Multiple issues when preparing and preserving Late Miocene beaked whale (Gram Fm.) for research and exhibition

Trine Sørensen, Department of Conservation, Museum of Southern Jutland, Fabriksvej 17-21, DK-6510 Gram, Denmark. e-mail: trsr@museum-sonderjylland.dk

Introduction

A fossil whale was discovered in Gram Clay Pit (Late Miocene, Gram Fm.) in 1986. It turned out to be the first fossil of a beaked whale (*Ziphiidae*) to be found in Denmark.
Various taphonomic processes made the rare specimen far from the best preserved whale fossil from Gram consisting of only a damaged skull and a few postcranial elements. Still, clear diagnostic characters are present making the specimen of great value to science.
In 1988-94 the best preserved parts, the mandible, rostrum and several teeth, were prepared, treated for pyrite oxidation and consolidated. In 2005 the mandible was mounted for an exhibition along with 45 teeth. Funds received in 2006 made it possible to start the preparation and conservation of the cranium. Research of the whale specimen began in 2013.
Being very fragile, but heavy, the fossil needs support both when exhibited and for handling. Casts were made of the main parts of the mandible to ease research and spare the specimen.
In the process new, severe pyrite oxidation was found hidden below the fossil surface out of plain sight. This was very unexpected and prompted a revision of preventive measurements.

Taphonomy

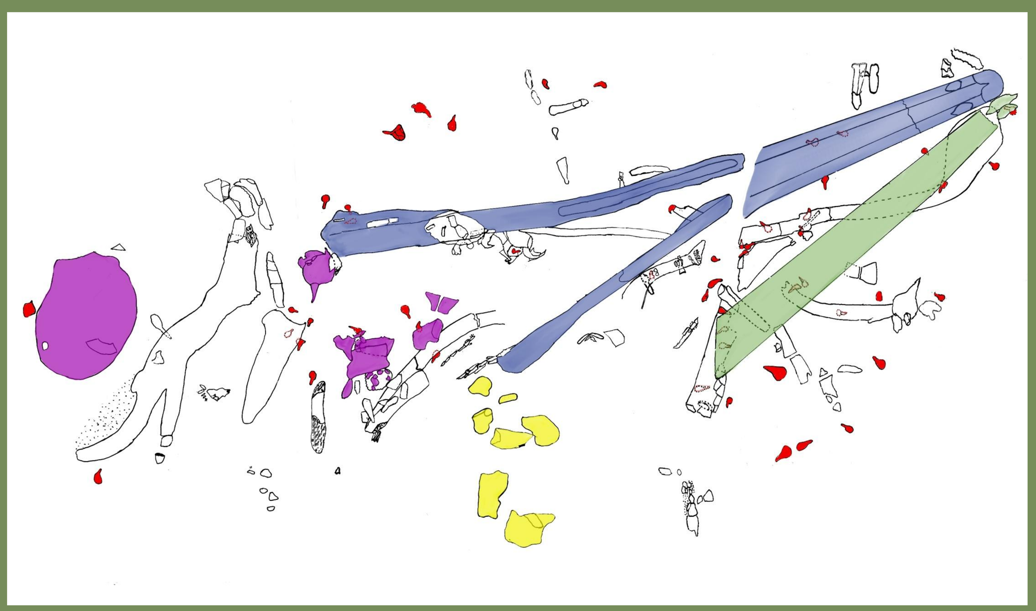

Fig. 1: A sketch of a large part of the specimen and how teeth and bones were related to each other in situ. The comprehensive disarticulation is evident.
Red: Teeth. Blue: Mandible. Green: Rostrum. Yellow : Fragments of cranium. Purple: Vertebrae. Not coloured parts are mainly ribs and unidentified fragments. Modified from preparators drawings from 1988-94.

The taphonomic processes ensured preservation of the specimen through permineralization and substitution, but it also caused a lot of damage to the beaked whale of Gram. The specimen became completely disarticulated and fragmented by currents and scavengers before buried in sediment (fig.: 1).
Compaction and other movements within the sediment added severely to the fragmentation and displacement of elements especially in the cranium (fig.: 2).
However, local cementation by carbonates most likely contributed to the preservation -physically by giving support and chemically by slowing down the flow of water through the bones and acting as a buffer (fig.: 4).
Another authigenic mineral present is pyrite with approximately 3 % in the fossil and sediment.
Of more recent date, the digging machinery used by the brickwork processing clay from the locality also caused damage. The specimen was found when the shovel of the digging machine scraped the skull, completely destroying the ventral side facing upwards (fig.: 4).

Fig. 2: The cranium of the beaked whale (MSM 1001), dorso-lateral view. Fragmentation and displacement of vertex is seen (red circle).Some displacement and twisting occurs along the line of premaxillae and vomer (as indicated by yellow arrow).

State of preservation

Disarticulation and compaction have led to severe fragmentation throughout most of the specimen.
Oxidation of pyrite was noticeable especially on the fossil parts that had to wait 20 years for preparation. Matrix was porous and crumbling, and fossil surfaces were deteriorated by the acidic oxidation by-products (fig.: 3).
In 2013 new pyrite oxidation was discovered below surface of sound-looking fossil parts exhibited in a commonly accepted relative humidity (RH: 35-50% over one year) (fig.: 5 and 6). This was highly unexpected.
The degradation of pyrite in matrix and fossil has become an ongoing, potentially risk to the future state of preservation of the fossil. Combined with the fragmentation and displacements, it makes the fossil, particularly the cranium, unsuitable for exhibition and difficult to study. And it complicates preparation, conservation and molding.

Fig. 3: Part of the surface of the cranium, ventral side, showing several types of degradation. A long the arrow from left to right: Fragmentation and displacement → pyrite oxidation of fossil and matrix → highly degraded fossil surface.

Preparation

Preparation was performed to ensure visibility, but also to remove degraded pyrite within the unlithified matrix. Adhesion and supports were needed to stabilize the fragmented specimen.
During the initial preparation in 1988-1994 most of the mandible, the rostrum and teeth, were exposed, treated for pyrite oxidation and consolidated. The preparation was done with brushes with metal bristles and sandblasting followed by consolidation with PVB in vacuum. The last parts of the mandible were prepared in 2005 with brushes and scalpel. The entire jaw was mounted and 45 teeth were put on display.
In 2006 funds finally made it possible to start the preparation and conservation of the cranium. This was done with brushes and scalpel. Local cementation within the brain cavity was removed with pneumatic chisel. In this case pyrite oxidation went on unnoticed in a thin layer of soft matrix between concretion and fossil causing damage to the latter and had to be removed (fig.: 4).
A copolymer of acrylates was used as an adhesive (MA/EMA, Paraloid B72). It proved difficult to make good, strong adhesions due to crumbling and degraded fracture surfaces.
Absolute support during preparation was crucial. It proved difficult to make a re-usable cradle that offered enough support to the very complex and fragile shape of the cranium (fig.: 2).

Fig. 4: The cranium of the whale in postero-ventral view during preparation. Rostrum is pointing away (red arrow), the concave shape of the brain cavity is seen in the foreground. The white lines mark the border between carbonate concretion and the fossil.
Carbonates, mainly siderite, had cemented within the brain cavity in a 1-2 cm thick layer. Instead of the concretion laying right next to and into the fossil surface, there were several areas with a thin layer of grayish, soft matrix between the concretion and the inner cranium wall. The transition of the layer was abrupt both in colour and hardness. While pyrite was oxidizing in the grey layer, it was necessary to remove most of the concretion to get to the oxidizing layer and stop it from damaging the fossil.

Pyrite oxidation

Pyrite has oxidized years apart in both poorly stored, degraded parts of the whale fossil with no preparation done and in sound looking, prepared parts stored in fairly good climate.
But the latter happened out of sight below the surface of the fossil (fig.: 5 and 6).
Consequently, a revision of the remedial and especially the preventive methods of pyrite oxidation of the museum started.
As remedial treatment degradation products of pyrite oxidation were mechanically removed and the fossil was neutralized in ammonia/PEG 400 vapour (Irving, 2001). Careful monitoring in the years to come will show if it is the better solution. So far, pH spot tests 6-9 moths after treatment indicate no new oxidation. The spot tests are done by dissolving few grains in an indication fluid made of shavings of a pH pencil (Insta-Check pH pencil) (Odegaard, 2005).
In earlier years, immersion in ethanolamine thioglycollate solution was used as treatment of pyrite oxidation (Cornish, 1987). In spite of being the most thorough treatment at present, both neutralizing and removing by-products, the method has been opted out, because it is being difficult to control, very toxic and it made the fossil even more fragile.
The preventive preservation consists of controlling the relative humidity, visual inspection and pH spot tests above and below fossil surface. Oxidized fossils waiting for treatment are stored in a dry and oxygen free microenvironment (Collins, 2010). While many of the oxidized fossil parts had to wait a long time before treatment they were, a bit unconventional, neutralized before waiting in line for the removal of the degradation products.

Fig. 5: Tooth where authigenic pyrite inside the pulp has oxidized (white by-products). This was not visible on the outside of the tooth. The problem was discovered in a tooth, that fell apart during handling. Afterwards X-rays of the remaining teeth suggested pyrite inside most of them. Combined with the appearance of microscopic cracks, also seen on X-ray, the possibility of oxidation in most teeth is believed to be high.

Fig. 6: Mandible with pyrite oxidizing beneath the surface seen as white powder on fragment in close-up (MSM 1001 x44, x133, x143). The fossil material destroyed by oxidation had to be replaced for the surface fragment to have something to rest on. Epoxy putty (Apoxie Sculpt) was used with an acrylate adhesive barrier between fossil and putty.

Molding and casting

The specimen shows an unique configuration on the rostrum. When fit of the mandible to rostrum had to be tested for research it was obstructed by the support of the heavy and fragile mandible . For this reason, and to spare the fossil, it was decided to make casts of most parts of the mandible. 3D-scan and -printing were not an option for financial reasons.
Because of the multiple fragments, the tricky part was to find a suitable barrier to safeguard the fossil while making the molds. Dental modeling wax (Anutex Kemdent Modeling wax) turned out to be the best solution. It was easy to apply and could cover up very big cracks, areas of hundreds of cracks and open pores to avoid silicone entering the fossil, and it was fairly easy to remove afterwards. The modeling wax consists of plates less than 2 mm in thickness and a method was developed to stretch the wax when warm to a fraction of a millimeter thick before applying (fig.:76).
A less precise cast had to be tolerated though as the modeling wax added a bit to the size of the fossil in the most fragmented areas. Measurements for research purpose were still to be made on the original fossil so small deviations in size on the casts were accepted.

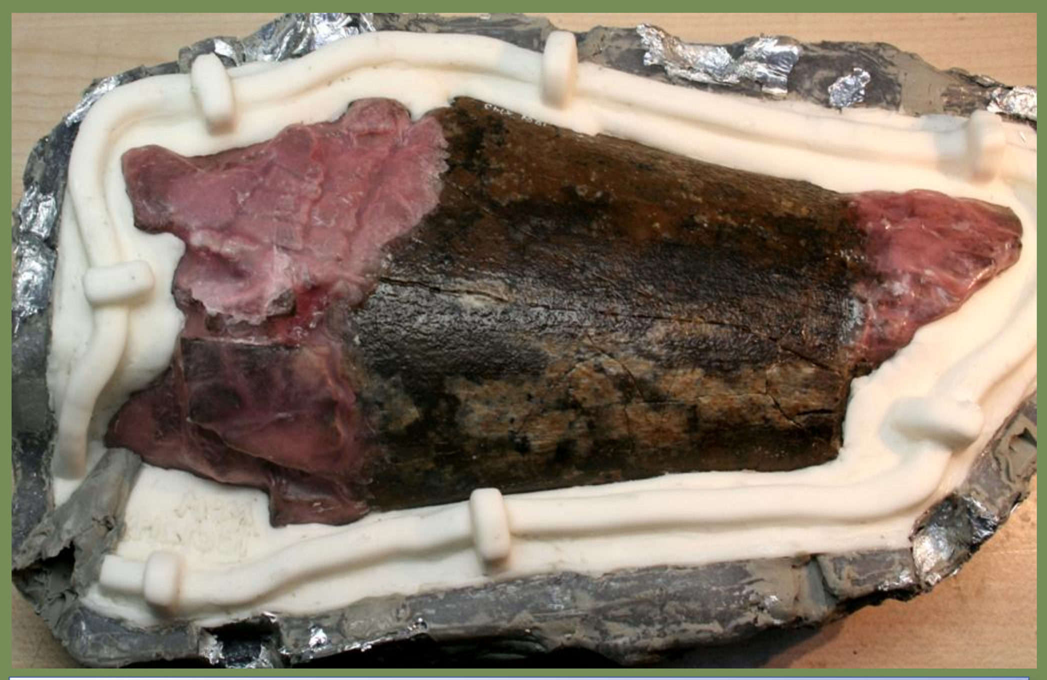

Fig. 7: Making the mold of proximal part of right mandible (MSM 1001 x 143). The barrier of dental modeling wax is seen as the pink , semi-transparent layer. The modeling wax softened at very low temperature, so it was possible to apply warm (approx. 40°C) to make a tight fit to the fossil. The edges of the wax plates were sealed with a warm spatula.
Copolymer acrylate adhesive functioned as barrier between fossil and silicone rubber where wax was not needed. A thin barrier of the adhesive was also added between fossil and wax and between wax and silicone to protect the fossil and mold against any oils and additives from the wax.
To remove the wax after casting the large pieces were softened with heat and removed. Any remains were removed with scalpel and acetone.

Conclusions

The taphonomy obviously has great influence on the preservation and subsequently also on the degradation of a fossil. It can be worthwhile to be well aware of local taphonomic features and the differences in preservation in different parts of a specimen, and think ahead what future degradation issues it can lead to. This knowledge should be used to act accordingly during handling, preparation, conservation, storage, exhibition and research at all times.
In this case a local carbonate cementation, that otherwise usually provide physical support and chemical buffer action, hid a layer of pyrite oxidation, and had to be removed contrary to common practice.
Careful monitoring of a collection should be part of all preventive conservation as a mean to discover first signs of deterioration -and do look for the unexpected at times!
Pyrite oxidation monitoring should be done very carefully visually and with e.g. pH-tests as well. And, very important, they should also be done beneath surface were fragmentation allows it.
Materials and methods should be chosen and adapted to keep the specimen safe at all time whether it is transported, prepared, molded, done research on, exhibited, etc. The more degraded a specimen the more information loss is usually the outcome.

Perspectives

•The recent pyrite oxidation in mind a minute examination of this particular fossil, both visual, microscopic and chemical, should be done to compare treated (both old and new treatments) and non-treated fossil parts above and below surfaces.
•More needs to be done lowering temperature and RH in exhibition, storage and working area.
•The use of anoxic microenvironments will probably expand to treated fossils in storage.
•The monitoring-program will be revised and severely expanded:
- *Fossils in storage will be included by random sampling.*
- *It will go below fossil surface by using x-rays and by removing surface fragments to perform visual inspection and sampling for pH spot tests.*
- *Improvement of on sight working conditions by adding a mobile work station with table, good light and fume extractor.*
- *Improvement of on sight documentation. Using a tablet taking photos and writing notes directly on them.*
- *Improvement of where and how data is stored for easy access and standard of reference at next inspection.*

•Research needs still to be done on the pyrite oxidation issue. Pyrite and its stability differ a lot from fossil locality to fossil locality. One should be very familiar with the particular type of pyrite in ones care.
•More research is needed on the different oxidation treatments and their long-time effects.

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Acknowledgment

I thank all colleagues at Department of Conservation and Department of Natural History and Paleontology, Museum of Southern Jutland. Especially Mette Steeman for god advice and Benjamin Ramassamy for fruitful comments and discussions.
Parts of the preparation vas funded by Danish Agency for Culture.
Danish Art Workshops and Natural History Museum of Denmark has supported the preparation by supplying workshop facilities.