESIS AWAL PADA FOSIL ROMBAKAN L. DIMIDIATUS DI ENDAPAN PANTAI MODERN

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ABSTRACT: Sediment recycling has been known to occur within Quaternary coastal barriers of the greater Coorong Coastal Plain, southern Australia. A high degree of reworking of skeletal carbonates from Late Pleistocene deposits (Robe Range) is evident based on the novel application of amino acid dating of the single-foraminifera species *Lamellodiscorbis dimidiatus*. More importantly, some apparently transparent, well-preserved tests indicate anomalously high extents of amino acid racemization, implying that reworked fossils could not be easily distinguished based on taphonomic signatures such as corrosion. Here, we examine the surface microtexture of this species, constrained with well-preserved specimens, on a modern beach of Canunda, southern Australia, using scanning electron microscopy coupled with energy dispersive spectroscopy (SEM-EDS). The aim is to identify surface features of foraminifera in more detail, capturing signs of early diagenesis associated with weakly consolidated Late Pleistocene coastal barriers. The results reveal that some well-preserved tests show localized blocky calcite cementation, most notably within intraseptal spaces or impact sites. The EDS spectra of cement indicate lower Mg content than unaltered foraminifera surface. This suggests low-Mg calcite precipitation due to meteoric diagenesis experienced by the onshore Late Pleistocene coastal barrier. It implies that these foraminifera shells are reworked fossils originated from older successions and were subsequently redeposited within the present-day beach. SEM-EDS used in this study demonstrates its capability in examining small-scale carbonate diagenesis products beyond the traditional binocular microscope. Thus, this tool is recommended to aid amino acid dating in detecting reworked fossils.

Keywords: scanning electron microscope, sediment reworking, foraminifera, *L. dimidiatus*

***ABSTRAK**: Perombakan sedimen sangat umum ditemukan pada lingkungan pantai berumur Kuarter di Dataran Pesisir Coorong, Australia bagian selatan. Proses perombakan yang sangat intensif pada partikel karbonat dari endapan Pleistosen Akhir (Robe Range) telah terbukti berdasarkan aplikasi geokronologi asam amino dengan metode analisis foraminifera tunggal Lamellodiscorbis dimidiatus. Lebih penting lagi, beberapa foraminifera yang transparan dan terpreservasi baik menunjukkan tingkat rasemisasi asam amino yang sangat tinggi, mengindikasikan bahwa fosil yang mengalami perombakan tidak dapat dibedakan hanya berdasarkan ciri taphonomik seperti korosi. Pada studi ini, kami melakukan analisis mikrotekstur pada permukaan pada spesimen foraminifera dengan preservasi tinggi di pantai Canunda, Australia bagian selatan, menggunakan mikroskop elektron yang dipadukan dengan spektroskopi dispersi energi (SEM-EDS). Tujuan penelitian ini yaitu mengidentifikasi fitur permukaan secara detil untuk melihat tanda-tanda diagenesis awal pada endapan pesisir pantai berumur Pleistosen Akhir yang terkonsolidasi lemah. Hasil penelitian menunjukkan bahwa beberapa foraminifera dengan tingkat preservasi tinggi memiliki sementasi blok kalsit yang terlokalisir pada intraseptal, pori atau area bekas tumbukan. EDS* *spektra pada semen menunjukkan konsentrasi Mg yang relatif lebih rendah daripada permukaan foraminifera yang belum teralterasi. Hal ini menunjukkan pengendapan kalsit dengan Mg rendah hasil diagenesis meteorik yang dialami oleh endapan pantai berumur Pleistosen Akhir. Foraminifera tersebut kemungkinan merupakan fosil rombakan hasil erosi dari suksesi yang lebih tua, yang kemudian diendapkan kembali di pantai Canunda pada saat ini. Pada penelitian ini, SEM-EDS dinilai baik untuk mengetahui produk diagenesis karbonat secara lebih rinci dibandingkan dengan mikroskop binokular untuk membantu geokronologi asam amino dalam mendeteksi butiran karbonat hasil rombakan.*

Kata Kunci: *mikroskop elektron, perombakan sedimen, foraminifera, L. dimidiatus*

INTRODUCTION

Scanning electron microscopy (SEM) has been extensively applied to geological samples because of the versatility of this instrument in providing direct, high-resolution images with excellent depth of focus at a minimal cost and time for sampling preparation (Chen et al., 2015). In the study of sedimentology, SEM is commonly used to examine micro-characteristics of grains, mainly quartz, to understand the specific transportation and inferred environments (Krinsley and Doornkamp, 1973; Vos et al., 2014). SEM, coupled with elemental analysis such as energy dispersive spectroscopy (EDS), is also important in deciphering the diagenesis route of Quaternary coastal dune deposits (Loucks and Patty, 2017). In terms of calcareous microfossils, several studies have relied on EDS instrument to determine unusual elemental compositions that lead to abnormal shells influenced by contaminated marine environments, particularly foraminifera and ostracoda (Dewi et al., 2016, 2015).

It has been suggested that Holocene skeletal particles act as the primary carbonate sediment source for the accumulation of modern beaches along the Limestone Coast (Joury et al., 2018). The basis of this interpretation is the assumption of high preservation of skeletal particles determined from an optical microscope that is in line with inferred age. In this case, the well-preserved appearance of skeletal carbonate grains is defined as modern to the Holocene, whereas brownish iron-stained particles indicate relict grains from preceding successions, particularly older than the last glacial period (Joury et al., 2018; Rivers et al., 2007). However, using optical luminescence dating, Oliver et al. (2019) mentioned the possibility of enhanced sediment delivery not only from Holocene-modern carbonate factories but also a reworking process based on the relatively rapid shoreline progradation since middle Holocene in this region of up to 74,000 m^3/yr. A subsequent study by Hidayat (2022) has supported this argument, suggesting that these reworked carbonate particles were derived primarily from the eroded Late Pleistocene Robe Range and thus, it plays a significant role in supplying sediments for present-day beaches in this region based on the results from amino acid dating of the single-foraminifera method. Moreover, some apparently pristine foraminiferal tests are reworked fossils due to the unusually high amino acid racemization, which could not be assigned as Holocene or modern specimens. This challenges conventional optical microscopes to provide more detailed characteristics of skeletal particles to be confidently given as reworked fossils from Late Pleistocene succession.

Here, we apply SEM-EDS to provide further evidence of diagenetic features within reworked benthic foraminifera *Lamellodiscorbis dimidiatus* from Late Pleistocene Robe Range successions that show relatively pristine conditions from binocular microscopy. *L. dimidiatus*, a member of the genus *Lamellodiscorbis*, is regarded as one of the key faunal assemblages supplying beach deposits in the Limestone Coast, having typically inner shallow-marine habitat associated with temperate carbonate environments, mainly in seagrass meadows (James and Bone, 2011). The selection of this species is based on its robustness and relatively high abundance within the beach deposits (Joury et al., 2018; Oliver et al., 2020), acting as one of the primary carbonate sources from shallow-marine environments. The meteoric diagenetic signatures experienced by Late Pleistocene successions within the southern part of the Coorong Coastal Plain have been noted by James et al. (2018) based on the slight increase of low-Mg calcite proportion compared to Holocene deposits. The significantly higher magnification provided by SEM performed in this study may be suitable to determine possible small-scale (microns in size) of early diagenetic products from subaerially exposed Robe Range coastal dune deposits found within foraminiferal tests, which could not be previously seen in conventional microscopy.

Coastal Setting

Canunda Beach (140°13'17.1"E, 37°39'31.7"S) is located south of Rivoli Bay, southern Australia. It has characteristically long elongated beach landform, around 45 km, with extensive and partially vegetated Holocene coastal dunes up to 4 km in cross-section (Fig. 1). The beach shows large foredunes around 10 m tall, whereas the Holocene coastal dune may reach 30 m high. The earliest development of the Holocene dunes is thought to begin ~8 ka ago (Ohmori et al., 1987). In contrast to the shoreline north of Rivoli Bay (Beachport to Robe), eroded remnants of the Late Pleistocene coastal barrier, termed the Robe Range, such as sea stacks (Fig. 1), nearshore islands, shore platforms, and pocket beaches, are only present locally in this area. It is suggested that limited erosional remnants from older deposits with high accumulations of Holocene coastal dunes may be related to the higher degree of

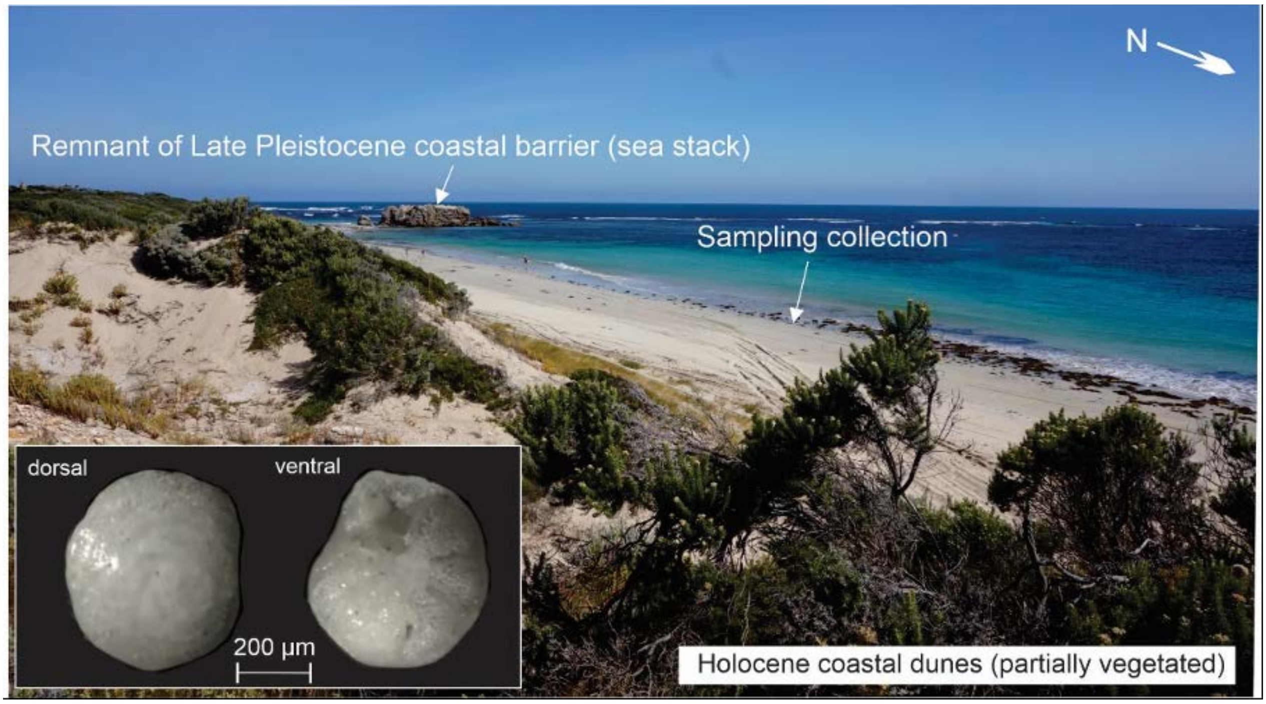

Figure 1. Location of sampling sites at Canunda Beach, southern Australia, showing coastal landform with partially vegetated Holocene onshore dunes and an erosional remnant of older successions associated with Late Pleistocene Robe Range coastal barrier. Inset: representation of *L. dimidiatus* taken from the sample.

coastal erosion. This is supported by the high proportion of reworked *L. dimidiatus* tests and the relatively high extent of racemization from the whole-rock and foraminifera samples previously reported in this region, particularly in prograding ridges of Guichen Bay, 70 km northwest of Canunda Beach (Murray-Wallace et al., 2001; Oliver et al., 2020).

From a hydrodynamic perspective, Canunda Beach is considered one of the most energetic beaches on the southern Australian coast (Short, 2020). Joury et al. (2018) modeled the conditions of Canunda Beach, suggesting that the relatively steep bathymetry immediately offshore of this area generates optimum bottom velocity outside the surf zone up to 1.1 m/s at 15 m of water depth, providing a high chance of waves to scour seafloor sediments. This results in the typically unconsolidated bare sand blanketing the nearshore zone of Canunda (Miller et al., 2009).

MATERIALS AND METHODS

The sediment sampling of foraminifera *L. dimidiatus* was carried out 20 cm from the beach surface. Ten transparent and well-preserved tests were collected from the binocular microscope with a typical size of around 500 microns. Following this, an ultrasonic bath for 1 minute and 2 hours of hydrogen peroxide soak were established to remove adhering particles. Collected *L. dimidiatus* tests were mounted on adhesive-taped SEM stubs for surface micromorphology. After that, specimens were coated using gold to avoid the charging effect. Microtextures were analyzed using a JEOL JSM-6490LV instrument, combined with a large EDS X-ray detector (80 mm^2) at the Electron Microscopy Centre, the University of Wollongong, Australia. We generally used secondary electron imaging between 15 and 20 kV depending on magnification, with a 10 mm working distance and spot size at 60. Elemental spot analyses, covering an area of around 1 μm^2, from the EDS tool focused on carbonate diagenetic features within the surface of identified *L. dimidiatus* and compared them with the fresh or unaltered part to determine any relative difference in Mg intensity. Chave (1954) noted that the genus of this species commonly has around ~15% $MgCO_3$ and is considered as intermediate to high Mg-calcite carbonate skeletons.

RESULTS

In this study, we opted to select four *L. dimidiatus* tests (CB/LD-1 to CB/LD-4) for further discussion regarding microtextural characteristics. The description of each foraminifera is based on a general overview of the overall conditions, followed by a more detailed image of the focus area with associated EDS analysis. EDS spectra were also established in the fresh, unaltered part to understand the typical Mg intensity compared with diagenetic features found on the foraminiferal tests.

Generally, the well-preserved foraminifera shell (CB/LD-1) displays minor physical breakages, around 20 to 80 microns, and some pittings resulting from bioturbation (Fig. 2a, 2b). Small pits may be formed by the energetic hydrodynamic setting of offshore Canunda, as previously suggested by Joury et al. (2018). Despite the relatively clean and smooth surface of *L. dimidiatus* coded

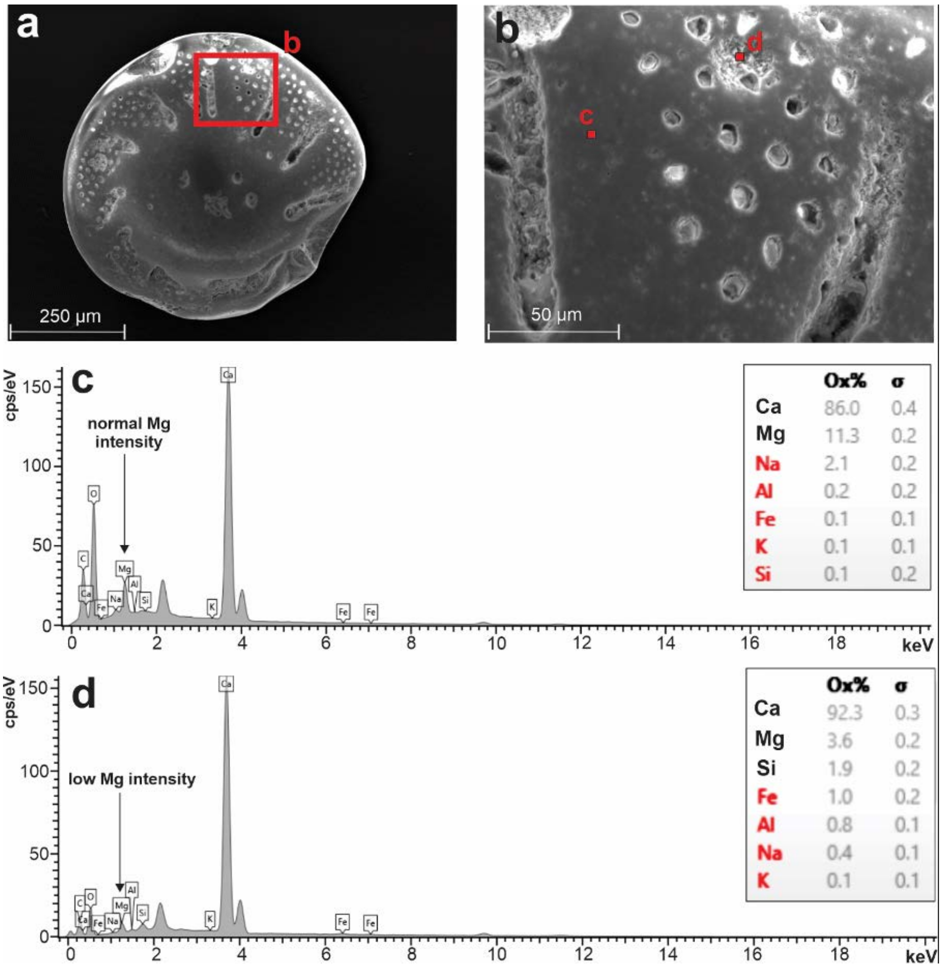

Figure 2. (a) SEM image of overall foraminiferal test CB/LD-1. (b) Localized blocky cement occurs within the impact zone and intraseptal spaces. (c) The EDS spectrum of the fresh surface indicates relatively high Mg intensity as opposed to (d) the EDS spectrum of cement within the impact zone.

CB/LD-1, some isolated blocky calcite crystals can be seen within a small impact pit (Fig. 2b). The more detailed EDS spectrum of this crystal indicates a significantly lower Mg intensity than the unaltered test wall (Fig. 2d), possibly associated with low-Mg calcite precipitation from meteoric diagenesis. It shows that the blocky crystal has a content of Mg at 3.6%, significantly less than the unaltered part at 11.3% (Table 1). Similarly, within the intraseptal space of *L. dimidiatus* coded CB/LD-2, some features of blocky calcite can be seen within the smooth depression region (Fig. 3b). Further assessment from the EDS tool suggests a comparatively low Mg concentration (Fig. 3d), whereas the intensity of Mg from the fresh surface shows similar values with foraminiferal test CB/LD-1, at 9.7% of normalized oxide (Table 1).

Despite the apparently well-preserved microtexture, the SEM image of *L. dimidiatus* CB/LD-3 indicates more substantial diagenetic alteration than other specimens (Fig. 4b). Some subtle depressions occur as a result of slight dissolution, possibly happened during the vadose subaerial exposure after deposition of Late Pleistocene Robe Range dune complexes. This was followed by extensive precipitation on the majority of the outer surface by low-Mg calcite, as recorded from the low intensity of Mg peak determined from EDS analysis (Fig. 4d) compared to surfaces lacking diagenetic features. The precipitate contains almost entirely Ca with low Mg (5.0%) whereas the unaltered part has a substantially higher Mg concentration at 10.5% (Table 1). In contrast to other *L. dimidiatus* tests selected in this study, foraminiferal test CB/LD-4 yields an overall unaltered surface with limited impact pits (Fig. 5a, 5b). There is no visible localized calcite precipitation within pores or intraseptal spaces, where the EDS analysis of these regions often demonstrates consistently high Mg intensity, showing 12.2% respectively (Table 1). Interestingly, within depression area, many parts contain silica globules as evidenced by elevated Si intensity based on EDS spot check (Fig. 5d), resulting in 2.1% of normalized oxide (Table 1). Based on the insufficient evidence of meteoric diagenesis products, it is suggested that this foraminiferal test may be modern or Holocene in origin and unlikely to be reworked fossils.

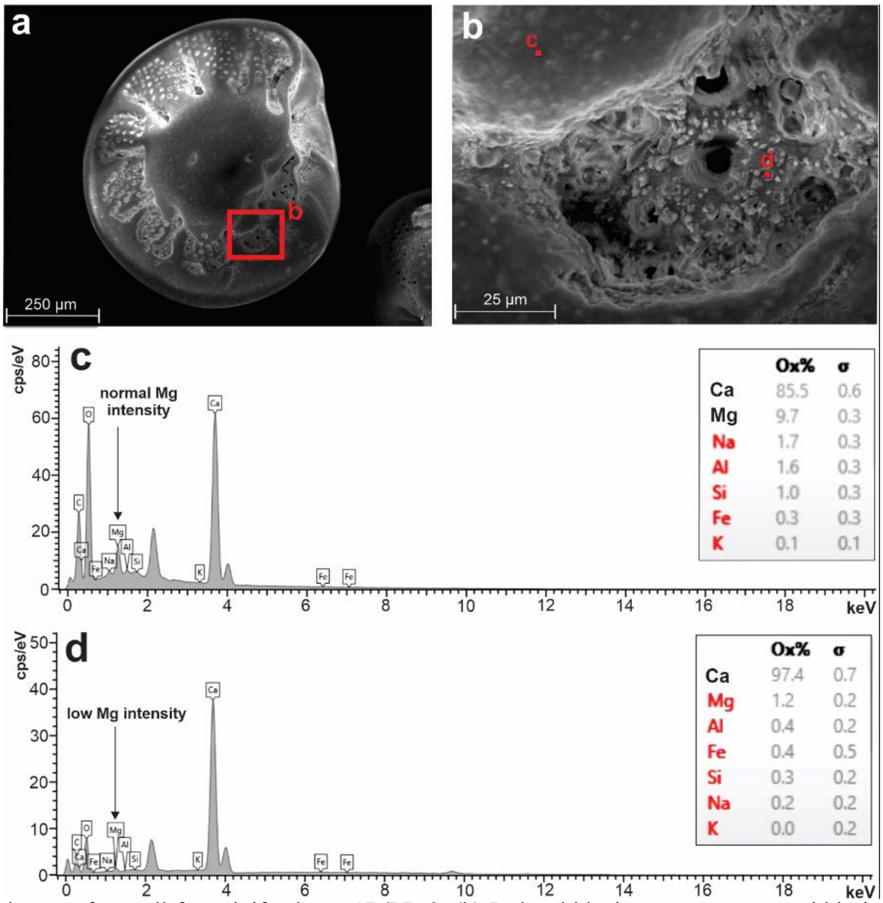

Figure 3. a) SEM image of overall foraminiferal test CB/LD-2. (b) Isolated blocky cement occurs within intraseptal spaces. (c) The EDS spectrum of the unaltered surface indicates relatively high Mg intensity as opposed to (d) the EDS spectrum of cement within intraseptal space.

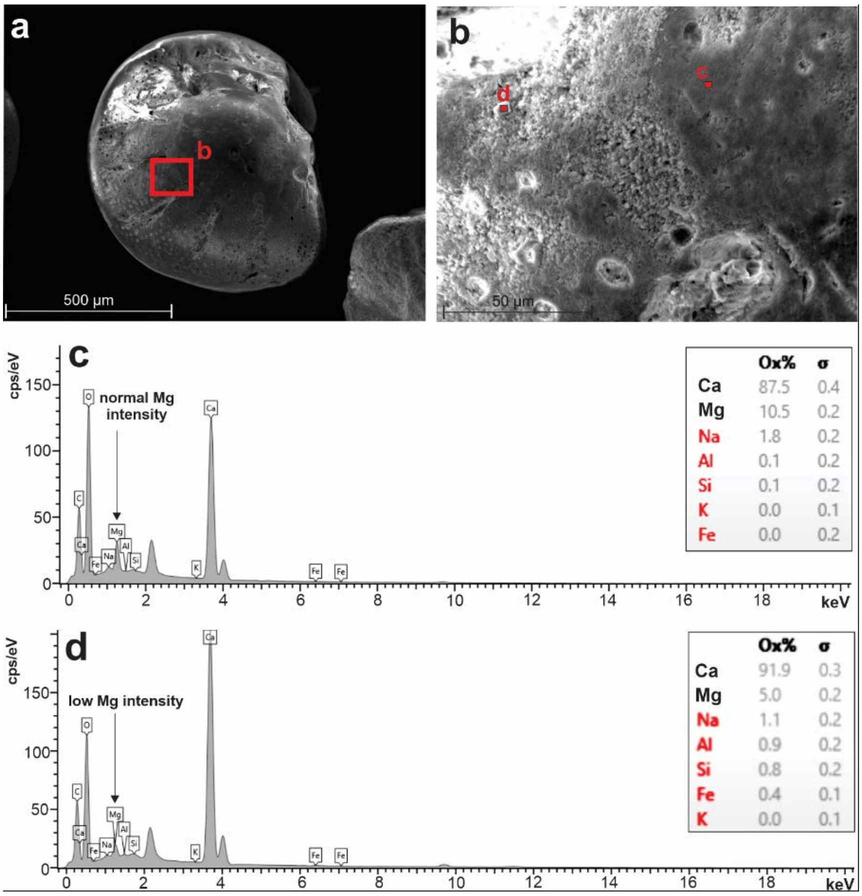

Figure 4. (a) SEM image of overall foraminiferal test CB/LD-3. (b) Broad cementation occurs within the subtle depression, likely created by dissolution. (c) EDS spectrum of fresh surface indicates relatively high Mg intensity as opposed to (d) EDS spectrum of cement within the depression.

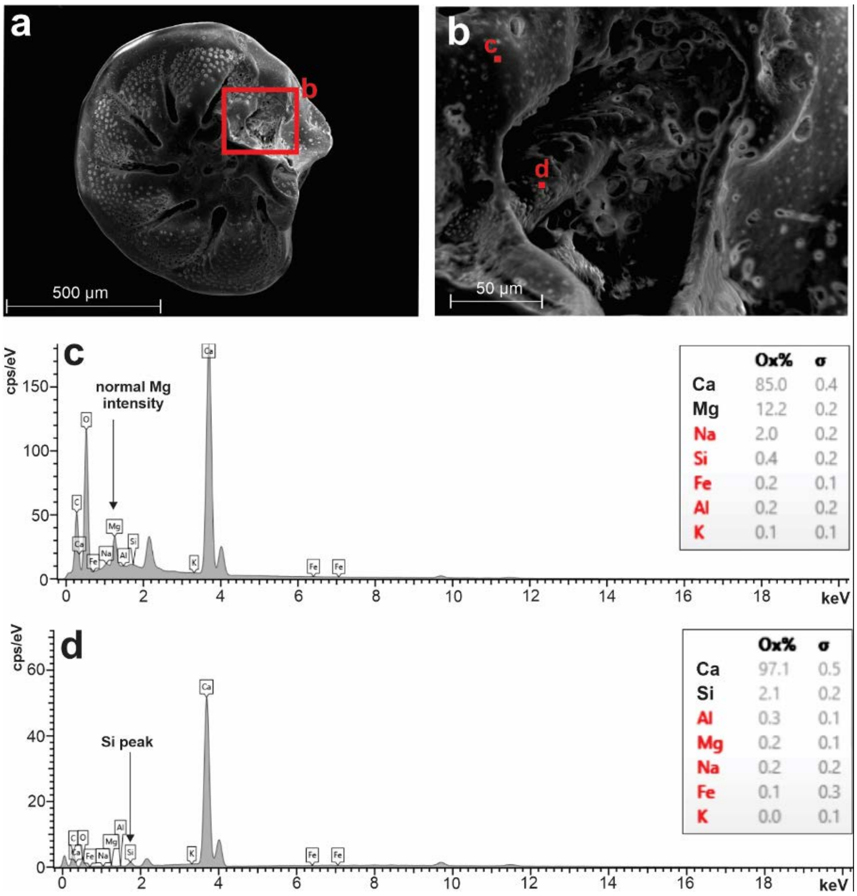

Figure 5. (a) SEM image of overall foraminiferal test CB/LD-4. (b) Silica globules on the intraseptal spaces but no indication of blocky calcite cementation. (c) EDS spectrum of the fresh surface indicates relatively high Mg intensity, whereas (d) the EDS spectrum of silica globules with a notable peak of Si.

Table 1. Chemical composition foraminifera *L. dimidiatus* resulted from EDS spectra shown in Figure 2-5.

Sample	Features	CaO	MgO	SiO_2	Al_2O_3	Fe_2O_3	NaO	K_2O
CB/LD-1 (Fig. 2c)	Unaltered surface	86.0	11.3	0.1	0.2	0.1	2.1	0.1
CB/LD-1 (Fig. 2d)	Precipitates on impact zone	92.3	3.6	1.9	0.8	1.0	0.4	0.1
CB/LD-2 (Fig. 3c)	Unaltered surface	85.5	9.7	1.0	1.6	0.3	1.7	0.1
CB/LD-2 (Fig. 3d)	Precipitates on intraseptal space	97.4	1.2	0.3	0.4	0.4	0.2	n.d.
CB/LD-3 (Fig. 4c)	Unaltered surface	87.5	10.5	0.1	0.1	n.d.	1.8	n.d.
CB/LD-3 (Fig. 4d)	Precipitates on depression area	91.9	5.0	0.8	0.9	0.4	1.1	n.d.
CB/LD-4 (Fig. 5c)	Unaltered surface	85.0	12.2	0.4	0.2	0.2	2.0	0.1
CB/LD-4 (Fig. 5d)	Globules	97.1	0.2	2.1	0.3	0.2	0.2	n.d.

n.d.: note detected

DISCUSSION

The surface microtexture of benthic foraminifera *L. dimidiatus* provides supporting evidence of amino acid dating from previous studies (Oliver et al., 2020) that the determination of reworked sediments based on the visual taphonomic assessment by using a conventional microscope should be used with caution. Here, the well-preserved foraminiferal tests may experience diagenesis based on the presence of calcite precipitates, localized to mainly depression areas such as impact pits or intraseptal spaces (Fig. 2b, 3b). The consistently low intensity of Mg compared to the unaltered part based on EDS spot analyses suggests the regular route of early vadose diagenesis associated with blocky low-Mg calcite. Thus, despite their pristine appearance, these foraminifera are considered a product of sediment recycling from currently eroded Late Pleistocene coastal deposits. Hidayat et al. (2023) noted that the development of onshore Late Pleistocene Robe Range barrier structure correlates with marine isotope stages (MIS) 5c–5a and early MIS 3. During glacial sea levels, particularly MIS 4 and MIS 2, the more arid conditions may induce precipitation of low-Mg calcite on this coastal barrier. Calcretization also developed extensively at this stage, covering and protecting the morphology of Late Pleistocene sedimentary units, mainly landward-advancing eolianite facies, prior to the formation of overlying Holocene coastal dunes (Murray-Wallace, 2018). In Late Pleistocene eolianites in Warrnambool, Victoria, the typical precipitation of blocky low-Mg calcite is also common, filling some of the voids and dissolving shells, particularly with aragonitic skeletons (Reeckman and Gill, 1981). The small scale of these precipitates may not be seen with the low resolution of binocular microscopy, reducing the reliability of identifying reworked skeletal carbonate grains.

It has been known that the iron-oxide coating of grains of the coastal dune is attributed to the prolonged pedogenic-related weathering process (Khadkikar and Basavaiah, 2004); thus, the majority of bioclasts show a brown-staining or corroded appearance. Based on an amino acid dating study (Hidayat, 2022), overall brown-stained *L. dimidiatus* yielded exclusively high extents of amino acid racemization corresponding to the Late Pleistocene age. However, given the nature of the general characteristics of the internal part of Robe Range eolian dune outcrops that are weakly consolidated (Hidayat et al., 2023), the influence of pedogenesis is possibly limited temporally and not as spatially extensive to the overall stratigraphical succession. Except for the uppermost part associated with protosol or *terra rossa* paleosol and associated calcrete layer, it is possible that some bioclasts within the Robe Range, particularly away from the pedogenized interval, have a minimal influence of iron-oxide staining or excessive vadose diagenetic precipitation. This may explain the occurrence of some well-preserved reworked skeletal grains derived from erosion of Robe Range barrier structures. During the coastal erosion that started since the end of post-glacial marine transgression, both preserved and iron-oxide-stained bioclasts within Robe Range eolianites were scoured, remixed, retransported by wave action, and ultimately redeposited in the modern beach deposits.

The imaging of SEM-EDS results can also be used to characterize Holocene foraminifera specimens. As opposed to the reworked fossils, the overall exterior of the shell does not show features associated with low-Mg calcite formation (Fig. 5a). In depression regions, the surface remains entirely smooth without any development of small calcite crystals (Fig. 5b). Some undulations are observed in some areas, probably linked to the formation of amorphous silica derived from ocean water. The occurrence of silica globules is possible due to silica precipitations where the foraminifera experienced wetting-drying around the intertidal zone (Chakroun et al., 2009). Moreover, wetting-drying is more effective in the coastal environments within the Mediterranean-type climate setting, as Chakroun et al. (2009) reported. In Canunda Beach, Hidayat (2022) also reported that many *L. dimidiatus* tests yield low extents of amino acid racemization, indicating a minor contribution of skeletal carbonate grains from the modern–Holocene carbonate factory. Based on visual inspection, these foraminiferal tests showed exclusively excellent preservation without signs of corrosion or iron-oxide staining (Hidayat, 2022). Since the incorporation of these Holocene skeletal grains into the present-day beach deposit took place in a relatively short period, it is unlikely that these grains have experienced sufficient subaerial weathering that leads to brown staining and shell corrosion in contrast to the relict counterpart.

CONCLUSIONS

The SEM-EDS established in this study has aided the previous amino acid racemization dating studies related to the presence of well-preserved reworked foraminiferal tests from the Late Pleistocene Robe Range coastal barrier that were re-deposited as present-day Canunda Beach. The capability of SEM to capture small-scale and localized calcite precipitations within depression areas such as intraseptal spaces, pores, and impact pits of these foraminiferal tests is essential to assigning foraminifera or other calcareous skeletons as relict or reworked more reliably than the optical microscope. More importantly, the semi-quantitative analysis of EDS provides valuable information that these precipitations consistently have lower Mg intensity as opposed to the unaltered or fresh surfaces. This implies typically early diagenetic calcite cementation (low-Mg) that is common in Late Pleistocene coastal dune deposits elsewhere with similar climatic settings, for instance, in Warrnambool, Victoria.

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