Fossilization Type of *Elephas hysudrindicus* from Blora on the Basis of Petrographic and Scanning Electron Microscopic Analyses

Tipe Fosilisasi Elephas hysudrindicus dari Blora berdasarkan Analisis Petrografi dan Scanning Electron Microskopis

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Abstract

Either fossils of the hominid or vertebrate have long been known from terraces along the Solo River in Central and East Java. Most terraces consist of andesitic sand to andesitic tuffaceous sand with either gravel-pebble or conglomerate and some of them contain vertebrate fossils. It is in this place, an ancient elephant fossil named *Elephas hysudrindicus* was discovered in 2009. This fossil was discovered at an abandoned sand quarry of Sunggun area, Medalem Village, Kradenan Subregency, Blora Regency which and can be mentioned as a great event for the Geological Museum. It was said as a phenomenal discovery, because the fossil was found within the terrace with condition of nearly complete skeleton of an individual elephant. Some bone fragments of *Elephas hysudrindicus* fossil is treated as rock specimens because a number of minerals fill in either pore spaces or cavities or cracks within bones, and such infilling minerals can be observed in cut sections of the bones. Main goals of the study are to determine the distribution and type of minerals within the bones, interpret environment of deposition, and identify fossilization type. The methodology used in this study consists of petrographic and Scanning Electron Microscopic (SEM) analyses. Based on the petrographical observation, some bone specimens of *Elephas hysudrindicus* fossil are characterized by fibrous and porous feature with cracks occuring locally. Whilst, examination with SEM shows that the bone specimens are apparently composed of collophane or massive cryptocrystalline variety of apatite as the principal component of fossil bone, having physical characteristic of spheroidal structure and cavities of 100 to 1500 micron (μ) in diameter. Most cavities and pore spaces are predominantly filled in by either authigenic crystals of rhombohedral calcite and lesser pseudohexagonal kaolinite with either slightly minerals of manganese oxide or iron oxide or ilmenite, including oxidized kaolinite and calcium iron silicate. Impregnation during diagenesis may be the most appropriate expression for fossilization process of the *Elephas hysudrindicus*. It is indicated by the existing authigenic minerals within the bones cavities, pore spaces, and cracks which are possibly due to precipitation of mineralized fluids originated from groundwater within the terrace.

Keywords: Sunggun terrace, *Elephas hysudrindicus*, authigenic minerals, fossilization

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Fosil-fosil hominid atau vertebrata telah lama dikenali dari endapan-endapan undak sepanjang Bengawan Solo, Jawa Tengah dan Timur. Sebagian besar endapan tersebut terdiri atas pasir andesitis dan pasir tufan andesitis dengan kerikil-kerakal atau konglomerat, dan sebagian mengandung fosil-fosil vertebrata. Di tempat ini pula ditemukan fosil gajah purba yang dinamai Elephas hysudrindicus pada tahun 2009. Fosil tersebut ditemukan di daerah bekas penggalian pasir, daerah Sunggun Desa Medalem, Kecamatan Kradenan, Kabupaten Blora dan dapat disebut sebagai peristiwa besar bagi Museum Geologi. Sepantasnya penemuan ini dikatakan fenomenal karena kerangka seekor gajah ditemukan di dalam endapan teras dengan kondisi hampir lengkap. Beberapa fragmen fosil tulang Elephas hysudrindicus diperlakukan sebagai percontoh-percontoh batuan karena mineral-mineral mengisi ruang-ruang pori atau bukaan-bukaan atau rekahan-rekahan, dan setiap mineral pengisi dapat diobservasi melalui sayatan-sayatan tipis tulang. Tujuan studi adalah untuk menentukan sebaran dan jenis-jenis mineral di dalam tulang, menginterpretasikan lingkungan pengendapan, dan mengidentifikasi jenis fosilisasi. Metodologi yang digunakan berupa analisis petrografi dan mikroskop pemindai

elektron (Scanning Electron Microscope - SEM). Berdasarkan observasi petrografi, diketahui bahwa sebagian contoh tulang Elephas hysudrindicus berserat dan berpori dengan rekahan-rekahan yang terbentuk setempat. Sementara pengujian dengan SEM menunjukkan bahwa percontoh-percontoh tulang disusun oleh collophane atau beraneka kristal halus apatit masif sebagai komponen utama tulang, memiliki ciri fisik struktur mengulit bawang dan bukan-bukaan berdiameter 100 hingga 1500 micron (μ). Sebagian besar bukaan dan pori diisi terutama oleh mineral-mineral autigenik berupa kristal-kristal kalsit rombohedral dan sebagian kaolinit pseudo-heksagonal, dengan sedikit oksida mangan atau oksida besi atau ilmenit termasuk juga kaolinit teroksidasi dan silikat Ca-Fe. Impregnasi selama diagenesis merupakan ungkapan yang tepat bagi proses fosilisasi terhadap Elephas hysudrindicus tersebut. Hal tersebut ditunjukkan oleh keberadaan mineral-mineral autigenik di dalam bukaan-bukaan, ruang-ruang pori, dan rekahan pada tulang-tulangnya yang kemungkinan disebabkan oleh pengendapan fluida-fluida termineralisasi yang berasal dari air tanah di dalam endapan undak.

Kata kunci: *undak Sunggun, Elephas hysudrindicus, mineral-mineral autigenik, fosilisasi*

Introduction

A terrace is a step-like landform that borders river floodplain containing alluvial deposits or alluvial fill (Bates and Jakson, 1980). It's referred too as a flat landform with horizontal or gentle sloping surface, sometimes long and narrow with one side is bounded by a steeper ascending slope and on the other by a descending steeper slope (Zuidam, 1985).

Either hominid or vertebrate fossils have long been known from terraces along the Solo River in Central and East Java – for instance, at Sambung-macan, Trinil, Ngawi, and Ngandong (Dubois, 1894; Es, 1929; and Haar, 1934). These deposits have remained as a focus for palaeontological and archaeological researches up to the present.

Sartono (1976) distinguished six phases of ter-race formation along the Solo River comprising: 1) The Rambut Terrace is 97 m above the Solo River bed and is assumed of Early Pleistocene age; 2) The Kedungdowo Terrace is 82 m above the river and of Early Pleistocene age; 3) The Getas Terrace is 57 m above river surface and of Middle Pleistocene age. 4) The Ngandong Terrace is 20 m above the river and of Upper Pleistocene age; 5) The Jipangulu Terrace is 7 m and of Early Holocene age; and 6) The Menden Ter-race is 2 m above river surface and of sub-Recent age.

Sidarto and Morwood (2004) identified that most terraces of point bar sediments along the Solo River area were deposited in a meandering system. They can be identified as much as 23 deposits and combined into four chronological groups as follows (Figure 1 and 2):

1) The Sembungan Terrace
2) The Pandean, High Pendem, and High Karsono Terraces
3) The Jimapulu and High Sugihan Terraces,
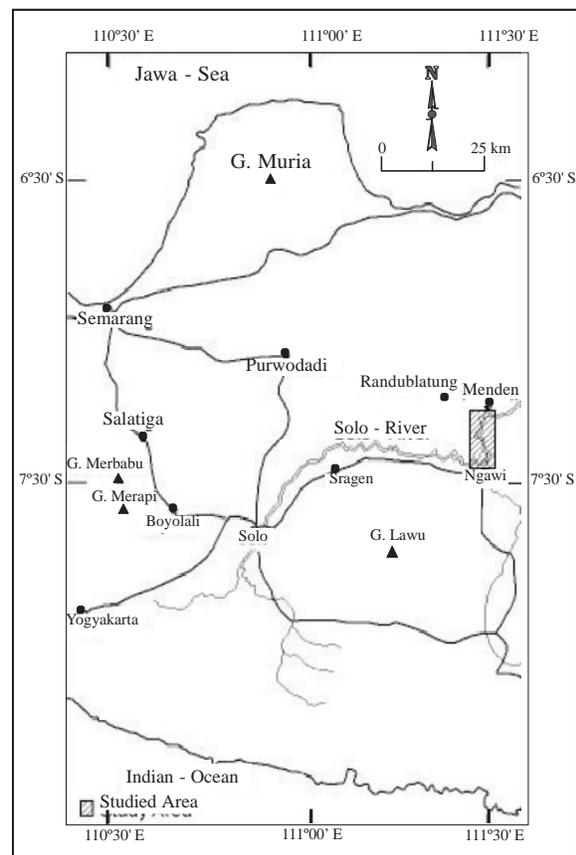4) The Padasmalang, Low Karsono, Low Sugihan, Low Pendem, and Kudu Terraces correlated with The Menden Terrace.

Figure 1. Location map of studied area along the Solo River within Blora Regency, East Java Province (Sidarto and Morwood, 2004).

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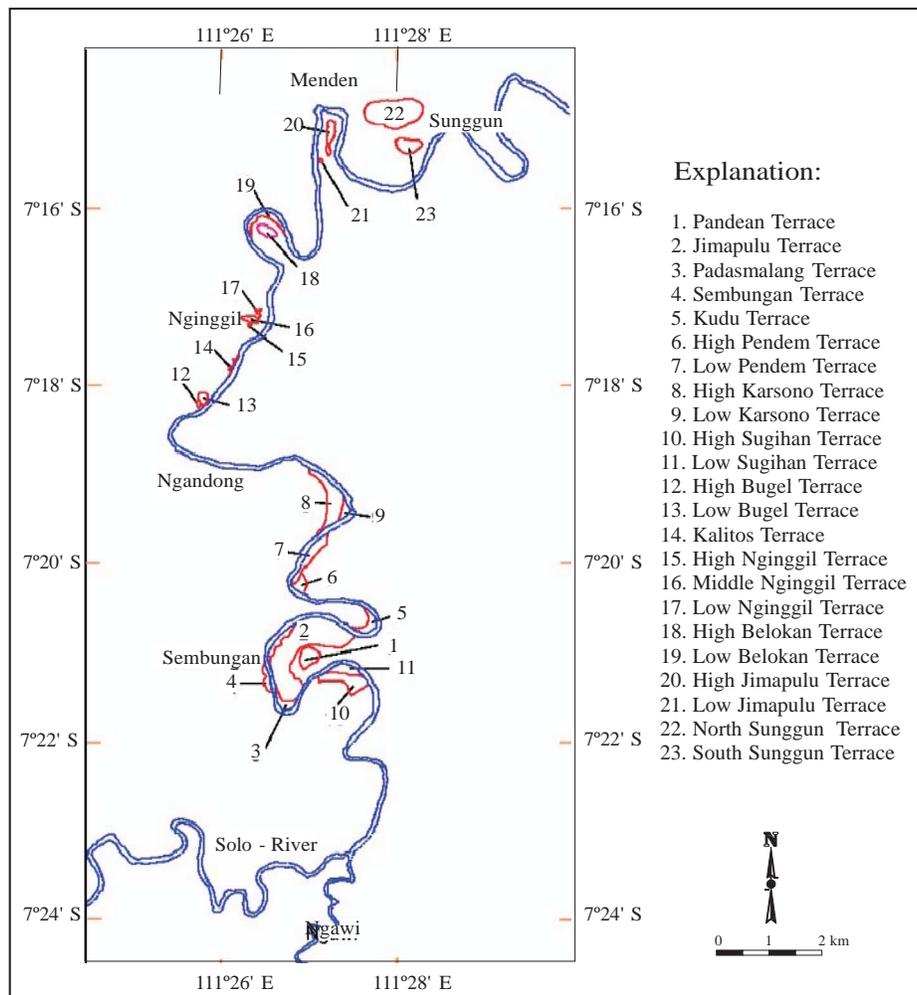of Petrographic and Scanning Electron Microscopic Analyses (D. Z. Herman)

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Figure 2. Distribution of terraces along the Solo River within Blora Regency, East Java Province (Sidarto and Morwood, 2004). One terrace in Sunggun area contains an ancient elephant fossil named *Elephas hysudrindicus*.

Most terraces consist of andesitic sand to andesitic tuffaceous sand with either gravel-pebble or conglomerate and some of them contain vertebrate fossils. It is in this place that an ancient elephant fossil named *Elephas hysudrindicus* was discovered in 2009.

The discovery of this fossil at an abandoned sand quarry of Sunggun area, Medalem Vilage, Kradenan Subregency, Blora Regency can be mentioned as a great event for Geological Museum. It was said as a phenomenal discovery, because the investigation on it had been conducted since 1850s, and it is for the first time that the fossil was found within the terrace with nearly complete skeleton of an individual elephant (Figures 3 and 4).

Figure 3. *Elephas hysudrindicus* fossil was being dug from a terrace of Sunggun area.

Figure 4. Close up photograph of lower jaws of *Elephas hysudrindicus* within a terrace of Solo River Sunggun area (Geological Museum Document, 2010).

Methodology

In this study, some bone fragments of *Elephas hysudrindicus* fossil are treated as rock specimens because minerals fill in either pore spaces or cavities or cracks within bones, and such infilling minerals can be observed in cut sections of the fossil bones. Main goals of the study are to determine the distribution and type of minerals within fossil bones, interpret environment of deposition, and identify fossilization type during diagenesis process. The methodology used in this study consists of petrographic and Scanning Electron Microscopic (SEM) analyses.

A polarizing microscope is used to identify and estimate relative abundance of infilling mineral content from some thin sections of a certain bone. Whilst, two selected specimens of fossil bone are examined by using Scanning Electron Microscope (SEM) for determination three-dimentional of their textures and mineral contents. SEM Type JEOL JSM-6360LA – 2003 is a microscope equiped by Energy Dispersive X-Ray Spectrum (EDX) JED-2200 Series and proved useful for determination great depth of object fields, including texture and mineral content of specimens. The microscope is finely focused on a beam of electron or magnetically moved across specimens to be examined, from point to point, and the reflected and emitted electron intensity was measured and displayed, sequentially building up three dimentional image with magnification rate of 10 to 300.000 time and bar scale of 0.1 to 10 micron (μ).

Analysis Result

Petrographic observation identifies that bone specimens are characterized by fibrous and porous features with cracks occuring locally. Cavities or pores and cracks seem to be more susceptible to infilling of authigenic opaque minerals which are possibly as either hematite or goethite or limonite (Figure 5). Whereas, some cracks were either filled in by chlorite or oxidization (Figure 6). Other observation exhibits that cavities of bone were infilled by kaolinite (Figure 7).
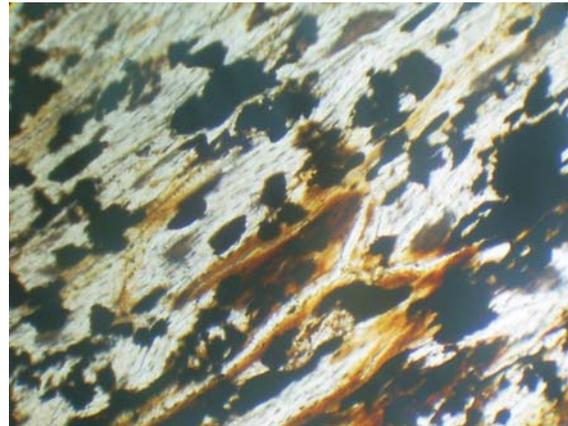

Figure 5. Photomicrograph shows fibrous and porous bone which have been filled in by authigenic opaque minerals (yellowish brown - blackish, possibly hematite or goethite or limonite), whereas cracks are filled by chlorite (pale yellow) (24 x enlargement, crossed Nicols).
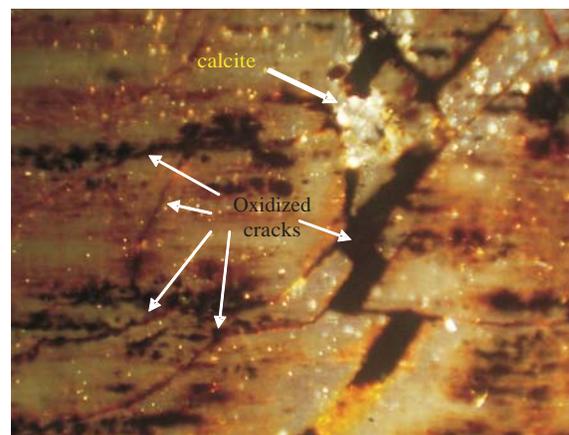

Figure 6. Photomicrograph shows that the bone are cracked and oxidized (yellowish - reddish brown) with partly replaced by authigenic calcite (24 x enlargement, crossed Nicols).

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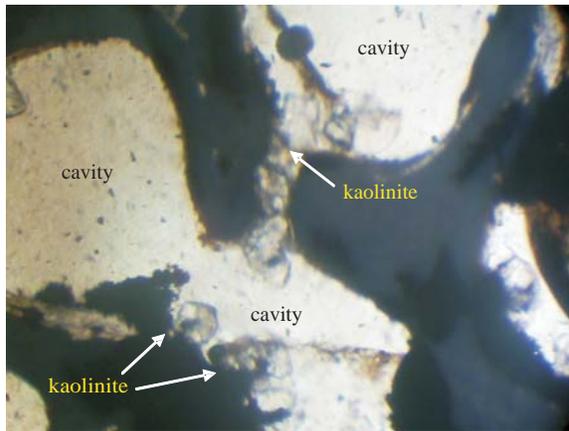of Petrographic and Scanning Electron Microscopic Analyses (D. Z. Herman)

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Figure 7. Photomicrograph shows authigenic kaolinite within bone cavities. Kaolinite is colorless - pale yellow and low relief (32 x enlargement, crossed Nicols).

Figure 9. Photomicrograph SEM exhibits that cavities are filled in by authigenic mineral crystals of predominantly rhombohedral calcite (Ca) and less pseudohexagonal kaolinite (K) (Geology Laboratories PSG, 2010).

Based on Scanning Electron Microscopic (SEM) analysis (Geology Laboratories PSG, 2010), both specimens of bone (BL-1 and BL-7) are apparently composed of collophane or massive cryptocrystalline variety of apatite $[Ca_5(PO_4)_3(OH)]$ as the principal component of fossil bone, having physical characteristic of conchoidal structure and cavities of 100 to 1500 micron (μ) in diameter (Figure 8). Most cavities of the first specimen are filled in by authigenic mineral crystals of either rhombohedral calcite or pseudohexagonal kaolinite, and less manganese oxide (Figure 9).

Whereas, the second specimen cavities are predominantly filled in by either authigenic crystals of calcite and lesser kaolinite with either slightly minerals of manganese oxide (MnO) or iron oxide (Fe_2O_3) or ilmenit ($FeTiO_3$) or oxidized kaolinite and calcium iron silicate ($CaFeSi_2O_6$) as shown in Figures 10 to 13.

dIscussIon

Fossilization process of organism is complex, and the result is determined by a variety of factors such as physics, chemistry, and biology. However, three main stages in fossilization of an organism might effectively be identified as death, pre-burial, and post-burial.

Physical and chemical causes of death are useful clues for geologists in determining the nature of ancient environments. A death of an organism in a low-oxygen (anaerobic) environment will also ensure that a process of decay will be arrested. In most cases, this is rare and most fossils have been subjected to some decay and degradation process before burial. Soft parts will be attacked firstly through the action of scavengers and the biochemical process of decay. In low-oxygenated conditions, this process may be slow but not arrested, and decay leads to the production of more complex hydrocarbon molecules. Preservation of soft parts is therefore unusual
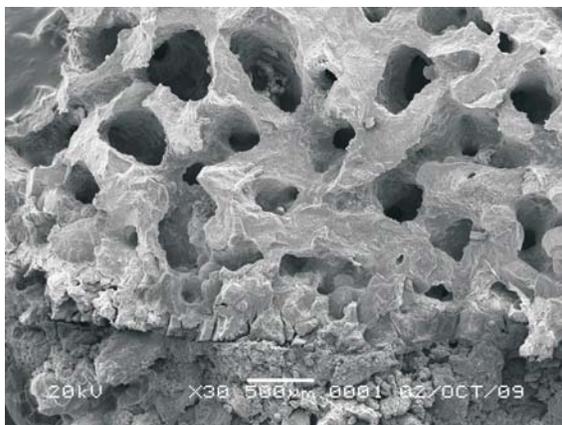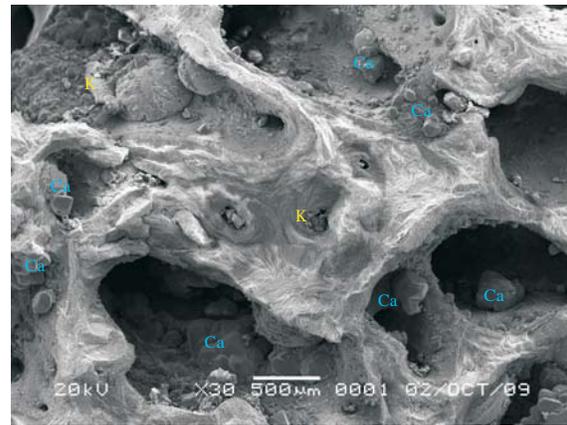

Figure 8. Photomicrograph SEM shows that fossil bone is apparently composed of collophane or massive cryptocrystalline variety of apatite $[Ca_5(PO_4)_3(OH)]$ as the principal component, having physical characteristic of spheroidal structure and cavities of 100 to 1500 micron (μ) in diameter (Geology Laboratories PSG, 2010).
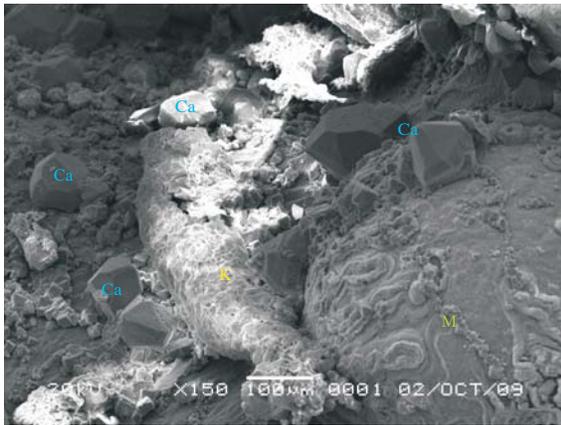
Figure 10. Photomicrograph SEM indicates an appearence of predominantly authigenic calcite (Ca) and kaolinite (K) and less manganese mineral/MnO (M) which have been sticked on bone wall (Geology Laboratories PSG, 2010).
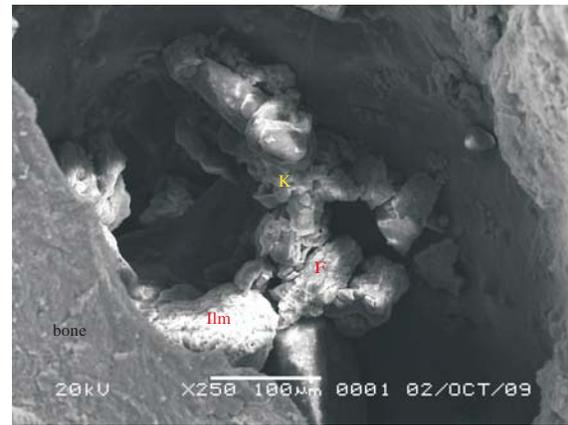

Figure 12. Photomicrograph SEM shows an appearence of authigenic kaolinite (K), ilmenite/$FeTiO_3$ (Ilm), and iron oxide/Fe_2O_3 (F) as infillings in a cavity of bone (Geology Laboratories PSG, 2010).
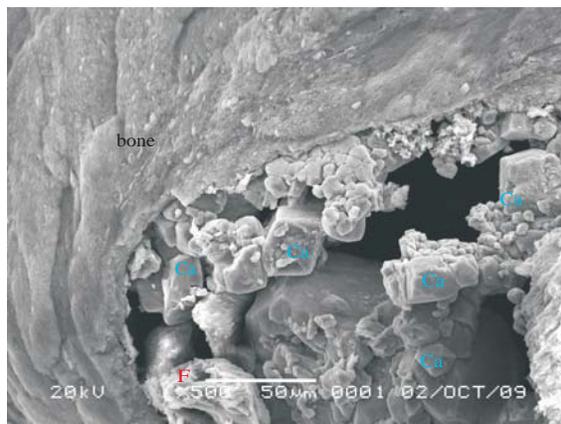

Figure 11. Photomicrograph SEM exhibits an appearence of authigenic calcite crystals (Ca) and less iron oxide/Fe_2O_3 (F) as infillings in a cavity of bone (Geology Laboratories PSG, 2010).
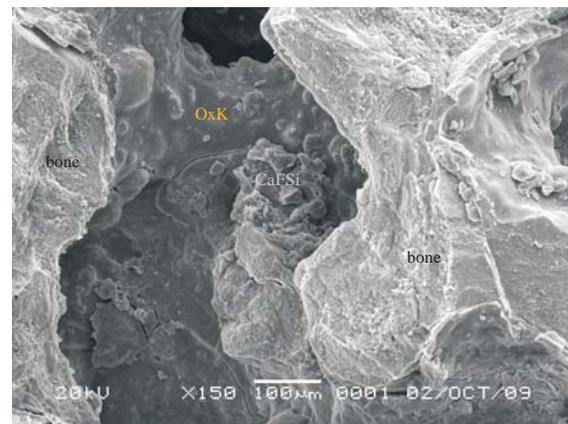

Figure 13. Photomicrograph SEM indicates that cavity wall had been concealed by oxidized kaolinite (OxK) and calcium iron silicate/$CaFeSi_2O_6$ (CaFSi) (Geology Laboratories PSG, 2010).

prior to burial, although early mineralization of soft tissue has been recorded (Martill, 1988). However, in hot and arid or cold environments, unburied soft parts can become dried leaving mummified remains (Spindler, 1995). Inspite of this, in most cases preservation of soft parts is through the growth of minerals early after the burial of an organism.

After burial, the dead organism can be affected by a variety of chemical and physical factors. Rapid burial ensures that the organism is not under physical attack from scavengers, and/or mechanical transport by currents and wind action. Soft parts may be pre-

served in this way, and delicate articulated skeletons may be held in place.

Most records of soft part preservation are associated with low-oxygen (anaerobic) conditions and rapid burial associated with a high sedimentation rate. The decay process is slowed but anaerobic bacteria which can survive in such environments assist in breaking down the soft parts into hydrocarbons. Usually, it is not actual soft parts which are preserved, but a replica or outline created by the growth of minerals soon after the death of the organism. The type of mineral (*e.g.* pyrite, carbonates, phosphates,

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and silicates) replacing the soft parts is determined by the nature of the chemical environment; in pyrite growth it is favoured in anaerobic conditions. The commonest type of preservation is mineral coating, where mineral growth occurs on the surface of the decaying soft body, leaving behind an outline of the original soft body.

In other cases, the physical impressions of the soft parts may be preserved in some sediments, particularly in fine-grained ones. These preserve no mineralized trace of the soft parts, but rather the physical presence and surface features. In general, burial increases the changes of soft-part preservation, as it helps prevent biochemical break down through the exclusion of oxygen. However, once buried, the organism comes under a chemical attack from pore fluids contained in the sediments, and from physical disturbance as the sedimentary pile is compacted. These changes are collectively referred to as the process of diagenesis.

Specifically, diagenesis is the cumulative physical, chemical, and biological environments; these processes will modify an organic object original chemical and/or structural properties and will govern its ultimate fate, in terms of preservation or destruction (Wilson and Pollard, 2002; Zapata, 2006). In order to assess the potential impact of diagenesis on bone fossil, many factors need to be assessed, beginning with elemental and mineralogical composition of bone and enveloping soil, as well as the local burial environment (geology, climatology, and groundwater).

The composite nature of bone, comprising one third organics (mainly protein collagen) and two thirds mineral (calcium phosphatemostly in the form of hydroxyapatite) renders its diagenesis more complex (Nicholson, 1996). Alteration occurs at all scales from molecular loss and substitution, through crystalline reorganization, porosity and microstructural changes, and in many cases, to disintegration of the complete unit (Nielsen-Marsh, 2000). Three general pathways of the diagenesis of bone have been identified (Collins, 2002; Hedges, 2002) as follows:

1. Chemical deterioration of the organic phase. The dissolution of collagen depends on time, temperature, and environmental pH. At high temperatures, the rate of collagen loss will be accelerated and extreme pH can cause collagen swelling and accelerated hydrolysis. Due to the increase in porosity of bones through collagen loss, the bone becomes susceptible to hydrolytic infiltration where the hydroxyapatite, with its affinity for amino acids, permits charged species of endogenous and exogenous origin to take up residence.

2. Chemical deterioration of the mineral phase. The hydrolytic activity plays a key role in the mineral phase transformation that exposes the collagen to accelerated chemical- and bio-degradation. Chemical changes affect crystallinity. Mechanisms of chemical change, such as the uptake of F^- or CO_3^- may cause a recrystallization where hydroxyapatite is dissolved and re-precipitated allowing for the incorporation of substitution of exogenous material.

3. (Micro) biological attack of the composite. Once an individual has been interred, microbial attack, the most common mechanism of bone deterioration, occurs rapidly. During this phase, most bone collagen is lost and porosity increased. The dissolution of the mineral phase caused by low pH permits access to the collagen by extracellular microbial enzymes thus microbial attack.

There are some fossilization processes associated with diagenesis: preservation of original material, recrystallization, impregnation, encrustation, and compaction. The first process indicates that the flow of pore fluids is restricted, the original chemistry may remain unaltered, and colour patterns or coatings may be preserved intact; original skeletal material is more commonly preserved in younger rocks, as with increasing age the likelihood of alteration increases. Recrystallization is achieved without change in the chemical composition, but involving the replacement of the original skeletal material by a new mineral. Replacement can be through the growth of large mineral crystals at specific points, at the expense of the original structure (grain growth) or through chemical change (metasomatism) of the original material. Impregnation (is also often called as petrification) occurred where skeletal is notably porous. In this case, mineralized pore fluids lead to precipitation of minerals inside the pore spaces of bones, for instance. Encrustation takes place where the skeletal is surrounded by a crust of a new material, and this is common in areas of hot springs where mineral salts supersaturate the warm waters and are quickly precipitated onto bone material.

Vertebrates are reasonably well represented in the fossil record because they have hard parts, bones, and teeth made up of apatite $[Ca_5(PO_4)_3(OH)]$. In rare cases, soft parts may be preserved when decay is prevented. Vertebrate bodies decay as they are valuable sources of food for other organisms. When large animals feed on the flesh of a dead vertebrate, the process is termed scavenging; and as microbes transform the tissues, the process is termed decay. In terrestrial settings, carcasses may be picked over by large scavengers, and when they have had their fill, smaller animals such as meat-eating beetles, may move in (Benton, 2006). Certain vertebrates are found in situations of exceptional fossilization, where early mineralization has preserved even soft tissues.

Fossil bone is an important tool for paleoenvironmental studies, because it retains information about the prevailing conditions before and during fossilization. Most studies have been focused on differences in surface features or the chemical composition of bones, correlating certain features with particular environmental contexts. Though the presence of authigenic mineral growth has been noted on modern, archeological, and either fossil bone surfaces or cavities or pores, it is commonly overlooked. Many of the minerals formed under particular conditions (pH, Eh, porewater chemistry) are suitable (paleo) environmental indicators. Despite the observed relationship between environmental conditions and mineral formation on bone, little is understood regarding how bone interacts with the burial environment during diagenesis.

Authigenic minerals are formed in sediments or sedimentary rock. Their in-place origin distinguishes them from minerals that are formed elsewhere and transported to the site of deposition (detrital minerals). Authigenic minerals were formed at the earth surface as well as during subsequent burial. The postdepositional processes are referred to as diagenesis, and the resulting minerals are important clues to postdepositional physical and chemical changes in the rock.

Authigenic minerals precipitate from the overlying water column, pore fluids in the sediment, recrystallization or alteration of pre-existing minerals, and structural transformation of one mineral to another. The minerals change in an attempt to equilibrate to the physical and chemical conditions present at any given time. Critical factors in their formation are initial mineral assemblage, temperature, pressure, ionic concentration, pH, electron availability, and the fluid flux through the rock.

In sedimentary rocks, it is common to find a record of multiple diagenetic events based on the authigenic minerals. For example, in sediments near the surface, meteoric water may displace original marine pore water, resulting in distinct types of cements. Iron oxide can result from oxidizing fluid. Depletion of oxygen by bacteria may result in the formation of iron sulfides. During burial, the sediments respond to increasing temperature (up to 200°C), pressure (up to 2.5 kilobars), and fluid movement from compaction-driven waters or influx of water from the basin flanks. As a result, the sedimentary rock may contain authigenic minerals that record a sequence of events ranging from processes occurring near the sediment-water interface to those forming during deep burial. Unlike metamorphic rocks, the pre-existing (detrital) mineral assemblage is at least partially retained, in part due to the sluggish reaction rates at diagenetic conditions. Early cementation processes often seal up the rock, preventing subsequent diagenetic reactions and preserving the original detrital mineral assemblage.

Examination with petrographic mode indicates either bone cavities or pores seem to be more susceptible to filling in by some authigenic minerals which are possibly as either hematite or goethite or limonite. Whereas, some cracks within bone were either filled in by chlorite or oxidization or calcite. Other observation exhibits that cavities of bones were infilled by kaolinite. Examination with SEM shows infilling crystals within pores and cavities are calcite and lesser kaolinite with either slightly minerals of manganese oxide (MnO) or iron oxide (Fe_2O_3) or ilmenit ($FeTiO_3$) or oxidized kaolinite and calcium iron silicate ($CaFeSi_2O_6$).

Authigenic minerals occur in all sedimentary rock and can vary from trace amounts to virtually the total rock. Carbonate mineral calcite is the most common type and form in a wide range of depositional environments and at varying burial depths. Considering that most terraces are predominantly composed of andesitic sand to andesitic tuffaceous sand, they may originate from volcanic product or pyroclastic. Though, calcite may be released by dissolution of old feldspar within those volcano-

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genic sediments and then deposited and infilled cavities or pores within bone fossil. Borowski *et al.* (2001) mentioned on the basis of his experiment that calcite has been formed at current (or slightly shallower) burial depths from pore water dissolved inorganic carbon with $\ddot{a}^{13}C$ being controlled by methane oxidation or generation at depth less or greater than 200 m below sea floor (bsf). The preferred alternative interpretation is that all of the carbonates were formed at shallower depths (20 - 200 mbsf) or just below the sulfate/methane interface.

Because of the chemical reactivity and stability of their particles, pyroclastics are highly susceptible to diagenetic alteration. Glass may alter to clay minerals, zeolites, chalcedony, opal, quartz, or to microcrystalline particles. Formation of infilling or concealing kaolinite on certain cavity walls of fossil bone is possibly having relationship with this alteration process. On the other hand, chlorite is possible converted from clay mineral (kaolinite) and then isolated cracks within bone fossil provide an interesting window into the behaviour of this mineral. Lundegard and Land (1989) mentioned that its layer structure with a high concentration of hydroxyl and precipitation of chlorite enabled to make a potential reverse-weathering reaction.

The authigenic iron and manganese oxides, including calcium iron silicate are believed to have been precipitated from groundwater containing dissolved ferrous, manganese, and calcium ions. These ions were derived from the intrastratal solution of detrital iron, and manganese bearing grains, including ferromagnesian silicates. Whereas, the materials of entirely elements are probably originated from the terraces as just only source.

Conclusions

- Based on petrographical observation, most bone specimens of *Elephas hysudrindicus* are characterized by a fibrous and porous feature with cracks occuring locally. Whilst, examination with SEM shows that the bone specimens are apparently composed of collophane or massive cryptocrystalline variety of apatite $[Ca_5(PO_4)_3(OH)]$ as the principal component of fossil bone, having physical characteristic of

conchoidal structure and cavities of 100 to 1500 micron (μ) in diameter.
- Most cavities and pore spaces are predominantly filled in by either authigenic crystals of rhombohedral calcite and lesser pseudohexagonal kaolinite with either slightly minerals of manganese oxide (MnO) or iron oxide (Fe_2O_3) or ilmenite ($FeTiO_3$), including oxidized kaolinite and calcium iron silicate ($CaFeSi_2O_6$).
- Impregnation during diagenesis process may be the most appropriate expression for fossilization of the *Elephas hysudrindicus*. It is indicated by the existing authigenic minerals within the bones cavities, pore spaces, and cracks which possibly due to precipitation of mineralized fuids originated from pore spaces of the terrace.

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