Determination and Radiocarbon Dating of Marine Mollusc Fossils in Ancient Sea Shelf of Central Java Indonesia

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Abstract. Mollusc phylum is one of the most adaptive animal groups on Earth. They occupy and thrive in incredibly diverse habitats. Their distribution in the fossil record is equally diverse. Indonesia is one of the country with marine mollusc fossil variety, such as in the archaeological site of Sangiran, Patiayam (Ancient Muria Strait) and Grobogan – Central Java. Radiocarbon Dating is the method for dating analysis using ^{14}C. By measuring ^{14}C content, we can estimate how long ago the fossils died. Radiocarbon dating is an extremely useful technique for determining the ages of geological materials (that have some organic-derived carbon in them). Field sampling had found variety of marine mollusc fossils such us 1) Sangiran: *Dosinia* sp., *Telescopium telescopium, Anadara* sp., and *Dosinia insularum*; 2) Patiayam (Ancient Muria Strait): *Pseudodon vondenbuschianus, Elongaria orientalis, Conus (Pionoconus)* sp., *Ampullina bandongensis, Anadara pilula* and *Filopaludina javanica*; 3) Grobogan: *Antigona chemnitzii, Cultelus dilatatus, Plotia scabra* and *Tibia modesta*. Mollusc fossils sample was analysed using Accelerator Mass Spectrometer (AMS) radiocarbon system. It is a good method for dating specific samples. The result showed that Sangiran as an ancient deepsea has fossils age 38710 – 31947 years, then Patiayam (Ancient Muria Strait) with fossils age 26248 – 11994. Mollusc fossils from Grobogan was the youngest area has fossils age 25692 – 6479 years.

Keywords: Radiocarbon Dating, Mollusc Fossils, Patiayam, Grobogan, Sangiran

1. Introduction

The interrelationships and evolutionary history of molluscs have seen great advances in the last decade [1]. Indonesia is one of the country with marine mollusc fossil variety, such as in the Sangiran, Patiayam (Ancient Muria Strait) and Grobogan – Central Java. Long time periode, Indonesian archipelago had been formed through tectonic movement, volcanic eruption process, earthquake, sedimentation and erosion process simultaneously. Similar phenomenon had happened in ancient Muria Mountain with deformation phases of ocean go through with land had result sedimentation process in Demak, Jepara and Pati (the part of Central Java) to become ancient Muria Strait. Long time before Muria region had

separated from Java Island. Existence of land erosion and sedimentation process from Java Island by means of river trivialisation which stream down to Muria Strait. By the time then the strait closed and become land until now.

Muria regional stratigraphy indicates that the upstream Pontang River, Muria – Kudus, approximately 50 m to the west. In this area are found rocks containing carbonate and rich in marine mollusc fossils (deposition of marine origin) [2]. Similarly, archaeological site of Sangiran and Grobogan, allegedly the area was formerly ocean which are then degraded into a land, because of the evidence of the ancient life, especially the discovery of marine fossils. Sangiran is one of the most important ancient sites in the world. Sangiran saves a lot of evidence of ancient life and formation lithology. Sangiran become data sources of archeology, geology and paleontology [3].

Understanding the ancient environment in relation to ancient climate could help us to study the evolution of fauna (marine organism) in relation to environmental changes and predict the result of changes in the distribution of fauna today [4], [5] and also it is hoped as a new marine palaentological paradigm can be used to develop the past marine ecosystem or a new site for paleo-oceanographic and marine-paleometeorological studies [6]. The area were axamined using mollusc fossils object with the aim of identifying mollusc fossils and to examine the age of mollusc fossils with radiocarbon dating method. The research was conducted in two stages; first is field study at ancient Muria Strait, ancient reef Grobogan and ancient deepsea Sangiran (Central Java) to determine the ancient coastline coordinate, sampling marine fossils and observation of sedimentary stratigraphy. Second is laboratory for marine mollusc sample analysis and radiocarbon dating.

2. Material and Methods

2.1. Sampling and Identification Mollusc Fossils

Mollusc fossils sample collected from Sangiran, Patiayam (Ancient Muria Strait), and Grobogan (Figure 1). Sampling was used purposive sampling technique based on coordination and discussion with museum staff. Mollusc fossils would have found then identified in determine genera of mollusc fossils. Identify according to marfometric identification [7].

2.1. Radiocarbon Dating Method

Radiocarbon Dating is the method for dating analysis using ^{14}C. In order to evaluate the validity of a ^{14}C age of any material, the original sources of the carbon must be known. The carbon in gastropod shell carbonate originates from as many as four different sources: atmospheric CO_2, food, water, and carbonate rocks [8]. Analysis of the age of mollusc fossils with radiocarbon dating method could estimate the ages any organic thing. ^{14}C is an unstable radioactive isotope.

Radiocarbon dating analysis using Accelerator Mass Spectrometer (AMS) system. AMS detect atoms of specific elements according to their atomic weights. They, however, do not have the sensitivity to distinguish atomic isobars (atoms of different elements that have the same atomic weight, such as in the case of carbon 14 and nitrogen 14 – the most common isotope of nitrogen). There are essentially two parts in the process of radiocarbon dating through AMS. The first part involves accelerating the ions to extraordinarily high kinetic energies, and the subsequent step involves mass analysis. There are two accelerator systems commonly used for radiocarbon dating through AMS. One is the cyclotron, and the other is a tandem electrostatic accelerator [9], [10]. This research is supported through Micro Analysis Laboratory, Tandem accelerator (MALT), The University Museum, The University of Tokyo, Japan. The protocol of the radiocarbon dating procedure from marine mollusc fossils by Matsuzaki (2015) [11].

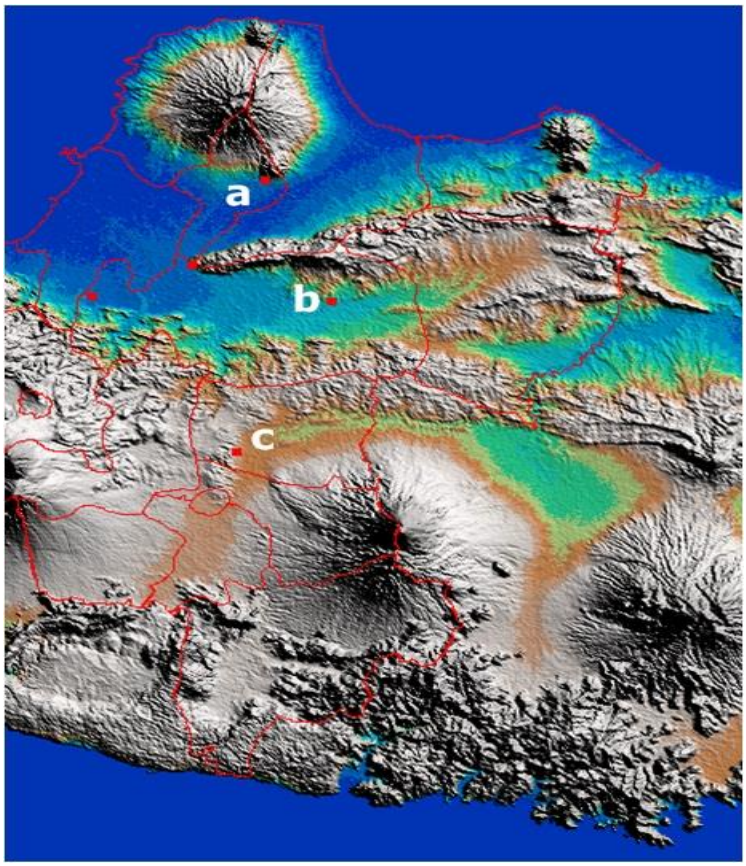

Figure 1. Sampling Site for the Collection of Mollusc Fossils from Patiayam (Ancient Muria Strait) (a), Grobogan (b) and Sangiran (c) [6]

The samples were preparation less than 10 mg and treated twice with 1.2 M HCl for 6 hr at 80°C to remove any possible carbonate contaminants. Next, the samples were treated with 0.005 M – 1.2 M Sodium hydroxide at 80ºC. Then samples were washed by ultra pure water and treated again with 1.2 M HCl for 2 hr at 80 °C. To remove HCl component completely from those small samples, centrifuge and decant were repeated after adding some distilled water.

The samples were then dried in an electric oven at 90 °C. For the small sample, we added a few hundred-milligrams of CuO powder (Filamentous Copper Oxide II) as a carrier to the sample in the centrifuge tube, to collect carbonaceous materials completely. The total amount of pretreated charred samples was used to produce CO_2. The samples were placed in Vycor tubes of about 9 mm in outer diameter, with about 1 g of granular CuO. Then the tubes were connected to a vacuum line, evacuated completely, and sealed to a tube length of 300 mm. The Vycor tubes were heated to 850°C for 2 hr to completely convert carbon to CO_2. The CO_2 produced was purified cryogenically in a vacuum line and reduce to graphite on about 2 mg Fe powder in the presence of hydrogen. The graphite materials were pressed into aluminum target holders for AMS ^{14}C dating. We used the Hox-II standard as a ^{14}C-concentration reference [11]. Then, the ^{14}C ages were calibrated to a OxCal [12].

3. Result and Discussion

3.1. The Age of Marine Mollusc Fossils with Radiocarbon Dating

Analysis of marine mollusc fossils ages using Accelerator Mass Spectrometry (AMS) radiocarbon with four steps: sampel preparation, chemical pretreatment, combustion (oxidation) and sample processing

with AMS. The result of marine mollusc fossils ^{14}C ages has to calibrate using OxCal Calibration (Table 1).

Table 1. ^{14}C ages and Radiocarbon Ages Calibration

No	No. Sampel	Location	^{14}C Age (BP/Before Present)	Calibrated Age (BC/ Before Christ)
1	MOL-CD1	Grobogan	23127 ± 107	25692 - 25252
2	**MOL-CD2**	**Sangiran**	**35390 ± 258**	**38710 - 37383**
3	MOL-CD3	Patiayam – Muria	23871 ± 112	26248 - 25735
4	MOL-CD4	Grobogan	7571 ± 39	6479 - 6379
5	MOL-CD5	Patiayam – Muria	12227 ± 56	12426 - 11994
6	**MOL-CD6**	**Sangiran**	**30216 ± 183**	**32672 - 31947**

***Bold: the oldest area**. Underline: the youngest area

3.2. Marfometric Identification of Mollusc Fossils

The specimens of mollusc fossils were collected from Sangiran, Patiayam (Ancient Muria Strait), and Grobogan (Figure 2, 3 and 4).

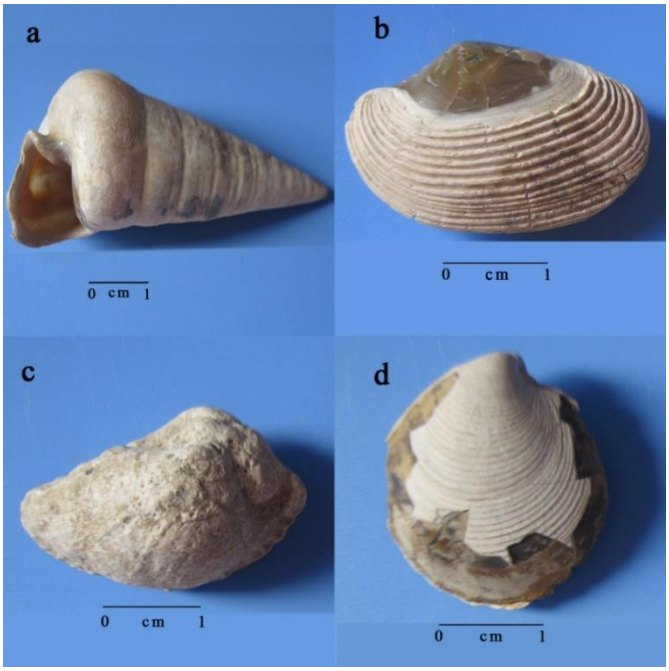

Figure 2. Collected samples from Sangiran: a) *Telescopium telescopium* Length: 111.05 mm; b) *Dosinia sp.* Length: 57.60 mm; c) *Anadara sp.* Length: 55.25 mm; d) *Dosinia insularum* Length: 42.05 mm;

The result showed that ^{14}C analysis of marine mollusc fossils collected from The Archeological Site of Sangiran, Patiayam (Ancient Muria Strait) and Grobogan. The AMS ^{14}C result has to calibarate using radiocarbon calibrated age (e.g. OxCal Calibration) [12,13,14]. The oldest age (38710 – 31947 years) was obtained for mollusc fossils sample collected from the Archeological Site of Sangiran. The result showed that marphometric identification of mollusc fossils from Sangiran are *Dosinia sp.*, *Telescopium telescopium*, *Anadara sp.* and *Dosinia insularum* (Figure 2). Sangiran during the Pleistocene became one part of the Java Island have experienced several deformation due to fluctuations

in sea level surface [15]. Lithology change stems from the ocean turns into a swamp and then go trought with land by the activity of an ancient volcano with different formation lithology [3].
Based on stratighraphic analysis, Sangiran area were started with Kalibeng formation (Pliocene), Pucangan formation (early Pleistocene), Kabuh formation (middle Pleistocene) and Notopuro formation [3][6]. Marine mollusc fossil could be found near to Kalibeng formation in Puren River and Karangrejo. Field validation sampling had confirmed that the result of radiocarbon dating sample (absolute ages) and geology age are the same result. Sangiran was the oldest formation as the deepsea zone.

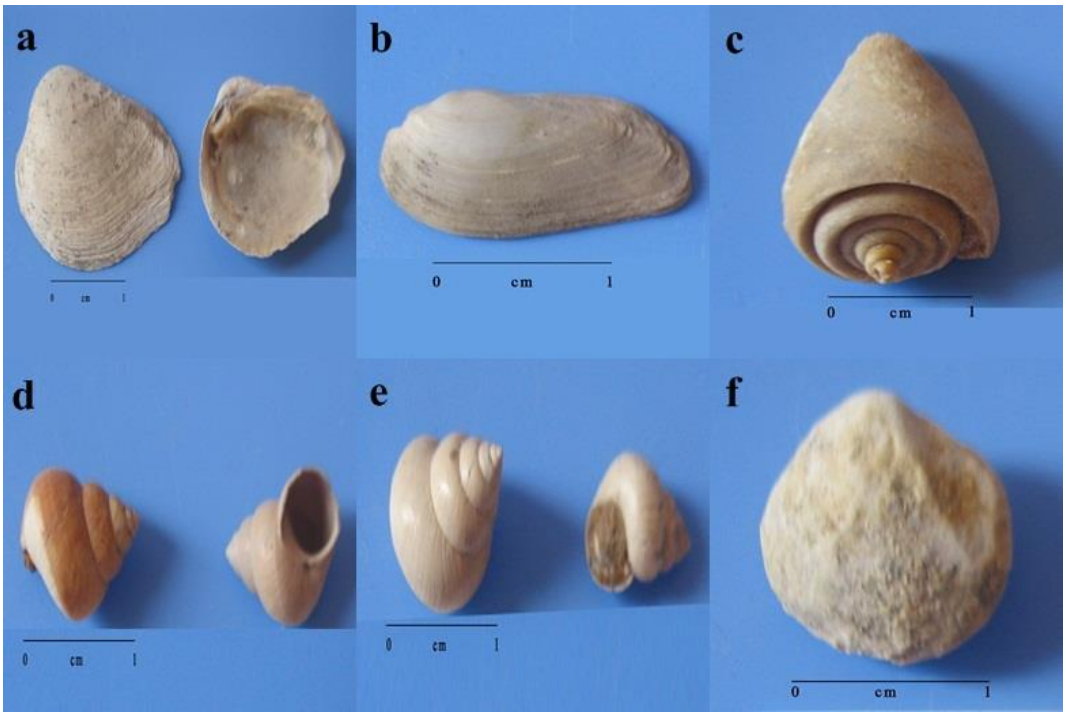

Figure 3. Collected samples from Patiayam – Ancient Muria Strait: a) *Pseudodon vondenbuschianus* Length: 49.80 mm; b) *Elongaria orientalis* Length: 37.95 mm; c) *Conus (Pionoconus)* sp. Length: 43,70 mm; d) *Ampullina bandongensis* Length: 22.05 mm; e) *Filopaludina javanica* Length: 27.85 mm; f) *Anadara (cunearca) pilula* Length: 28.55mm.

Fossils age from Patiayam (Ancient muria Strait) gave the result 26248 – 11994 years with mollusc fossils: *Pseudodon vondenbuschianus, Elongaria orientalis, Ampullina bandongensis, Filopaludina javanica* and *Anadara (cunearca) pilula* (Figure 3). Lithology of Ancient Muria Strait (Patiayam) divided into six formations, they are Jambe formation (Pliocene), Kancilan formation (early Pleistocene), Slumprit formation (middle Pleistocene), Kedungmojo formation, Sukobubuk formation (the end of Pleistocene) and "endapan teras" (Holosen). There are two of the period of the environment precipitation in Patiayam, they are Jambe formation in a shallow seas and four formation that the youngest area. In addition Kancilan formation was the beginning of the changes to the environment [2][16].

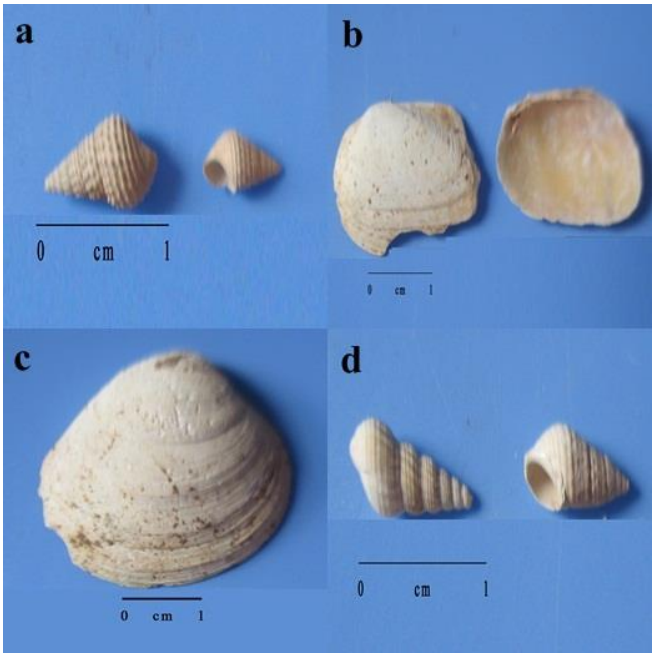

Figure 4. Collected samples from Grobogan: a) *Plotia scabra* Length: 14.05 mm; b) *Cultelus dilatatus* Length: 49.60 mm; c) *Antigona chemnitzii* Length: 68.05 mm; d) *Tibia modesta* Length: 19.75 mm.

While in above formation was found as the late miocene in Grobogan with limestone and identified as shallow seas. This area was the youngest area with radiocarbon dating ages 25692 – 6379 years. The zone was known in geological term as 'the north Kendeng limestone/carbonate mountain or the front-arch formation consisting with the denundation of Globigerina – limestone [6]. The field validation on the coordinate position of Grobogan had discover sampels of limestone sediment and mollusc fossils such as *Antigona chemnitzii, Cultelus dilatatus, Plotia scabra* and *Tibia modesta*.

4. Conclusion

Mollusc fossils sample was analysed using radiocarbon dating with the result that Sangiran as an ancient depsea (the oldest area) has fossils age 38710 – 31947 years, then Patiayam (Ancient Muria Strait) with fossils age 26248 – 11994. Mollusc fossils from Grobogan was the youngest area has fossils age 25692 – 6379 years.

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