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Comparative characteristics of cement materials in natural and artificial beachrocks using a petrographic method

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23 **[Abstract]**

24 Beachrock is among the important features of tropical coastlines. It appears to have an anchoring
25 effect on dynamic islands that provides protection from erosion. However, the origin of cement
26 micritic peloidal remains uncertain. Petrographic analysis is a method used by many geologists to
27 accurately identify specific aggregated minerals present in an area. It also helps to understand
28 historical petrogenesis interpretations of a sedimentary rock formation and cementation process
29 inside rock particles. In this study, petrographic analysis was used to identify the structure, texture,
30 composition, and presence of minerals from beachrock samples collected from Okinawa, Japan
31 and Sadranan beach, Yogyakarta, Indonesia. Field investigations and laboratory analysis
32 (petrographic and geochemical measurements) were aimed at understanding the formation
33 mechanism of natural fresh beachrock.

34 Subsequently, laboratory-scale experiments on artificial beachrock were based on solidification
35 tests and were conducted to use microbial-induced calcium carbonate precipitation (MICP) with
36 *Pararodhobacter* and *Ocenisphaera* bacterium species to draw comparisons between natural
37 beachrock and artificial beachrock. The cementation process based on petrographic analysis of
38 thin sections has an assumption that the cement type and other added materials determine the
39 strength of the material, and that the cement mineral occurring represents the sedimentary
40 environment. The cement mechanism behavior of natural beachrock has potential in
41 manufacturing artificial beachrock using the MICP method, an eco-friendly development method
42 for coastal areas.

43 [Keywords]: petrography, beachrock, coastline, MICP, artificial beachrock

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1. INTRODUCTION

Marine seafloor sedimentary processes and the study of mechanism behaviors opens the door to many questions. Beachrock is among the recent seafloor sedimentary products that occurs on the shoreline as a carbonate compound and natural wave breaker. The terminology of beachrock is generally found in the tropical-subtropical zones that had warmer temperature climate. Beachrock studies have shown that the formation of the diachronic process occurs at lower latitudes. Thus far, beachrock deposit occurrences have been noted in Turkey, Bangladesh, Scotland, Japan, Greece, the Great Barrier Reef of Australia, Sri Lanka, Bahamas Island, Brazil, South Africa, and in Florida in the United States (Danjo and Kawasaki, 2014; Khan and Kawasaki, 2016). Beachrock is cemented and rapidly forms in the areas as a recent carbonaceous deposit (Voudoukas, et al., 2007; Desruelles et al., 2009; Mauz et al., 2015a; Ozturk et al., 2016; Karkani. et al., 2017; Kolaiti and Mourtzas, 2016).

Recent findings on beachrock are still debatable, as there are various issues still unresolved such as the geographical locations and the key processes that occur in the formation of beachrock from observations in the field. Some researchers have proposed intertidal formation (e.g., Neumeier, 1999; Voudoukas et al., 2007; Desruelles et al., 2009; Mauz et al., 2015; Saitis, 2016) while other specific results of beachrock show sedimentary binding with an aragonitic composition cement occurring in the shallow marine zone, while low-magnesium calcite (LMC) indicates the supratidal zone, high-magnesium calcite (HMC), and also aragonite that indicates the shallow-marine zone (Stoddart and Cann, 1965a, b; Scholle and Ulmer-Scholle, 2003; Mauz, et al., 2015). However, learning from a natural analogy of the sedimentary process may yield valuable information to understand the genesis of beachrock on the basis of the cementation characteristics and sediment bedding information. The mineral composition of beachrock deposits is mainly CaCO_3

polymorphs, which are aragonite, LMC, and HMC. The dominance of calcium mineral compounds depends on physicochemical parameters, the genetic processes involved in them, and metabolic residual products of living organism activities (Calvet, et al., 2003; Kneale and Viles, 2004; Díez, et al., 2007).

Several techniques and research practices have been put forward to identify beachrock formation whilst characterizing them. Petrographic analysis is an important method to identify specific aggregate minerals and understand petrogenesis interpretations with a focus on the structures, textures, and compositions of minerals inside rock samples. The mineral content and the textural relationship of rocks including cement material analysis is based on microscope petrographic images. Geochemical analysis as X-ray diffraction (XRD) and X-ray fluorescence (XRF) were conducted to reveal the micromorphology of the cement in the beachrocks. This study aims to investigate the cementation characteristics of natural beachrock and compare them to those of artificial beachrocks, using petrographic measurement and geochemical for the additional analysis. There have been many assumptions that the cement types and other additional materials determine the strength of the material. The distributions of the beachrocks deposits so far found in the tropical-subtropical countries, but there is limitation data in Asia. The natural beachrock were found in Asia came from Japan and Bangladesh (Danjo and Kawasaki, 2014; Khan and Kawasaki, 2016). Sup-tropical and tropical country in South East Asia, like Indonesia may have potential of natural beachrock deposits. In other hand, artificial beachrock is a developing technology that has self-repairing capabilities for use in coastal areas. It is an eco-friendly, inexpensive technology and the carbonate cement is assumed to harden over time for the shoreline development technique. It may be able to form at an accelerated rate of consolidation with a microorganism reagent. In the future, MICP technique may be possible to manufacture artificial rocks for erosional control

purposes using compositional substances as similar to this artificial beachrock. Bio-cementation technology due to its suitability to field application (Khan and Kawasaki, 2018). In this research were explained only how to make artificial beachrock using solidification test within limitation of cultivation bacteria, such as temperature, pH, medium, and marine environment. A comparison of natural and artificial beachrock and their cementation characteristics are vital information when trying to decide the nature of beachrock with the focus is to compare the characteristic cementation process.

2. METHODOLOGY

The research was completed using geological research methods of identification of the sedimentary environment using petrographic analysis which included observations, physical characteristics, and data measurements using petrographic analysis and a geochemical approach (Potter, 1967; Pozo, et al., 2016; Ayinla, et al., 2017; Nabhan and Yang, 2018). This research was completed by observation and investigation from the geological field investigation on December 2015 in Okinawa, Japan and Sadranan, Yogyakarta on May, 2016. The identity of beachrock in the study area with an emphasis on the aspect of sedimentation and cementation. A flowchart for this research can be seen in figure 2.

2.1. Field Work

A total of 12 natural beachrock samples were collected from Okinawa, Japan and Yogyakarta. Moreover, nine samples of artificial beachrock based on solidification tests of calcium carbonate into syringe-tubes were studied.

2.2. Petrographic Analysis

The thin-section samples were collected at different zones from tidal to onshore locations to assess the degree of cementation and analyze under a microscope (Olympus CHA-P) to determine the carbonate-composition types of the cement and the characteristics of natural deposition. Qualitative analysis included an analysis of a thin section method with parallel and crossing Nicol polarizations microscope to identify the texture, structure, and composition of the thin-section samples. The point counting was conducted for classification of the composition fossils, matrixes of carbonate (cemented materials) and carbonate cementation (cement). The point-counting technique is based on grid lines from the microscope petrographic analysis, and statistical measurement with different angles of thin-section images. Petrographic studies have attempted to define the underlying reason for the composition, cement materials, depositional environment, and the origin of clasts for beachrocks (Arrietta, et al., 2011; Christ, et al., 2015; Avcioglu, et al., 2016; McCutcheon, et al., 2016).

2.3. Geochemical XRD and XRF

The research findings presented use geochemical analysis (XRD and XRF). This form of analysis is usually used when trying to determine mineral composition and the enrichment of elements that can be found in the beachrock. Each sample finding for different locations represented a swash zone (intertidal, the narrow region of sediment between land and sea) in the different locations. In the XRD experimental analysis using a MiniFlexTM (Rigaku Co., Ltd., Tokyo, Japan), a natural beachrock sample was dried at 105° C and ground into powder manually. XRD scans were recorded from 5 to 80° 2 θ , at rate of 20° per minute. The crystalline phases were identified using the International Center for Diffraction Data (ICDD) database and Match 3.4 (Klaus and Putz, 2017).

2.4. Artificial Beachrock

On the other hand, the important factors controlling calcium carbonate cementation of artificial beachrock are the chemistry of the seawater (the source of the cementing material); the physical conditions, in terms of hydraulic energy; sedimentary parameters such as permeability, texture, and composition of the sand; and an initial stabilizer of the loose sediment. Based on the research of Danjo and Kawasaki (2014), they analyzed the microorganism that worked on the precipitated solid and used urease bacteria for the microorganism reagent that is famous from an Okinawa sand sample termed *Pararhodobacter* sp. These types of species are strong during solidification at the laboratory scale because this species has the highest urease activity. This research tried to combine two species of urease bacteria, *Pararhodobacter* and *Oceanisphaera*, (Miyakojima bacteria-silica deposits) to precipitate artificial beachrock.

2.4.1. Isolation of Ureolytic Bacteria

Sand near the beachrock in Sumuide, Okinawa, Japan was collected into sterile tubes and then proceed in the Hokkaido University Laboratory, the bacteria inside tubes diluted 10^1 - 10^4 times with artificial seawater, after that 10 μ L of each dilution was added to ZoBell2216E agar medium for marine bacteria. After incubation at 30°C for 3 days, the bacteria colonies were isolated from one plate. Cells were cultured in ZoBell2216E medium, which contained 5.0 /L hipolypeptone (Nihon Seiyaku Co., Ltd., Tokyo, Japan), 1.0 g/L FePO₄ (Junsei Chemical Co., Ltd., Tokyo, Japan), and 1.0 g/L yeast extract (BD Bioscience Adv., Bioprocessing, Miami, FL., USA), prepared with artificial seawater with pH adjusted 7.6-7.8 using 1 M NaOH (Fujita, et al., 2017).

2.4.2. Microbial Population Count and Urease Activity Test

Seawater, beachrock samples, and sand from both sites were collected in the sterile tubes and isolation for colonies hybrids. Subsequently, isolated colonies from nature sand adjacent to

beachrock in Okinawa, Japan using ZoBell2216E medium containing *Pararhodobacter* sp., and *Oceanisphaera* sp., the time course of cell growth was determined based on the optical density (OD) value 600nm by UV-vis spectrophotometer (V-730, Jasco International CO., Ltd., Tokyo, Japan for 14 days. The urease activity of bacterial cells was determined by monitoring ammonium ions generated in urea hydrolysis using the indophenol blue method based on Berthelot's reaction in a water bath (Weatherburn, 1967; Natarajan, 1995). After bacterial cultivation for 48 hours, a bacterial suspension (1mL) ($OD_{600}=1.0$) was added to 0.1M phosphate buffer containing 0.1 M urea. Samples were then collected at intervals of 5 minutes (0, 5, 10, and 15 minutes) and passed through a 0.22 μ m filter to remove cells. Next, 2 mL aliquot of the filtered sample was mixed with phenol nitroprusside reagent (4mL) (0.25 M phenol in 100 mL of deionized water containing 23 μ M of sodium nitroprusside) and hypochlorite reagent (4mL) (0.05 M sodium hydroxide in 100 mL of deionized water containing 7.5 mL bleach (5% NaOCl)). Each reaction mixture was vortexed and then incubated at 60°C for 10 minutes. The amount of ammonium ion released because of urea hydrolysis was determined by referring to a previously prepared standard curve relating the absorbance at 630 nm to ammonium ion concentration (0-10 mg/L). Using this curve, one unit of urease activity (U) was defined as the amount of enzyme that would hydrolyze 1 μ mol of urea per minute. The release of 2 μ mol of ammonia is equivalent to the hydrolysis of 1 μ mol of urea (Mwandira, et al., 2017).

2.4.3. Syringe Solidification Test

A sand sample in a syringe with a volume of 35 mL was prepared for this test of nine different mechanisms. It was placed in 40 g of coral sands with three types of microorganism reagents (Table 1). The nutrient and calcium ions as bacteria food were injected into the sample test every day to accelerate the solidification process inside the syringe sample. After 14 days of curing,

petrographic thin-section and geochemical analysis were also conducted to analyze the cementation process inside the artificial beachrock.

First of this test, 100 mL ZoBell2216E medium solution was inoculated with 0.1 gram of the ureolytic bacterial isolated by the above test, then incubated at 30°C with gentle shaking at 80 rpm for 3 days. Next, 40 grams of coral sand collected from same area in Okinawa, Japan was dried at 110°C for 2 days and then placed in 35 mL syringe (diameter ϕ -2.5 cm). Subsequently, 16 mL of the bacterial culture and 20 mL of cementation solution (Table 2) were sequentially added to the syringe and drained, leaving 2 mL of solution above the surface of the cement. After curing, the cementation solution was added and drained at fixed interval. The Ca^{2+} concentration and pH of the drainage were also measured to determine temporal variations of these parameters in the samples. In addition, elemental measurement were made by X-ray fluorescence analysis (XRF) and mineralogy study using X-ray diffraction (XRD) (Multiflex 2kW, Rigaku Co., Tokyo, Japan) with $\text{Cu-K}\alpha$ radiation, with 2θ between 5 and 70°.

3. RESULTS

An understanding of the natural parameters and the interactions of beachrock requires an analysis of the genesis of all forms of beachrock. Various case studies were conducted based on three main samples; Sumuide beachrock sample, Okinawa, Japan; Sadranan beachrock sample, Yogyakarta, Indonesia; and artificial “human-made” beachrock. There is a lack of similarities in geology or geography but there are occurrences of outcrops of beachrock at 7° south latitude, where there is different climate, humidity, and source rocks compared to Indonesia which is mostly siliciclastic rocks. The beaches of Okinawa are at 26° north latitude. Invariably, there are advantages in

comparing samples from different latitudes. In the following sections, one will find comparisons between the Japanese and Indonesian beachrocks, whilst also comparing the characteristics of both natural and artificial rocks.

3. 1. Sumuide beachrock sample, Okinawa, Japan.

3.1.1. Geology of Okinawa, East China Sea.

The general geology of Ryukyu Island, Japan is summarized as a background to the discussion of the stratigraphy of the fossiliferous Tertiary and Quaternary rocks. The stratigraphic units are recognized as follows: the Shimajiri Formation, consisting of the Yonabaru clay member (late Miocene) in the lower part of the exposed section, and the Shinzato tuff member (Miocene or Pliocene) in the upper part; and the Ryukyu Group, consisting of, in ascending order, the possibly contemporaneous Nakoshi sand (Pliocene) and Chinen sand (Pliocene), the Naha limestone (Pliocene), the Yontan limestone (Pliocene), and the Machinato limestone (Pliocene) (McNeil, 1960).

In Okinawa there is Group Formation that working on this geology system in Okinawa. Shinjimari Group, Ryukyu Group, Uchihana Formation, and Holocene deposits such as alluvial deposits, beach deposits, low- tide elevation (coral reef) and also reclaimed land. These ages were from Neogene, Quaternary, and the youngest is Holocene product include beachrock deposits. The Ryukyu Island form one of the island arcs of the western Pacific, extending from southern Japan to the northern end of Formosa. The convex side of the island faces the Philippine Sea and the shallow East China Sea lies behind them. The island of Okinawa lies about midway in the Ryukyu Island. The Ryukyu Archipelago is 1046 km long and it consists of 55 islands and islets with a total land area of 3090 km². Beachrock has formed had an average dip of 3.5° to the sea. The

average altitude (above sea level) of the outcrops is 50 cm, with no evidence regarding the vertical position provided by a channeled coastline (Tokashiki and Aydan, 2010).

Sample of natural beachrock from Okinawa was taken in Sumuide coast area, whereas three differences of sample that assumed fresh rocks and the weathered deposit (Fig. 3). The megascopic scale of geology, Sumuide have a characteristics as uniform grain composition as material deposited. Solidified deposit and also non-solidified as beach sand deposit, rock fragments in Sumuide were dominance with shell and coral that formed relatives rounded with size 2 mm~2 cm grain sizes within carbonaceous cementation.

In a sample from Sumuide A, the grain size of the fragments is relatively large, > 3 mm to 3 cm, with a brown color; however, it is not a compacted because of the lack of cement. Samples of beachrock Sumuide B have similar characteristics to those of Sumuide A. The main differentiation is the granule which are of dime size but have more pores. Samples of Sumuide C appear more compact when compared to those of Sumuide A and B, possibly due to rare exposure to waves of ocean currents. Therefore, the solidified version was seen as the process of carbonated precipitation.

3.1.2. Petrographic Analysis Result

Microscopic analysis of thin-section samples from Sumuide, Okinawa was conducted. During microscopic examinations, it was clear that the grains mostly consisted of calcite, quartz, and fragments which known as micritic cement HMC $[(Ca, Mg) CO_3]$. In addition, the cement can be found as a matrix which is composed of very fine sedimentary particles (5–20 m) and micritic HMC. During the sedimentary process of the experiment the organic materials may have been affected. The most-impacted would have been grain sediments, as can be seen from the brownish

color on their external surface. The textures can be characterized as micritic with interclass (Folk, 1959). The results found occurrences of lytic fragments in the formed quartz (metasedimentary) with many matrixes in the form of shell fragments and quartz or coral opaque varying with size, where the fabric is grain-supported. Micritic or mud carbonate filling contacts at carbonate or non-carbonate fragments produced mud during deposition. General pore of Sumuide samples were filled with intra-granular microsparite as the cement, which although the evolution in the dry zone as Sumuide C compacted then the Sumuide A sample because the fragments, pore size, and lytic partially due had increased to the large amount of cement. The name of the rock sample was based on the carbonate classification based on rock sedimentary particles in the carbonate rock or limestone which is composed of ooids, termed, oolitic limestone. The most widely used classifications are those of Folk (1959) and Dunham (1962), regarding beachrock samples from Okinawa assumed to be namely *poorly sorted intracrine* (Folk, 1959) and *lithoclast grainstone* (Dunham, 1962) beachrock for samples Sumuide A and B, and for Sumuide C *poorly sorted intramicrite* (Folk, 1959) and *lithoclast packestone* (Dunham, 1962) beachrock (lithified). These differences in name classification are because of the different systems and cementation material inside beachrock from either the supratidal zone or intertidal zone.

Figure 4 explains the cementation process of peloidal micritic cement, cement needles of aragonite, micritized granules, and the cover of micritic seen in the Sumuide beachrock, which is a product of diagenesis in the phreatic environment of the sea. This cementation mineral as micritization gathered on other materials such as coral, foraminifera, skeletons, and others as solid rock. The exclusive presence of aragonitic cement (needle aragonite and micritic cement peloidal) in rocks of sedimentary diagenesis indicates that the environment was mainly marine. However, the presence of cement in beachrock peloidal micritic was very significant as it presented that the

micrite had played a vital role during the Sumuide beachrock sedimentary process. Whilst all of the aforementioned can be argued in terms of the origin of cement micritic peloidal, it remains uncertain. It can be suggested as precipitation occurring directly from seawater or it could just be a product of biological activity. The presence of microborings, however, may indicate that the micrites are also a product of biological activity. There is no direct evidence of the influence of meteoric water on beachrock sedimentation. Some have suggested that the process of diagenesis occurred much faster than meteoric; however, this is open to debate as both were subjected to weathering conditions during the sedimentary process.

From the data collated based on cement and material cemented inside the rock samples, it is accurate to suggest the material cemented formed from foraminifera, skeletons, pores, quartz, and other material and that the cement is micrite and sparite. Table 3 shows the compositional percentage of fragmented material based on petrographic measurements. The Sumuide, Okinawa samples are dominated by foraminifera, skeletons, and quartz in terms of material fragments of rocks.

3. 2. Sadranan beachrock sample, Yogyakarta, Indonesia.

3.2.1. Geology of Gunungkidul Regency, Yogyakarta.

Beaches of Gunungkidul Regency, Province of Special Region Yogyakarta (Prov. DIY), Indonesia have economic potential as limestone mine deposits and tourism. The karst beaches in Gunungkidul Regency, Sadranan Beach to be exact, have similarities in terms of geological conditions, methods, and geomorphologic processes beach formation. The research site is at coordinates 07°08'42,55" and 110°26'11,31". However, for each of its shores, it is

characteristically a coastal environment. The thin line of the characteristics of the environment are based purely on the shape of the beach and the diameter of the grains of sediment. This difference should also be noted as related to the condition of waves, ocean currents, temperature, salinity, and pH at each beach.

The characteristics of the Karst Mountains of South Beach indicate that there is steep topography that rises to the east of Java Island. Continuing eastward hummocky valleys occur (Drini Beach to Krakal to Sadranan). The coast of Sadranan have a slope were steep $\sim 12,33^\circ$ with the tidal range of 12 meter that makes the sedimentary process in this coast were thickness than the deeper slope. The tidal reach of the littoral zone provides an opportunity for strong sea wave energies to erode the beach. The energy comes from crashing ocean waves that break on the beach in the breaker zone that is not too far from the coastline. The result of the wave energy as well as the sharp, heterogenous rocks is the formation of an irregularly shaped beach at Sadranan.

Throughout the karst coast of the DIY Prov., Gunungkidul Regency the soil is derived from limestone bedrock, similar to the Mediterranean, i.e., the level of physical weathering is high. Grains of sediment along Krakal and Sadranan beaches are largely bright colored along most of the coastline as a result of the erosion of seabed being deposited on the beach. Composition of this sediment mainly formed by coral, skeletal fragment, Foraminifera and Mollusca.

It is evident that the megascopic Sadranan beachrock has the same grain characteristics and the same grain composition as the non-solidified material along its beaches, with a dominance of shell fragments, corals, Foraminifera, and relatively anhedral-shaped grains varying from 2 mm to 4 cm. There is also carbonate cement. Figure 5 shows a sample of Sadranan beachrock that has fragments of relative large size (> 3 mm to 3 cm) with a grey to brownish grey color.

3.2.2. Petrographic Analysis Result

The presence of different quartz crystals created an abundance of lytic fragments and more coral or shell-like fragments. The micrite or mud carbonate cement were filled the fragments showed that the cement formed during deposition, such as quartz, coral, and another fragmented material. Whereas some of the opaque and quartz were formed as supported fabric (Fig. 6).

Based on the analysis, the cementation mineral came from aragonite within needle crystalline fibrous, dark color micritized granules and cover a cryptocrystalline surrounded the fragments. This represented the Sadranan beachrock is a product of diagenesis in the phreatic environment of the sea sedimentation. The exclusive presence of aragonitic cement (needle aragonite and peloidal micrite cement) in rocks of the sedimentary diagenesis indicated that the coast occurred mainly within marine environments. The presence of cement found in the peloidal beachrock micrite cement is very significant, as it is a rare form of cement that can be found in rocks only along a coast, and therefore is very valuable. Research presenting some samples of cement showed gaps within the pores of the rocks, or an internal sedimentary intraparticle pore. The cement is comprised grains of round micritic and can be clearly observed in thin section which indicates that micrite played an important role in Sadranan beachrock cementation (see table 3).

In conclusion, it can be generalized that the Sadranan beachrock occurs mainly in sea conditions with different cements of the Sumuide sample, while the Sadranan sample is enriched in coral pores and pore-filling cement dominated by HMC. The names of the samples from Sadranan, Yogyakarta, are based on carbonate classification and are termed *poorly sorted biomicrite* (Folk, 1959) and *bioclastic pack stone* (Dunham, 1962) beachrock (lithified). In Sadranan, microorganism activities were the main source of material cemented inside the rock compared to

that of the Okinawa beachrock that formed from geochemical dissolution and reorganization followed by micritization of living organisms from the area.

3.3. Artificial beachrock sample

3.3.1. Isolation of Ureolytic Bacteria

The results of isolated surface sand within assumption microbial enrichment then selected the ureolytic bacteria species only from Okinawa, Japan revealed that *Pararodhobacter* sp., that increased pH solution to the highest value for calcium carbonate beachrock. On other hand, in Japan also found beachrock deposits occurrences of silica mineral which is Miyako-jima, Japan revealed *Oceanisphaera* sp. The *Pararodhobacter* sp., are Gram-negative, rod-shaped, aerobic, chemoorganotrophic bacteria, that are moderately halophilic with approximately 1 µm in diameter and 3 µm in length (Foesel, et al., 2011; Danjo and Kawasaki, 2016). Meanwhile, *Oceanisphaera* sp., had a spherical cell, 1.0-1.2 µm in diameter and motile by means of flagella. Gram-negative chemoorganotroph with absolute requirement for sodium ions, aerobic, moderately halophilic, and oxidase-catalase-positive (Romanenko, et al., 2003).

3.3.2. Microbial Population Count and Urease Activity Test

The time course of cell growth and urease activity by *Pararodhobacter* sp., and *Oceanisphaera* sp., for 15 days are shown in figure 7. Cell concentration increased up to 2 days and then reached stationary phase. The urease activity of the cell culture increased with cell growth. Based on this result, the production of urease would be a growth-associated system. Considering microbial-based sand solidification, which needs relatively long duration for adequate solidification, the long-term

stability of the enzyme would be advantageous in the MICP process (Danjo and Kawasaki, 2016; Fujita, et al., 2017).

3.3.3. Temperature and pH variation effects on urease activity

In the MICP biocementation technique, urease activity of enzyme activity is the key of role for bioindicator of artificial beachrock process success or not. The difference of temperature environment is shown in figure 8 for both species. High activity was obtained in the range temperatures of 30-70 °C, within the maximum temperature of urase activity was obtained 70°C in this study. Most of urease molecule is known to be composed of protein subunits and the subunits of this urease would strongly bind each other to leads the high stability of urease during long-term cultivation (Mobley and Island, 1995; Balasubramanian and Ponnuraj, 2010; Fujita, et al., 2017).

Urease activity under different pH is shown in figure 9. The results showed with the maximum activity around pH 7.0 – 8.0. For this temperature and pH variations, the results were different with Fujita, et al. (2017), where he is mentioned the results of maximum temperature exponential increasing toward environment temperature of the buffer solution and the method for urease activity measurement he used ammonia meter of Tris/HCl buffer solution. Meanwhile, in this research, for measuring urease activity we used EDTA buffer solution for indophenol blue method then detect with colorimeter changing with optical density 630 nm. The results of Fujita, et al. (2017) and this research quite different but the pattern of bacterial growth and urease activity, especially for *Pararodhobacter* sp., is almost same.

3.3.4. Solidification Test

Complementary to these results, in order to produce artificial beachrock, a biomineralization technique had to be used (Dhami et al., 2013). Bacteria found in urease activity can contribute to calcium carbonate precipitation. The precipitation of carbonate vs. the most straightforward and most easily controlled mechanism of MICP has the potential to produce high amounts of carbonates over short periods of time. These products are known to equilibrate in water to form bicarbonate, from mixes of chemical compounds, whereas 1 mol of ammonium and hydroxide ions result in an increase in pH. Bacterial surfaces also play an important role in calcium precipitation and biomineralization processes inside rock. Because of the presence of several negatively charged groups, at a neutral pH, positively charged metal ions can be bound on bacterial surfaces, favoring heterogeneous nucleation (Douglas and Beveridge, 1998; Bauerlein, 2003). Commonly, carbonate precipitation develops on external surfaces found in bacterial cells by successive stratification, and bacteria can be embedded in growing carbonate crystals (Saitis, 2016).

3.3.5. Petrographic Analysis Result

The size of grains was very important, especially from clay to sand (0.04–3 mm) and showed poor sorting. The shape of the grains found was angular and rounded/sub-rounded, suggesting fragments were damaged. There is a matrix formed by micro-quartz (lytic aggregate fractions) and there is an indication of growth of cement (sparite) in a blade shape, slightly modified by fossil contact, floating, and with varied fragment sizes (some of the cement formed inside coral or foraminifera as fibrous aragonite crystals). There can be a visible emergence of cement found on

samples of the artificial beachrocks, which is the presence of aragonite needles peloidal micritic cement (orange) with a dominance of cement aragonite needles that fill the intergranular or intragranular spaces. These peculiar substances were not only attached to grains of organic matter such as shell fragments, coral, or foraminifera, but on monocrystalline quartz granules. In application, it is suggested that artificial beachrock most likely can also cement as an adhesive in inorganic granules (Fig. 10). Fragmented material from thin-section analysis is dominated by (see Table 3) foraminifera, skeletons, and coral because the solidification test material is formed from coral sand within a bacterial reagent.

3.4. Geochemical Analysis

XRD and XRF were conducted on the beachrock samples, both natural and artificial. The results showed the mineral composition inside the rock samples, including aragonite, calcite, strontium, manganese, lime, phosphate, alumina, and other minerals.

3.4.1. Natural Beachrock

Based on the results of trace elements, two types of minerals of cement composition were selected. Therefore, more than 90% of the calcium detected inside the beachrock was within aragonite and calcite, >2 % of the manganese, and silicon was found only in several samples with an amount greater than 1% of the natural beachrock result (Fig. 11).

Equally important, because the XRF results represented the major element composition inside the rock samples, aragonite and calcite minerals were detected as one component of the calcium element. Calcite minerals that were found could be divided into HMC and LMC and calcium carbonate (CaCO_3). Results of XRF analysis are listed Table 4.

3.4.2. Artificial Beachrock

Meanwhile in the artificial beachrock, general mineral composition included calcium carbonate within aragonite and calcite (Fig. 12).

For XRF analysis on the artificial beachrock, the number of the values in the XRF results were not as significant because of the microcrystals found inside the syringe samples, even though it was a very small amount. Thus, we tried to compare to the original coral sand sample, before and after the solidification test. The differences were ± 1 ppm between the origin coral sand and the samples after the solidification test. The results of XRF analysis for artificial beachrock also listed Table 4.

4. DISCUSSION

Whilst the key aspect of mineral cementation is discussed, it is equally important to recognize the presence of aragonite primarily formed in a phreatic, low-marine, vadose environment. Aragonite formation has been related to the temperature of the solution; for example, the higher the solution temperature, the faster aragonite precipitates around calcite cement minerals (Burton and Walter, 1987) and the crystal form the cement takes is mostly fibrous. After this, the interplay between the different carbonate phases occurs in the cement, and the content of calcite, aragonite or Mg-calcite depends on the change in the water-table elevation, the temperature or the pressure, with a trend toward chemical stability. One of the most effective means to create a polymorph of calcite or dolomite is the infiltration of meteoric water rich in dissolved CO_2 , depleting the cement in Mg, Sr, and Na and enriching it in other elements.

Thin-section analysis from all of the Okinawa samples present results in one direction; fossils are dominated by foraminifera and coral (skeletal grain particles), while other materials depend on the genesis of the source rock (opaque) that cemented from micritic and sparite.

According to the theory of fossil marine paleontology, foraminifera are separated into two groups following their life strategy, namely planktonic and benthonic foraminifera. All benthic foraminifera occur in marine and neritic environmental zones, living largely in oligotrophic reefs and carbonate shoals and confined mainly to low-latitude areas. The prolific in nutrient-deficient of the nature, warm, shallow-water seas, often associated with coral reefs, an important key in producing carbonate sediment (Boudagher-Fadel, 2008). In general, the presence of benthic foraminifera (including larger foraminifera) indicates a warm environment, as the minimum water temperature tolerated by a living foram is 18°C, while the maximum water depth at which they live is 35 m (Murray, 1973) as related to the photic zone.

Peloidal micrite cement, aragonite needled cementation, micritization, and micritic were covered on the thin sections. The analysis concluded that natural beachrock was a product of a diagenetic marine phreatic environment. The significant cost of aragonite cement (aragonite needled and micrite peloidal cement) for cementation of beachrock was a key factor in shoreline sedimentary diagenesis in the marine environmental system. It can be directly precipitated from sea water or be a by-product of biological activities.

Beachrock is generally cemented in the tidal zone with high amounts of aragonite, HMC, and LMC. General carbonate cementation in the marine environment formed acicular-radial and spacious types of cement texture whereas the tidal zone is indicated by aragonite or HMC. Meniscus and bridge cement indicate LMC composition (Scholes and Ulmer-Scholes, 2003; Voudoukas et al., 2007; Mauz, et al., 2015; Avcioglu, et al., 2016). Regarding the aforementioned cementation processed related to microbial activity, it is commonly referred to as biogenic cement. On the other hand, another author has suggested that the microbes of metabolic bacteria are related to the precipitation of rocks (Danjo and Kawasaki, 2016; Khan, et al, 2018; Tirkolaei, et al., 2018),

and the end products lower the thermodynamic boundary for cement nucleation and increase the rates of the physicochemical processes involved (Kandianis, et al., 2008; Dupraz, et al., 2009). Bio-cementation using MICP technique of calcium carbonate precipitation can occur by biologically controlled or induced (Lowenstan and Wiener, 1988). Hydrolysis of urease bacteria (*Pararodhobacter* sp., and *Oceanisphaea* sp.) as a result of increased total carbonate content through the production of dissolved inorganic carbon and a pH increase through the production ammonia. Bacteria play a catalyzing role in urea hydrolysis through generation of urease enzyme to form calcium carbonate cement (van Passen, 2009; Tirkolaei, et al., 2018; Al Thawadi, 2008). Specific cement types are recognized as (i) acicular, fibrous, and bladed fabrics; (ii) radial fibrous and fascicular-optic fabrics; (iii) micro-and cryptocrystalline fabrics; (iv) equant spar, syntaxial overgrowth fabrics, scalenohedral calcites, peloidal fabrics and (v) non-carbonates. These types are incipient early marine cementation leading to a reduction in pore-space in near-seafloor sediments (Christ, et al., 2015).

Geochemical analysis such as XRD and XRF were conducted to obtain a valid analysis. XRD and XRF had significant results and graphically show the distribution of minerals and elements inside the sample of natural beachrock. Aragonite and calcite that cannot be analyzed under a microscope can be detected using geochemical analysis. Samples in the intertidal zone have a greater amount of aragonite minerals, but samples in the supratidal zone (dried zone-C) are dominated by calcite minerals rather than aragonite. This is because aragonite fibrous crystals form when the deposits are drowned in salt water and then precipitate as a calcium-carbonate compound.

Based on the results, analysis using petrographic and geochemical approaches of both samples from Okinawa, Japan and Sadranan, Indonesia have the same pattern which is the characteristics of cement solution were dominated with aragonite and calcite crystalline that

soluble in the water. However, the mechanism of cement sedimentary not exactly the same because of the geomorphologic settlement Japan in Indonesia were different and source of mineral in Indonesia were close to volcanic activities that really active, especially Yogyakarta mountain activity which is famous of eruption every 4 years. Therefore, the temperature and pH in the tropic-subtropical area is the main role the interaction between microbial activity and chemical reaction proceed toward cementation of a beachrock. Cement product of beachrock evolution based on field investigation (area A, area B, and area C) is represented of aragonite crystalline only occur in the swash-zone area (mostly area A) because the aragonite mineral is really easy to soluble in the fresh water that came from rain or fresh water from land. This assumption does not only come from Japan or Indonesia studies but perhaps the cementation process is relatively same all around the world especially in the tropic-subtropic climate.

Furthermore, artificial beachrocks used microorganism activities significantly successfully and were noted as man-made. The aragonite crystallization fibers formed between grain sand particles then interlocked with one another until the precipitate over time became a solid rock. The differences between natural beachrock and artificial “synthetic” beachrock are shown in table 5. The solidification experiment by using in-situ bacteria having urease activity and changing the composition of the solidification solution (Danjo and Kawasaki, 2014). Hardening of a substrate using calcium carbonate as cement materials, using calcite and aragonite were valuable to consider how to make artificial beachrock. (Hata, et al., 2011; van Passen, et al., 2010; Danjo and Kawasaki, 2014; Khan, et al., 2018). The comparison results were sufficiently strong to understand the mechanisms that proceed during the creation of artificial beachrock. Danjo and Kawasaki, 2016, reported UCS test of artificial solidification beachrock using local ureolytic bacteria from sand around the beachrock in Okinawa, Japan. The column specimen was cemented up to 10 MPa UCS

after 28 days under a curing temperature 30°C, injection interval of cementation solution of 1 day and 0.3 M Ca²⁺ and 0 M sodium malate in cementation solution. Therefore, based on experiments of synthetic beachrock, it could mitigate erosion impacts in coastal areas subjected to problematic erosional damage. Bio-cementation technology is promising technology due to its suitability in an environment problem such as coastal erosional preventing development in the future

5. CONCLUSIONS

To review each rock petrography analysis of both natural beachrock (Sumuide and beach Sadranan Beach) as well as a sample of the results was obtained by the presence of artificial beachrock cement filling intraparticle nor interparticle granules. On an average, the sample of natural beachrocks were very similar to Okinawa beachrock and Yogyakarta beachrock where this type of cement dominated by peloidal micritic and micritic dark granules that found in the same intertidal zone. Okinawa beachrock contained more lytic granules with fragments of quartz result material sediments there are predominantly with a fragment of coral shell, meanwhile Yogyakarta beachrock was more sparite cement which had filling of pore interparticle, so that it appears more compact and lower porosity. Micritization proceed in Okinawa beachrock or Yogyakarta is a product of diagenesis rock that formed on the phreatic environment of the sea within the presence of cement on the beachrock is peloidal micritic. Therefore, the origin of cement micritic peloidal is uncertain. It can be precipitated directly from seawater or it just be a product of biological activity.

Samples of artificial beachrocks had yield beyond allegations in which the presence of cement peloidal micrite and micritic. Nevertheless, the dominant presence of fibrous aragonite where an

organic granular inorganic (monocrystalline quartz) so that the cement is not only the inherent and formed on grained containing Ca^{2+} but also allows granular inorganic fragments on the other. These methods are quite successful based on accelerated precipitation of carbonate within microbial reagent, although high urease activity bacteria are needed for solidification test for temporal time measurement. The results of natural beachrock and artificial beachrock mechanism process using petrographic method were quite successful. Aragonite and calcite as fibrous micritization formed as cement material between sand particles onto solid rocks. In the future, we plan to investigate another physical property of natural beachrock for better manufacture artificial include in the extreme conditions.

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703

705 **List of figures:**

706 Figure 1. (A) Location map of Okinawa in Japan, (B) Research area in Sumuide, Okinawa
707 Island, (C) Location map of Indonesia, and (D) Locations in Sadranan area.

708 Figure 2. Workflow of research themes.

709 Figure 3. Sumuide beachrock sampling area, Okinawa, Japan.

710 Figure 4. Sumuide beachrock cement image under 100x (yellow pattern is grain matrix and
711 orange pattern is cement).

712 Figure 5. Sadranan beachrock sampling area, Yogyakarta, Indonesia.

713 Figure 6. Sadranan beachrock cement image under 100x (yellow pattern is grain matrix and
714 orange pattern is cement).

715 Figure 7. Cell growth and urase activity of *Pararodhobacter* sp. (a), and *Oceanisphaera* sp.
716 (b).

717 Figure 8. Effect of reaction temperature on urease activity of the whole cell at pH 7 (1 mM
718 EDTA-disodium salt solution). Experiment were conducted in triplicate: data represented
719 the average of three experiments with error bars indicating the standard deviation.

720 Figure 9. Effect of reaction pH on urease activity of the whole cell at 30°C 1 mM EDTA-
721 disodium salt solution). Experiment were conducted in triplicate: data represented the
722 average of three experiments with error bars indicating the standard deviation.

723 Figure 10. Artificial beachrock cement image under 100x (yellow pattern is grain matrix and
724 orange pattern are cement).

725 Figure 11. Graphic XRD analysis pattern using natural beachrock sample, Okinawa, Japan
726 (left) and Sadranan sample, Yogyakarta, Indonesia (right). Within abbreviation C: Calcite;

727 A: Aragonite; Sr: Strontium; Q: Quartz; Mg: Magnesium; Fe: Iron; K: Potassium; Pb:
728 Lead; CaO: Calcium Oxide; Si: Silica; and Al: Alumina.

729 Figure 12. Figure of graphic XRD analysis pattern using artificial beachrock. Within
730 abbreviation C: Calcite; and A: Aragonite.

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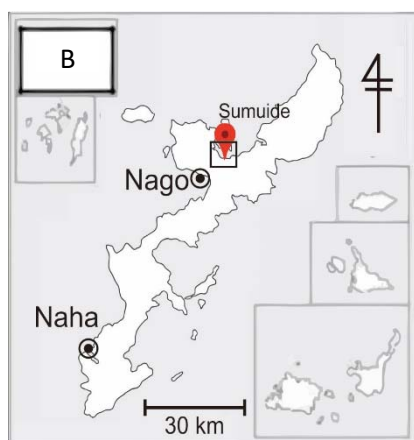
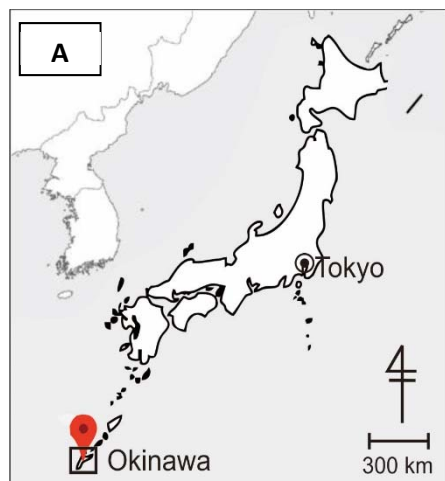


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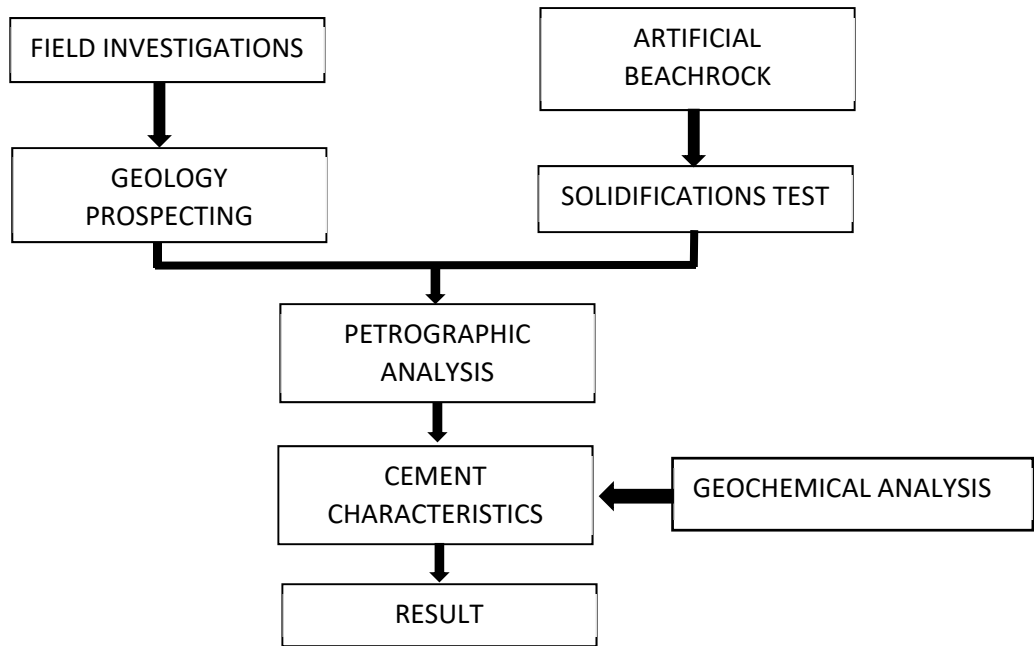
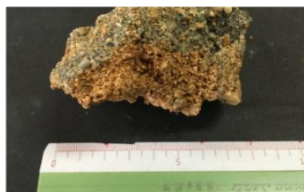


Figure 2. Workflow of research themes.



Sumuide A



Sumuide B



Sumuide C

Figure 3. Sumuide beachrock sampling area, Okinawa, Japan.

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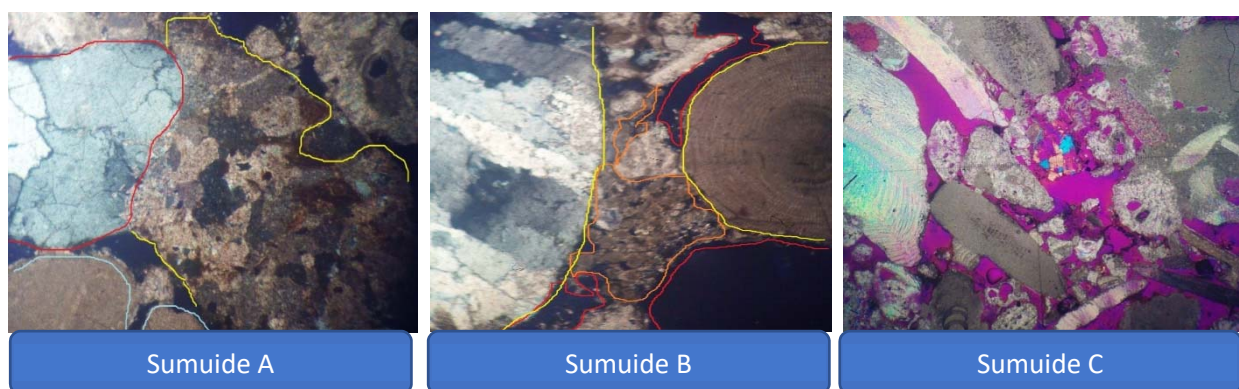
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820 Figure 4. Sumuide beachrock cement vision using 100x (yellow pattern represented grain
821 matrix and orange pattern represented cement).

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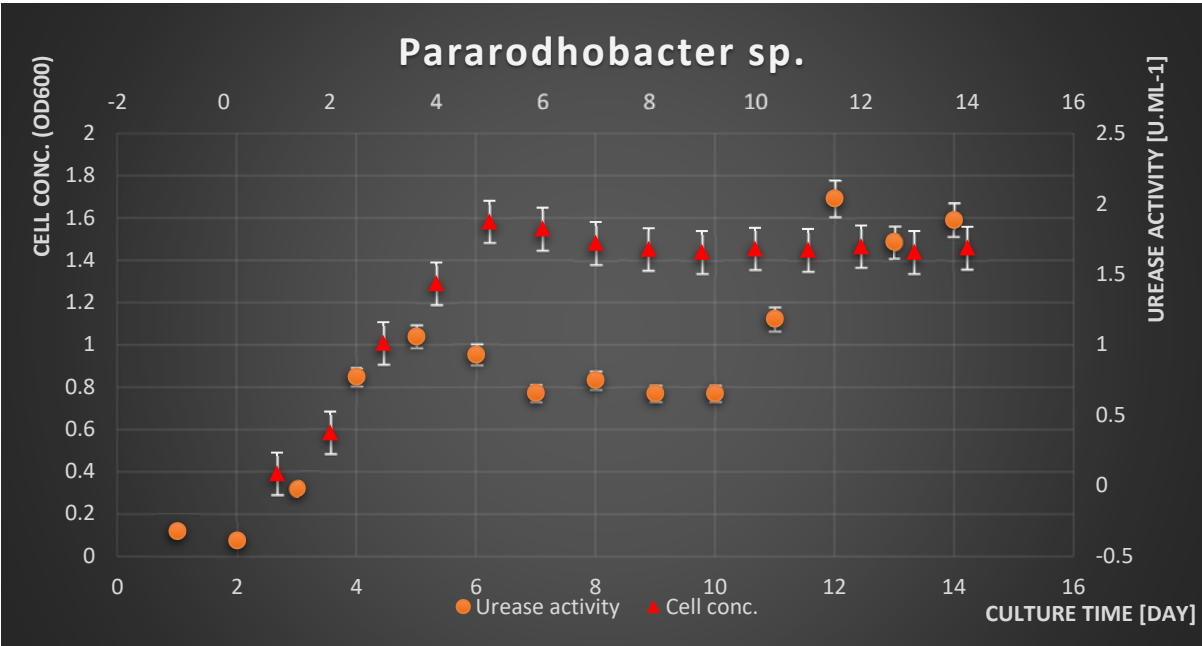
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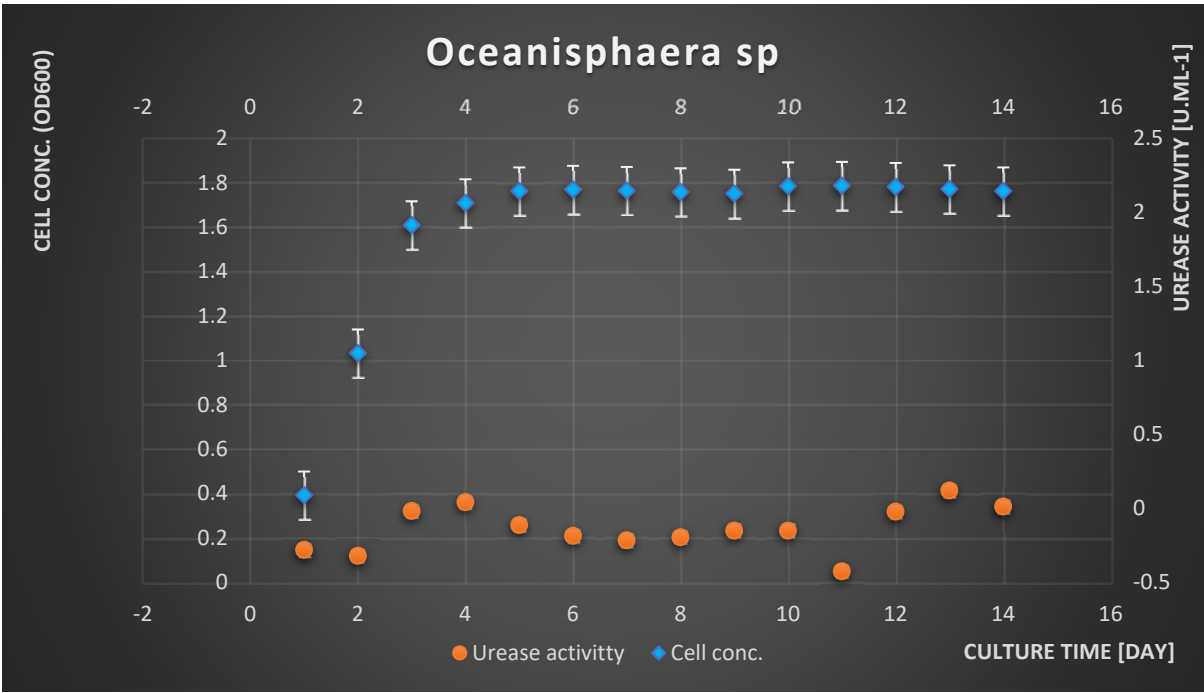
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Figure 6. Sadranan beachrock cement vision using 100x (yellow pattern represented grain matrix and orange pattern represented cement).



(a)



(b)

Figure 7. Cell growth and urase activity of *Pararodhobacter* sp. (a),
and *Oceanisphaera* sp. (b).

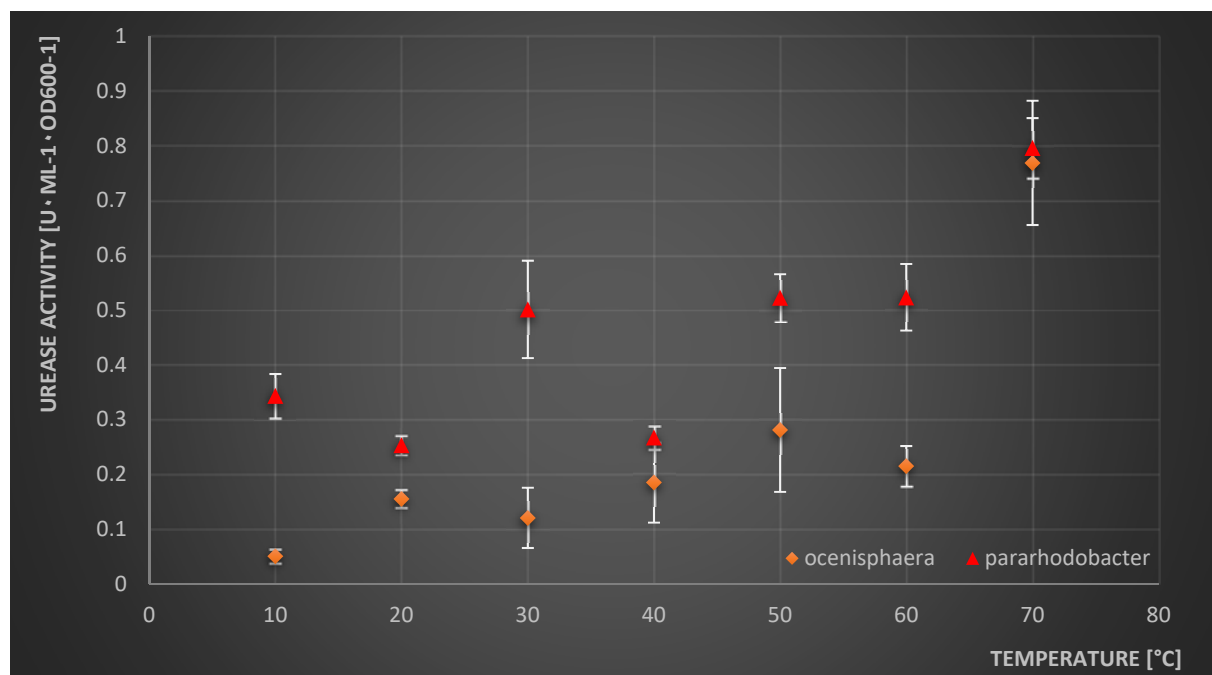


Figure 8. Effect of reaction temperature on urease activity of the whole cell at pH 7.

Experiment were conducted in triplicate: data represented the average of three experiments with error bars indicating the standard deviation.

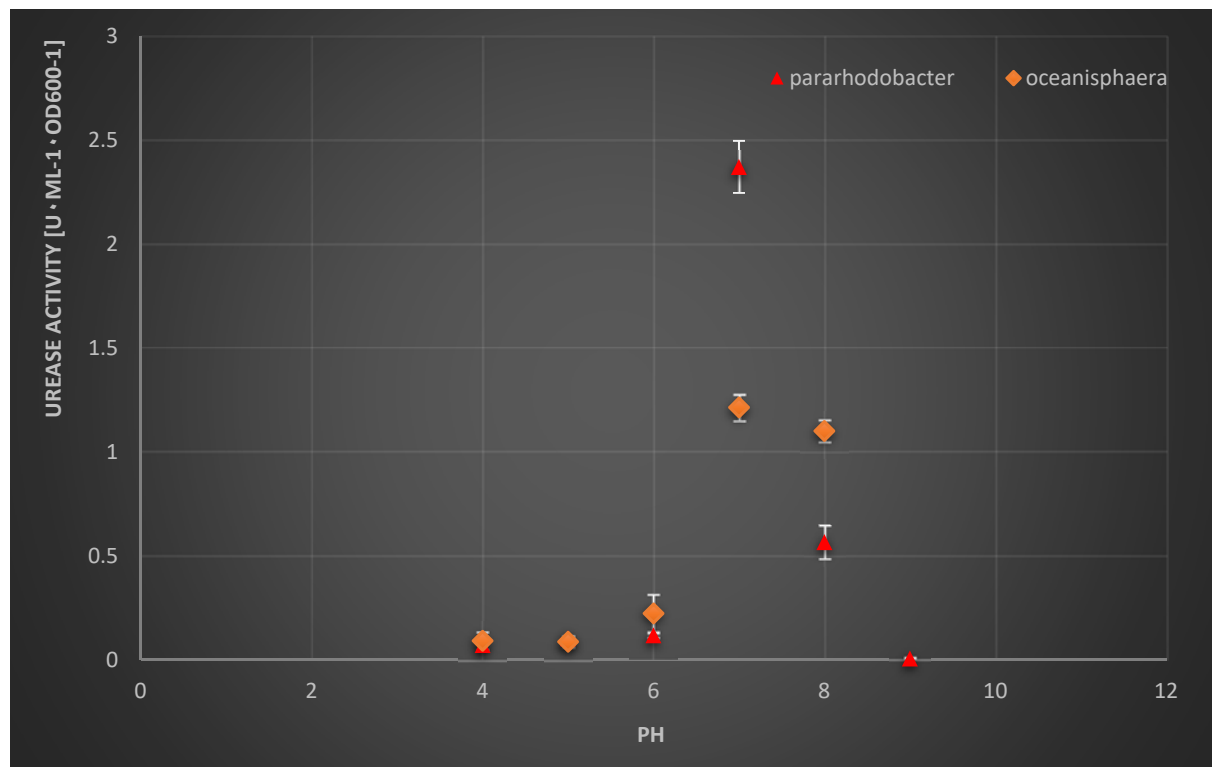


Figure 9. Effect of reaction pH on urease activity of the whole cell at 30°C. Experiment were conducted in triplicate: data represented the average of three experiments with error bars indicating the standard deviation.

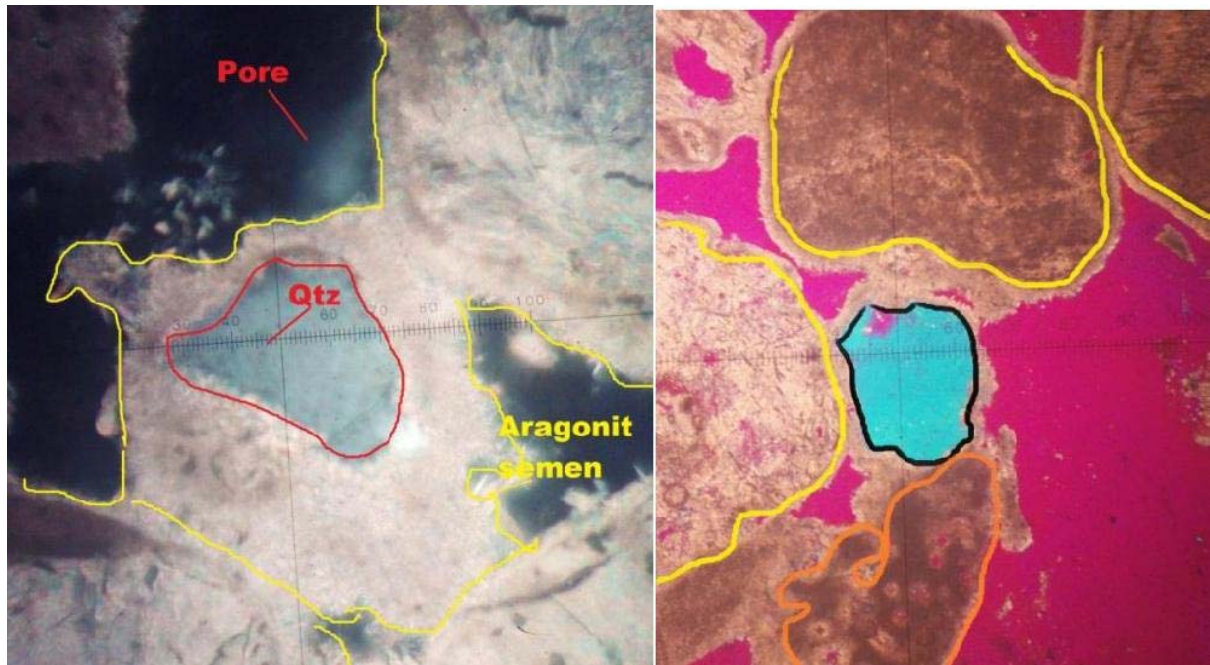


Figure 10. Artificial beachrock cement vision using 100x (yellow Pattern represented grain matrix and orange pattern represented cement).

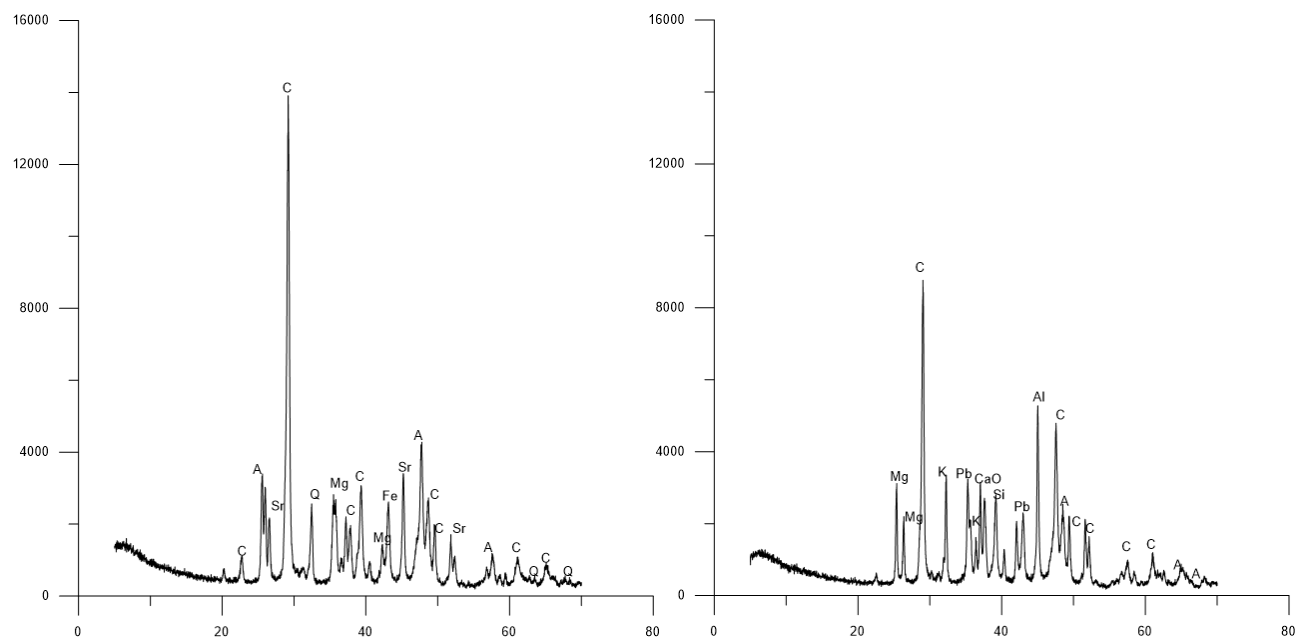


Figure 11. Figure of graphic XRD analysis pattern of natural beachrock sample, Okinawa, Japan (Left) and Sadranan sample, Yogyakarta, Indonesia (Right). Within abbreviation C: Calcite; A: Aragonite; Sr: Strontium; Q: Quartz; Mg: Magnesium; Fe: Iron; K: Potassium; Pb: Lead; CaO: Calcium Oxide; Si: Silica; and Al: Alumina.

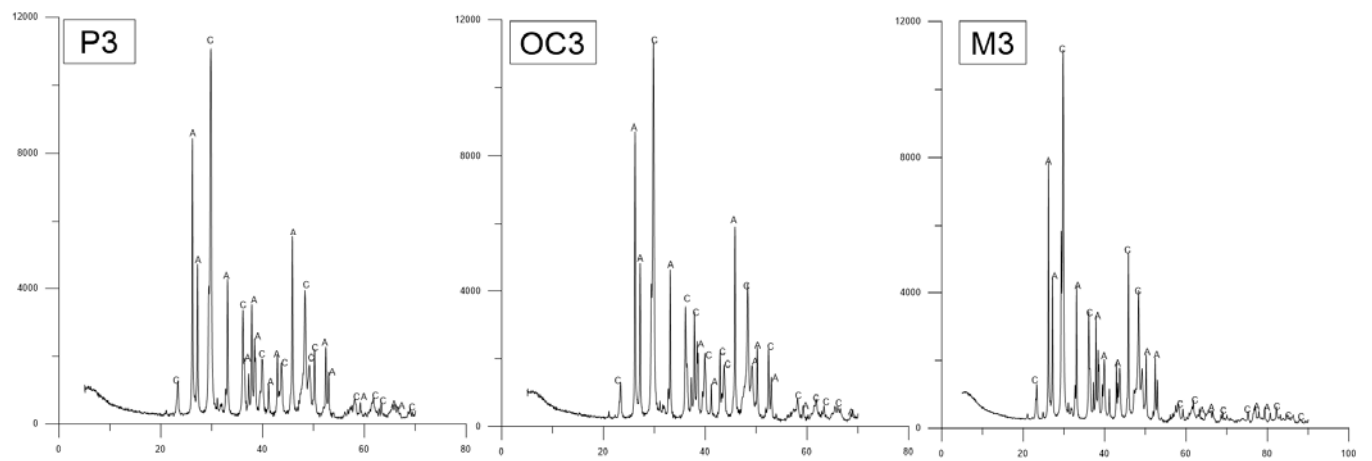


Figure 12. Figure of graphic XRD analysis pattern using artificial beachrock. Within abbreviation C: Calcite; and A: Aragonite.