Mechanical and chemical preparation techniques applied to Frasnian Cephalopods from Lompret (Belgium)

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Received 15 February 2021. Accepted 24 May 2021.

When embarking on a preparation project it is essential to consider a variety of techniques. A combination of different mechanical and chemical treatments may be necessary, even within the same formation. This article explores this principle using a case study of large accumulations of Frasnian cephalopods collected between 2015 and 2021 from the active quarry of Lompret near Chimay (province of Hainaut, Belgium). The quarry comprises strata that can be linked to the Kellwasser event, an important mass-extinction event near the Frasnian–Famennian boundary. Several of the lithological entities from this quarry require specific approaches in terms of preparation. This article will explicitly focus on preparation techniques applied to cephalopods. This informative and diverse group of macro-organisms can contribute to a better understanding of marine environmental changes during an ecological crisis. A thorough preparation of all the collected specimens from this specific location is required, as this peculiar fauna is in desperate need of a taxonomic review. We will demonstrate to what extents the uses of potassium hydroxide (KOH) and Rewoquat® W 3690 PG as solvents have proven to be particularly effective in dissolving clay-rich sediments during preparation.

Hellemond, A., Houben, K., Tolisz, N., Nolis, K., De Bock, F. and Van Uytfanghe, S. 2021. Mechanical and chemical preparation techniques applied to Frasnian cephalopods from Lompret (Belgium). *Geological Curator* **11** (5): 361-374.

Geological framework

In order to understand the environment in which these Frasnian cephalopods were deposited, we should first provide a larger picture of the present-day context in which they are encountered. Looking at the quarry of Lompret (N 50°04'14.0", E 4°23'08.0"), we observe that the lithology is dominated by mud- and limestones from the Late Frasnian (around 372.2 ± 1.6 Ma; Gradstein *et al.* 2020). The oldest deposits belong to the Grands Breux Formation (GBR) consisting of hard limestones that make up the vast majority of the quarry. On top of that, we have the Neuville Formation (NEU) as defined by the National Commission for Stratigraphy of Belgium (NCSB). The NEU consists of dense nodular shales with few inferior nodular and argillaceous limestone beds (*cf.* Tsien 1975; Coen and Coen-Aubert 1976; Bultynck *et al.* 1987; Boulvain *et al.* 1993). These strata were deposited in an open marine environment North of the Rheic Ocean (Gatley 1983; Wynants *et al.* 2018). The layers have been diagenetically transformed during the Variscan orogeny of the Rheno-Hercynian basin (Figure 1A, B), which makes the present outcrop tectonically deformed.

Other important lithological entities include the black shales belonging to the Matagne Formation (MAT). These fine, dark, greenish-brown to pitch-black shales, with generally a few dark limestone beds in the lowermost part, are very recognisable and have been studied for more than 150 years (Gosselet 1871). The black shales are linked to anoxic conditions and were also deposited in deep water (Sartenaer 1974; Mottequin and Poty 2016). The macrofauna consists of small bivalves, brachiopods and cephalopods, often coated with a thin layer of pyrite (Maillieux 1939). This particular formation, located between the Lower and Upper Kellwasser event, is synonymous with a profound change in

Figure 1. Overview of the Lompret quarry in July 2016. The Formations of Neuville and Matagne lie folded against the grey massive limestones of the Grands Breux Formation in the northern part of the quarry.

facies and associated fauna. It marks the transition from a reef-dominated environment to an anoxic setting defined by mass extinctions and ecological turnovers (Mottequin and Poty 2016).

Though the description of both formations provides us with seemingly adequate lithological information, we must bear in mind that a vast spectrum of strata with slightly variable lithological properties can occur within these formations. On the current geological map of Wallonia (57/7-8 - 1:25.000) the difference between the Neuville and Matagne Formations in Lompret is problematic to such an extent that they are grouped together in the "NM complex" (Marion and Barchy 1999). In this framework, additional biostratigraphical controls could help to further distinguish both formations in the future. Macrofossils such as cephalopods could turn out to be a suitable group for stratigraphical differentiation. The faunal variation in cephalopods from Lompret might encourage grouping similar species or morphologically similar specimens together. However, we suggest grouping specimens based on lithological properties, rather than biological (systematic) criteria (Figure 2). This storage method will not only prove helpful during preparation, but will also facilitate future cyclostratigraphic (sequential) research, during which lithological matching is of fundamental importance.

Cephalopod fauna

The Lompret quarry yields large numbers of well-preserved corals, sponges, brachiopods, bivalves, crinoids, conodonts, ostracods, graptolites, trilobites, placoderms, sharks and other classic reef (building) organisms (Houben and Hellemond 2016). In the past five years, a few papers on the well-preserved fauna from the Lompret quarry provided additional insights into the unique faunas of the Matagne and Neuville Formations (Gouwy and Goolaerts 2015; Houben and Hellemond 2016; Houben *et al.* 2020). Cephalopods are among the previously overlooked taxa and are rarely included within (private) collections. Solely based on these collections, they may seem underrepresented in the fossil fauna because they are difficult to distinguish from the often oddly shaped nodular limestone concretions in the field. Around 85 years after the first cephalopod review by Dr. Hans von Matern (1931), a palaeontologist from Frankfurt am Main, new material can be gathered systematically and in large numbers.

The cephalopod fauna from Lompret consists of two important subclasses, the Ammonoidea Zittel, 1884 and Nautiloidea Agassiz, 1847. The Ammonoidea are represented by a few genera of goniatites, of which the genus *Manticoceras* Hyatt, 1884 is by far the most common. The Nautiloidea are represented by a few

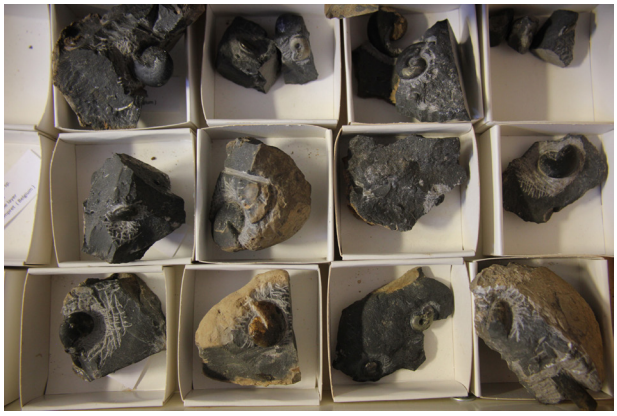

Figure 2. Instead of systematically grouping similar species together, we chose to set up a stratigraphical collection to facilitate future research and help us during the preparation process.

genera of orthocone (straight-coned) organisms belonging to the orders of the Bactritoidea (Shimansky, 1951) and the Orthoceratoidea (Zhuravleva, 1994). Other cyrtochonic (curved) Nautiloidae belong to the order of the Oncocerida (Flower and Kummel Jr., 1950). The observed genera include: *Tornoceras* Hyatt, 1884; *Crickites* Wedekind, 1913; *Manticoceras* Hyatt, 1884; *Trimanticoceras* House, 1977; *Carinoceras* Ljaschenko, 1957; *Beloceras* Hyatt, 1884 and *Bactrites* Sandberger, 1843. Regarding the Belgian Frasnian cephalopod fauna, there is an abundance of outdated literature, resulting in difficult taxonomic identification. The variety and concentration of cephalopods collected over the past five years will hopefully serve as a solid base upon which a refined classification can be established.

The vast concentration and diversity of cephalopods in Lompret, makes them an interesting study subject. Specific layers containing dozens of individuals buried in close proximity to each other can serve as proxies for marking environmental changes (Figure 3). Combined with auxiliary biological markers (both micro- and macrofossils), they also allow for statistical and biostratigraphical analysis to some extent (Korn 1996). Cephalopods inhabited an undisputable part of the Frasnian ecosystem, but in Lompret they remain a fairly understudied group of organisms despite their mass occurrence in specific layers. A careful preparation will help to facilitate future taxonomic studies, revealing certain anatomical details which might otherwise pass unnoticed.

Mechanical preparation

It is important that each specimen is prepared following a procedure suited to the distinct layer or bedding in which it was found. Thus, it is beneficial to physically separate fossils from different strata before undertaking any preparation. Prior to any mechanical or chemical manipulation, one should ensure that every specimen block is cut to a manageable size and cleaned as thoroughly as possible with water. Most of the downsizing should be done in the quarry using a cordless angle (disk) grinder equipped with diamond encrusted discs (Figure 4A). The mechanical preparation of medium- to very hard limestones requires the use of tools, each with their own benefits and weaknesses that need to be taken into account according to the purpose of the preparation (e.g. study, conservation or exhibition). From all available mechanical techniques (air abrasion, pneumatic percussion, manual removal, etc.) we would recommend the use of a traditional pneumatic pen as a first intervention for the reasons listed below. Although air abrasion removes the risks of unwanted fractures due to the vibration we find in pneumatic tools, it nevertheless involves significant risk in removing the very top material of the fossil once matrix elements are no longer in its way. This is especially critical when preparing fossils in which matrix- and specimen hardness are very similar, or when visual distinction between these layers is problematic. Air-scribing, when used properly, has the advantage of splitting matrix off at the very surface of the specimen along its natural separation from surrounding sediment, especially suited to the naturally spiral-shaped features of goniatites. Of course, a possible successive use of first pneumatic then abrasive preparation, if affordable, should be left to the judgement of every preparator (Figure 4B). Here, we used a Wegner (W224) pen, driven by a 25 L (whisper/silent) compressor generating between 7–8 bars of pressure. For the cephalopods of Lompret, we used a maximum of 36.000 bpm to chip away at the hard limestone.

Figure 3. **In situ** ***detail of one of the rich layers containing multiple cephalopods. This entity, informally known as the 'middle limestone layer', was exposed in 2017.***

Most of the cephalopods found within or alongside the nodular concretions, especially the goniatites, have two sides with different modes of preservation. Taphonomic conditions often produce one weathered side, exposed prior to burial, and another, more intact side, covered by the original seafloor sediment (Figure 5A). The soft and mostly eroded side is related to thermal, diagenetic or taphonomical alterations linked to the mudstone (clay-rich) beddings (Figure 5B). Hard carbonate beddings, or nodular concretions in general, assure a better state

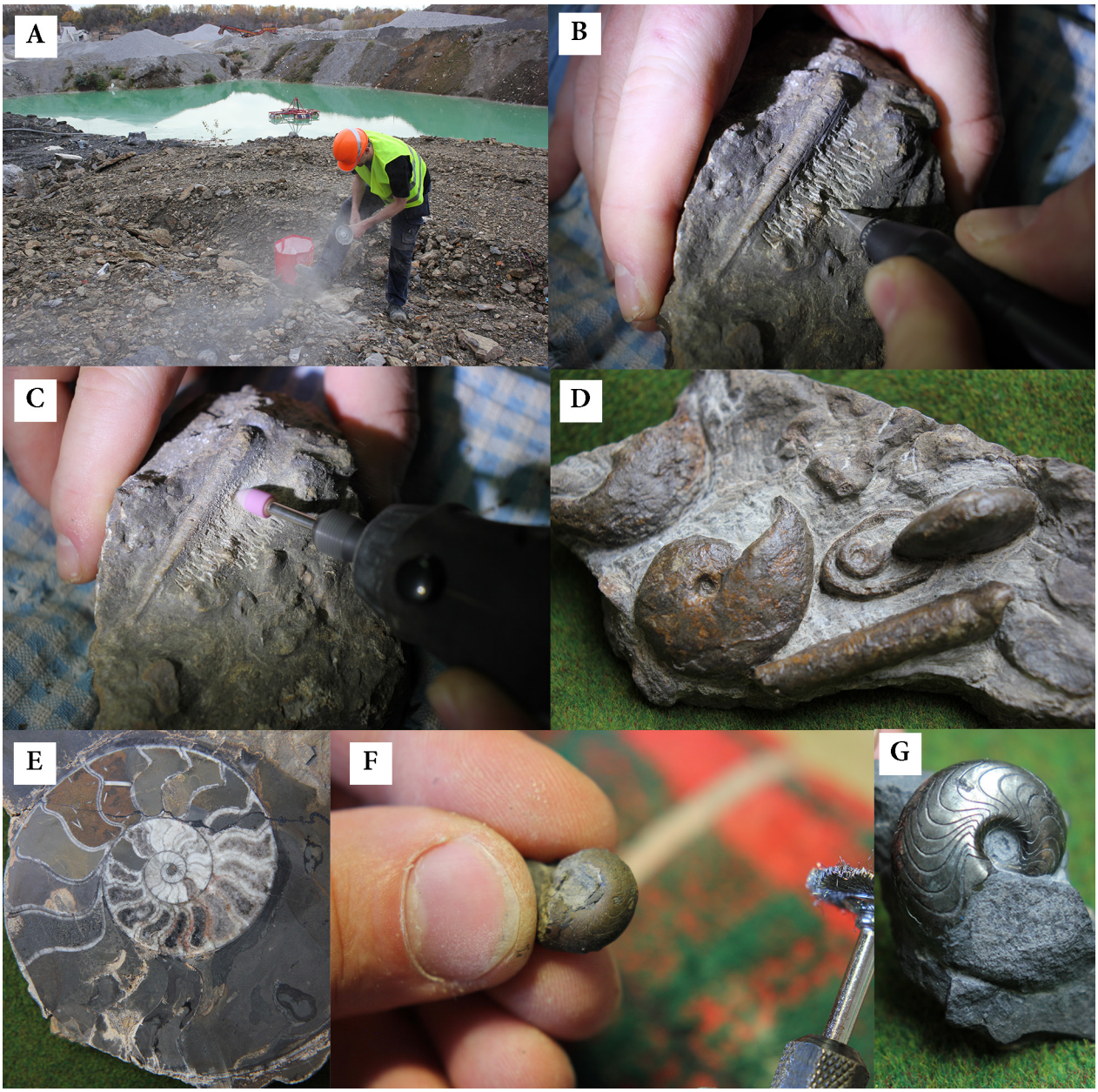

Figure 4. Mechanical preparation. A) In-situ cutting of cephalopods from their original bedding, using a battery powered disk grinder. B) Traditional mechanical preparation using an air scribe with tungsten needle at 36,000 beats per minute. C) Covering up the striations left by the air scribe, using a rotating multi-tool with cylindrical shaped aluminium oxide (Al2O3) grinding stone head. D) Example of a grinded matrix around several cephalopods from the BLL (c. 4 inches). E) A large (c. 6 inches) but broken Manticoceras *sp. goniatite, cut and polished dorso-ventrally to study the anatomical and taphonomical features hidden inside the shell. F) A multi-tool bristle steel brush is used to polish a pyrite coated* Tornoceras *goniatite. G) A fully polished* Tornoceras *sp. goniatite with visible sutures (c. 0.6 inches).*

of preservation. It is therefore tempting to consider only preparing the soft side of each specimen, but since preservation is significantly better on the harder side, it is preferential to prepare that side using an air scribe, as stated above.

The discovery of several cephalopods in close proximity within the same layer does not imply they are found in parallel orientation to the original bedding plane. In particular, the smaller goniatites within the nodules (concretions) are often not found parallel to the original bedding plane. These 'strange positions' often result in breaks through the fossil when cutting the limestone nodule in half. Very rarely, lucky splits result in some remarkably preserved calcified shells (Figure 6). The "unlucky splits", which resulted in broken specimens, were initially glued together using Araldite AW 2101, an irreversible fast-setting

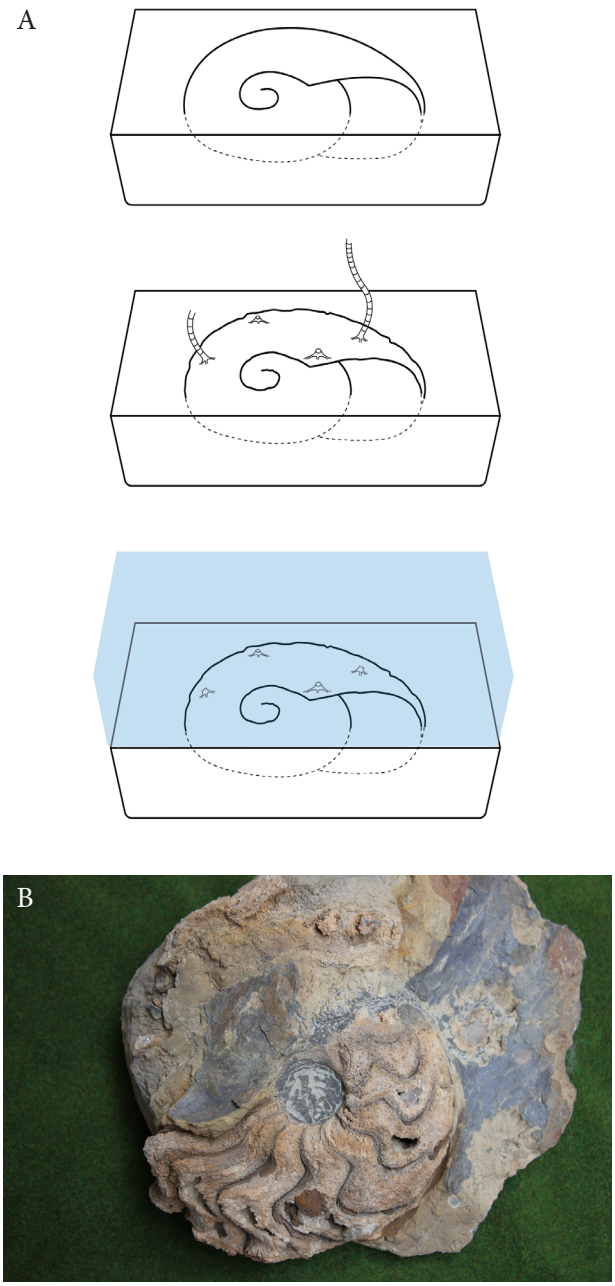

Figure 5. A, Taphonomic process visualising the cause of dissimilar preservation in goniatites from Lompret. B, Backside of a Manticoceras *sp. goniatite. This partially prepared specimen is representative for the state of preservation characterizing the majority of all cephalopods from Lompret.*

epoxy resin with hardener (HW2951), before switching to a reversible transparent adhesive (Locktite SG-3 precision), when the decision was made that this collection should become an important study collection. After adhering the nodules together, we started preparing them with the air scribe.

After carefully finishing all preparations using the air scribe, we removed the striated marks left behind by the pneumatic pen. To do this, we used a Dremel™ multi-tool equipped with a conical or cylindrical aluminium oxide (Al_2O_3) grinding stone head (Figure 4C). The use of this tool should be carefully considered before applying it close to the fossil(s). Alternatively, a Chicago Pneumatic CP9361 air scribe can be used in a circular motion, which allows for better control. The use of an air abrasion tool might also be considered in this case, but this was not used due to budget restrictions. Grinding away the surrounding matrix results in a smooth and polished look (Figure 4D). This might be advantageous because it makes the fossil stand out from its supporting bed, but consequently also alters the original look and lithological texture of the matrix. Without the proper accompanying documentation describing the original lithology, a visual link to the original matrix or stratigraphical entity will become difficult. The absence of any visual access to the original matrix on all specimens may highly hinder specific types of future research.

In some cases, the carbonate cement of the limestone (micrite) is tightly bound to the fossil, making separation difficult during preparation. In our

Figure 6. A nice example of a Manticoceras *sp., which was fortunate enough to come out of its concretion whole. Diameter: 9 cm.*

case, we noted that this was the case with goniatites from certain beds. This poor separation sometimes concealed a bad state of preservation. If this was the case, we would no longer try to preserve the fossil in its initial state but opted for a more dissective approach by adhering any displaced parts together and cutting the assembled cephalopod in half dorso-ventrally. This granted us visual access to the specimen's inner features (Figure 4E). Especially for the orthocones, this could help us to determine the orientation of the siphuncle, which is taxonomically informative. In order to observe the position of the siphuncle, it is recommended to cut the orthocones diagonally. The cutting of the cephalopods was done with the aid of a water-cooled table-top (bridge) saw with a diamond blade 35 cm in diameter. Only 10% of the total amount of collected cephalopods were cut in this manner as a result of poor preservation of the outer shells. Non-destructive methods such as (micro)CT scanning might achieve similar results, but given the mass occurrence of cephalopods in this deposit, we opted for physical separation.

Optional mechanical interventions to further reveal visual features such as the use of brass or steel brushes have shown some satisfactory results on pyrite-coated goniatites from the Matagne formation, although one could object that gentle air abrasion might be a less intrusive and lower risk equivalent. A simple manual brass brush was used to brush delicate specimens, but for larger specimens we used a Dremel™ multi-tool ½" (12.7 mm) bristle steel brush to superficially polish the pyrite-coated goniatites. This (temporarily) accentuated the gold colour and septa of the cephalopods for photographic purposes (Figure 4F-G). When using the bristle steel brush, we advise wearing safety goggles and, if possible, pre-setting the base of the brush in resin (where it connects to the hub). This way, ejected strands are prevented from flying off during brushing. The efficiency of the costlier air abrasion techniques on the Lompret cephalopods will be assessed in future preparation projects.

Chemical preparation

Keeping in mind that most of the cephalopods, especially the goniatites, are difficult to spot in the field, a chemical preparation can help to accentuate fossils from their matrix. In the course of the past five years, different chemical compounds have been tried for this purpose. Here we would like to emphasize that a detailed record of all applied chemical products and preparation techniques should be logged in the collection database under each specimen number. Traces of certain molecules will show up in future geochemical research and might interfere with the scope of an ulterior restoration or future investigation.

Rewoquat®

A popular and relatively modern product used for chemical preparation is Rewoquat® W 3690 PG (Jarochowska *et al.* 2013). This chemical compound was initially used as an industrial fabric softener (Krüger 1994), but its value has been recognised as a powerful agent for fossil preparation since the 1980s (Riegraf 1985). The use of Rewoquat® has also been popular in Germany, where it was used as a product to dissolve marly and clay-rich sediments (Lierl 1992). Over the years it established a solid use amongst fossil collectors, and different approaches and techniques using Rewoquat® can be found on online fora and regional paleontological journals. The product has since become a household name in paleontological preparation and can be bought in pre-made solutions, distributed by shops specialising in fossil preparation materials.

Rewoquat® W 3690 PG is a 1-methyl-2-noroleyl-3-oleic acid amidoethyl imidazolinium methosulfate with 24% polyglycol and a pH ranging between 4.0–5.5. The original formula works as a cationic hydrophilic softener. It is viscous and has a distinct yellow, transparent colour. It is useful as a coupler and co-emulsifier for cationic formulations. Within the framework of fossil preparation, it is commonly sold as a 5% solution in isopropyl alcohol (IPA - 2-propanol). The solution works as a surfactant, and it can be re-used several times. We strongly advise reading the Material Safety Data Sheet (MSDS) before using it. We would also like to specifically point out that the imidazole component is highly toxic and corrosive.

The use of Rewoquat® on the calcified mudstones of Lompret was highly effective. Promising results have already been demonstrated on several taxa of Silurian and Devonian microfossils, where it proved an excellent and fast working solvent for phyllosilicate minerals compared to caustic potash (Jarochowska *et al.* 2013). Instead of erasing important morphological and anatomical features, Rewoquat® seems to spare the often weathered and vulnerable cephalo-

pod shells and their associated epibiont fauna, such as crinoid-anchoring parts (holdfasts) and corals (Figure 7).

Our method of applying Rewoquat® to the cephalopods of Lompret is fairly straightforward. We first started by placing the mechanically-prepared specimen in a sealable container. We then applied the Rewoquat® on the clay-rich surface of the cephalopod with a paintbrush, or poured Rewoquat® at the base of the recipient. We subsequently placed the cephalopod in the box and, depending on its morphology or lithological properties, decided to submerge it fully or only face down on the side being treated (Figure 8A). We also applied Rewoquat® on any required area by using a small brush, syringe or transfer beral pipette (Figure 8B). This reduced the amount of Rewoquat used. This treatment should be performed in a ventilated space at normal room temperature or, as recommended by safety standards, under a closed chemical fume hood. After closing the box, we let specimens rest for 5–7 days, monitoring the process on a daily basis. On the last day, we carefully removed the Rewoquat® from the specimen and transferred the fossil to a tray where it was rinsed with isopropyl alcohol for 7 days (Figure 8C). In the second stage of the rinsing process, we washed our specimens with warm water and a toothbrush. As a surfactant, Rewoquat® can easily be reused, so we filtered the leftover product from the box with several sieves or a separating funnel to save for a second application (Figure 8D). The mixture of leftover Rewoquat®, isopropyl alcohol, water and dissolved sediment was collected and stored in a closed jar. The jar could be disposed of in a chemical waste container.

Potassium hydroxide

Potassium hydroxide (KOH), or caustic potash, is a strong base frequently used in fossil preparation. The characteristic white flakes have a pH ranging between 10–13 and are widely available. The use of KOH requires a series of precautions prior to any handling. We strongly recommend reading and carefully following the MSDS instructions before attempting any preparation. Nitrile disposable gloves, tweezers and safety goggles are mandatory, as well as protective clothing and a safe working place under a fume hood. The violent reaction of KOH with water can cause severe skin and respiratory irritations. Potassium hydroxide should therefore be stored in a controlled environment free of water, metal and acids. Its corrosive nature and heat generation during a reaction can cause glassware to break and will react with H_2O particles in the air.

Figure 7. Some of the typical epibionts we encounter on the cephalopods illustrate that they served as a basis upon which other organisms could grow for some time.

The majority of the large cephalopods from Lompret were treated with 99% KOH flakes (not pellets). For safety reasons, we worked inside a PVC (polyvinyl chloride) container which could be closed. Within the container, a PVC bag served as a reaction vessel in which to place the specimen, with the side requiring treatment facing upwards. We used a spray bottle with a pump atomiser to moisten the surface of the specimen (Figure 9A). Next, we carefully placed the KOH flakes on the wet surface with a pair of PVC-coated tweezers (Figure 9B). We recommend that areas with more matrix receive more KOH flakes. Once the surface was sufficiently covered in flakes, we used our spray bottle to moisten the KOH flakes. One should avoid aiming directly at the flakes, but rather spray just above them, allowing the dispersed water particles to gently mist down on the KOH (Figure 9C). We advise then closing both the PVC bag and the container. Keeping the fossil and chemicals contained at room temperature is saf-

Figure 8. Chemical preparation – Rewoquat. A) Submerging a small specimen in a jar filled with Rewoquat® W 3690 PG. We let our specimen rest between 5-7 days. Plate 2B: Applying Rewoquat® with the help of a transfer (beral) pipette. C) After the Rewoquat® treatment, we rinse our specimen with 2-propanol (isopropyl alcohol) for 1-2 days and afterwards wash it with water and a pH neutral detergent. D) Re-using the used Rewoquat® through a sieve and a separating funnel. E) Using a 1:1 linseed oil and turpentine solution to deepen the contrast of a goniatite. F) (left) The goniatite treated with linseed oil and turpentine (right) An orthocone treated with a polyvinyl acetate (Paraloid B72). Notice the reflections that occur as a result of the treatment. A coating with Butvar B-76 might be a better alternative against the reflection.

er and will greatly accelerate their reaction time. We recommend checking the contents of the bag two hours into the process. When the water from the spray and present in the pores of the matrix breaks the KOH ion bond, the solvated ions (K^+ and OH^-) endothermically react within their aqueous environment (1). This may cause the flakes to move during the reaction (Figure 9D), so we advise repositioning the displaced flakes using a pair of tweezers or adding additional flakes after two hours.

$$KOH\ (s) \longrightarrow K^+\ (aq) + OH^-\ (aq)$$

Potassium hydroxide (KOH) will break the ion bond when confronted with H_2O, resulting in an aqueous potassium ion and an aqueous hydroxide ion.

This procedure works particularly well for goniatites, whose relatively flat shells act like a table upon which the KOH can be placed. For conical fossils, such as our orthocones, the positioning of the flakes (and keeping them in place) can be more difficult. We used vacuum seal bags as described by Vercammen (2020). These transparent bags allowed us to monitor the position of the flakes and equally distribute them across the surface (Figure 9E). The time needed to complete a KOH treatment varies for each specimen; we suggest monitoring the treatment every 4–6 hours.

After leaving the fossils in their bags and containers overnight, we then started carefully brushing off the dissolved sediment. One should use large amounts of water to rinse the fossils. Brushes should be of plastic (polymers), not metal. During the cleaning, always wear safety goggles and protective clothing and make sure to protect your skin and face from ejected droplets at all times. Following the safety guidelines, both

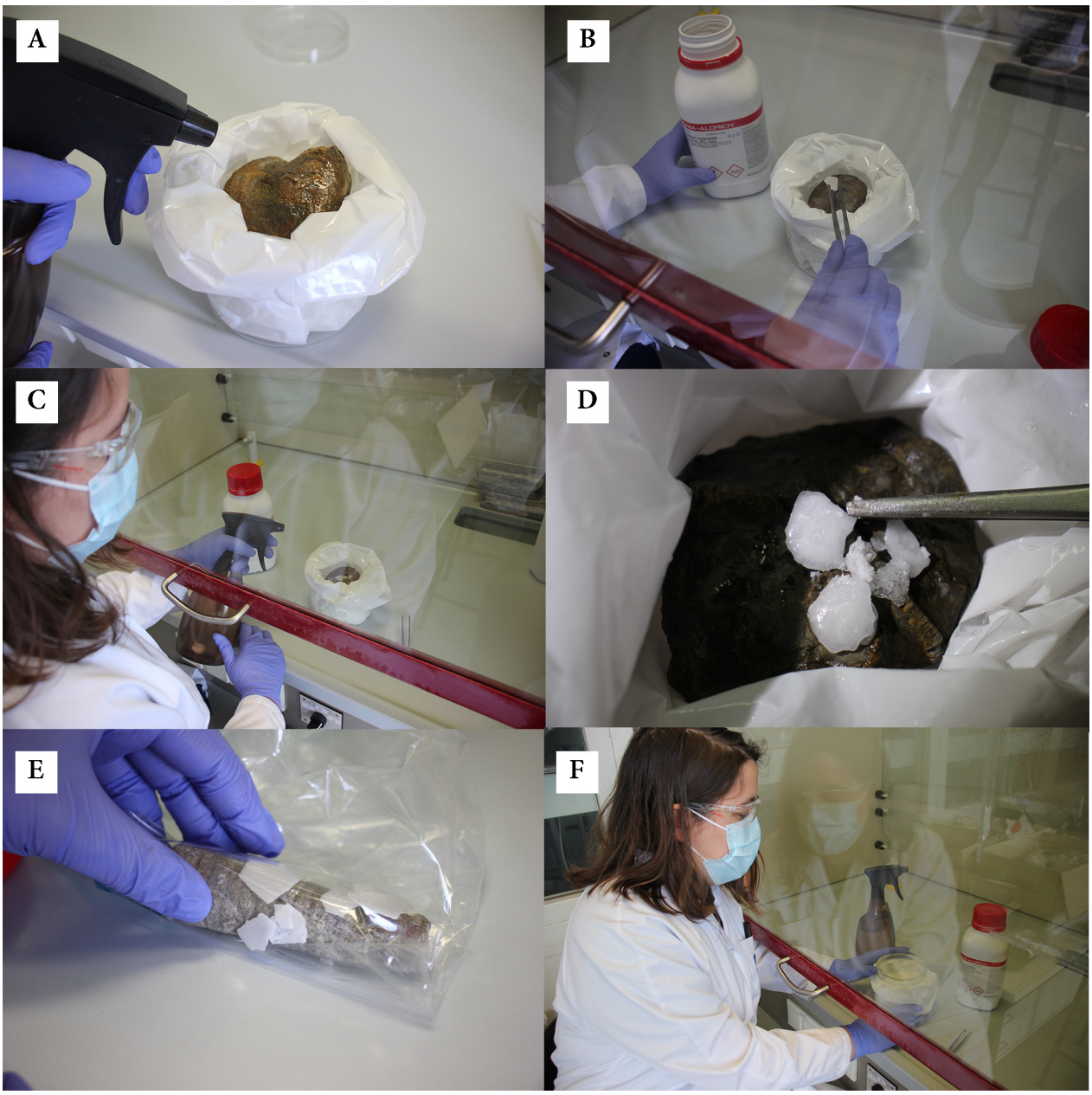

Figure 9. Chemical preparation – potassium hydroxide (KOH). A) Moisturising the specimen within a PVC bag on top of a Pyrex® jar. B) Carefully placing potassium hydroxide (KOH) flakes on the specimen using a pair of tweezers. Plate 3C: Spraying water above the specimen, allowing the mist to gently drizzle over the flakes. D) 2 hours into the preparation, we check our reaction vessel to see replace the KOH flakes who moved during the chemical reaction. E) Preparing a three-dimensional orthocone by using a vacuum seal bag. F) Safety clothing and precautions used when working with potassium hydroxide under a fume hood.

the treatment and the rinsing should be performed under a fume hood (Figure 9F). We submerged the brushed specimens in water that was replaced every 2–3 hours over a 12-hour span in order prevent any remaining KOH from reacting with the fossil in the future.

Although it may be tempting to neutralise KOH with mild acids like vinegar (5–8% acetic acid solution) or HCl (hydrochloric acid), we strongly discourage the use of acids for neutralising strong bases, even if they are diluted. Occurring reactions could result in the formation of orthosilicic acid, which would permanently damage. This also applies when using excessive amounts of KOH on your specimen, resulting in a white grey patina. Always make sure to use plenty of water to rinse your fossils after treatment. The residual KOH and sediment solution should be heavily diluted and disposed of in a chemical waste container.

Stone deepener and Paraloid® B72

Certain industrial products called 'stone deepeners' are designed to be used on polished stones and tiles or to enhance their colour and appearance. They can also be applied on fossils for photographic purposes, increasing the visibility of certain anatomical details. For mineralogical specimens, linseed oil is often used as a biological alternative to remove unwanted scratches or deepen the colour of specimens. On the calcified cephalopods from Lompret, this could also be applied to intensify the white calcified septa of certain specimens.

We used a commercial stone deepener, HMK S748 Stain Protection - Premium Color (made by the German company Moeller; Möller-Chemie GmbH), on some of our cephalopods. This solvent-based oleophobic impregnator is biodegradable and easily absorbed by the cephalopods from Lompret (Figure 8E). As the exact composition of this product is not known to us, we recommend applying it only to specimens whose sole purpose is photographic or educational display. Moreover, it contains highly flammable silane, silicone and unspecified petroleum derivatives, which should never be used in combination with a KOH treatment. In spite of the positive aesthetic results in this case, we advise against the use of stone deepener as a conservational practice. For enhancing the colours on the specimens, we first suggest experimenting with modern imaging techniques before resorting to stone deepeners.

To coat the small pyritised gephuroceratid cephalopods from the Matagne Formation, we used Paraloid® B72. Paraloid® is an acrylic resin based on methacrylate-ethyl methacrylate, applied in a 15% solution with acetone. This coating helps protect the specimen from oxygen and moisture in the atmosphere, reducing oxidation and possible pyrite decay. We left a number of specimens uncoated in order to monitor whether pyrite turns out to be unstable over time; thus far the pyrite on the uncoated specimens has not changed. The Paraloid® acrylate serves two purposes: first, it consolidates fragile suture lines and prevents chambers from falling apart. It also acts as a stone deepener, accentuating the calcified shell of our cephalopods. Preparators should decide whether this serves the intervention's purposes, as it also covers the specimen with a thick and reflective coating (Figure 8F).

A lithological approach

States of preservation of the cephalopod fauna varies widely across the Neuville and Matagne Formations (NM) in the quarry. The following overview will focus on specific strata exposed in the quarry, as well as provide an overview of the cephalopod faunae and respective preparation techniques we recommend. The names used here are informal and have been applied to different strata within the grouped Matagne-Neuville Formation outcrop over the years. They should not be regarded as part of any official lithostratigraphical classification recognised by the National Commission for Stratigraphy Belgium (NCSB).

Black 'anoxic' shales

The strata on the northern part of the quarry are dominated by black anoxic shales (mudstones), which are classified as the Matagne Formation (Wynants *et al.* 2018). Within this formation, there are some nodular concretions that contain small pyritized ammonoids belonging to the genera T*ornoceras* Hyatt, 1884; *Crickites* Wedekind, 1913; *Manticoceras* Hyatt, 1884 and *Bactrites* Sandberger, 1843. Preparing them may require magnification, as the diameter of some genera does not exceed 2.5 cm. In the field, we applied a primary coat of Paraloid® B72 to secure fragile specimens for transport. The small, pyritised cephalopods from the black shales are generally covered in softer mudstone (shale) which

can be removed mechanically with an air scribe or scraper hand tool. A final clean-up with a Dremel™ multi-tool equipped with a soft steel brush produces excellent results and brings out the shiny pyrite coating. Depending on the fragility of the specimen, we applied Paraloid® B72 or Mowilith® (a polyvinyl acetate) in an attempt to reduce the potential for oxidation of the pyrite coating. Butvar® B-76 was not used but might also be an appropriate alternative, as it is more resistant to warmer storage conditions and is not as reflective as Paraloid® B72.

The black anoxic shales also contain many small fossils, such as anaptychi (Figure 10). We also found large nodules with cephalopods up to 41 cm in diameter. The only way to begin preparing these large nodules is with hammer and chisel. Next, mechanical preparation can be carried out using a pneumatic pen (air scribe). The relative hardness of these concretions did not obstruct the mechanical separation of the fossil from its matrix and gave satisfactory results.

Bottom limestone layer (BLL)

This particular stratum is around 5 cm thick and encloses a considerable number of juvenile cephalopods. We predominantly observed orthocones and small goniatites mostly less than 2.5 cm in diameter. Similar to previous layers, we also found most of the specimens preserved on the top of this layer covered in claystone. The preservation was often poor, but, during preparation, favourable results were obtained by using the air scribe to remove the limestone. A final treatment with Rewoquat® also proved successful at removing excess claystone. From a taphonomic perspective, the BLL is an interesting case-study on the mass mortality of juvenile individuals.

Middle limestone layer (MLL)

This particular layer yielded an important concentration of large goniatites. The MLL has a thickness of approximately 7.5 cm and contains adult goniatites of the genus *Manticoceras*. Some of these specimens can reach a diameter of 13 cm. Many of the cephalopods found on the top of this layer are covered in (calcareous) mudstone. The preparation of this mudstone is quite straightforward and can easily be achieved with an air scribe and finished with a chemical treatment of Rewoquat® W 3690 PG. Unfortunately, the majority of the cephalopods in this layer are often heavily eroded on one side.

On the solid limestone side, we observed that the cephalopod shells had often experienced intense recrystallisation, making it difficult to separate the fossil shell from the hard matrix. During manual preparation, this resulted many broken specimens. Some of these specimens could not be further prepared, so we decided to glue them back together, cut them in half and polish them to reveal their inner anatomical features.

Top limestone layer (TLL)

The TLL is one of the more understudied cephalopod-bearing layers. This thick limestone bed (20–25 cm) near the MLL was almost impossible to take apart with traditional field equipment. The few collected specimens were mostly found ex situ after explosives were used to blast this layer. We found that most of the specimens in this layer were entirely covered in limestone (micrite).

Other strata

Throughout the rest of the quarry we observed cephalopods in the grey-blue limestones of the Grands Breux Formation. Most of these were cross-sections embedded in massive limestone boulders that were nearly impossible to remove. Compared to the Neuville and Matagne Formation, their presence in the Grands Breux Formation is rare. As a result of their heavy compaction, we were not able to retrieve any three-dimensional specimens (Figure 11).

Figure 10. Fossilised anaptychi are often overlooked as part of the cephalopod fauna. Their often bivalve-like appearance leaves them neglected in the field.

Discussion

Comparing the previously discussed solvents, no single option gave decisive results. Based on our experience within the framework of the cephalopod fauna from Lompret, we feel that both products have their own advantages and disadvantages. As we have adopted a very individual approach for each specimen, we have chosen to give an overview of both products based on their different properties (*cf.* Table 1). We hope that this allows our colleagues to experiment more confidently with both solvents, thus reducing the risk of damaging or irreversibly altering the chemical composition of the specimens.

We did not use both products on the same specimen in this study, because we obtained satisfactory results with a combination of mechanical and chemical preparatory methods, as mentioned above. In addition, we do not recommend combining several chemical compounds, as they may interfere with each other if the rinsing phase is not performed properly. Our advice for potential experimentations combining both products would be to focus on an extensive rinsing phase and allow for a sufficient time lapse of at least a few weeks between the use of both products.

Conclusions

When dealing with the preparation of fossils, it is of primary importance to examine the matrix surrounding the specimen(s). Trained preparators value a preliminary assessment of the involved lithologies by first submitting sterile fragments to chemical or mechanical preparation and/or conservation techniques. They will then monitor and keep a record of variations in order to choose the most appropriate technique, depending on whether the specimen needs to be sampled, anatomically exposed or preserved. Prior to any kind of preparation, a thorough knowledge of the physical, chemical or mineralogical properties of a matrix will undoubtedly reduce errors and save time spent on the preparation of the specimen.

Over a five-year span, approximately 450 cephalopods were collected from different strata within the Neuville and Matagne Formations of the Lompret quarry. Most specimens possess a (calcareous) mudstone side and a hard limestone side. The latter offers a challenge for the preparation of the cephalopods. It is important to adopt an individual approach for each specimen in order to obtain the best results. A combination of both mechanical and chemical preparatory methods is recommended, especially for the larger cephalopods.

90% of all cephalopods from Lompret survived three-dimensional preparation without breaking. The remaining 10% of specimens were adhered together and used for cross sectional study by cutting them in half and polishing the cut surface. In doing this, we rehabilitated partially damaged finds into useful specimens for future research or educational display, allowing for the observation of anatomical details and the identification of internal diagnostic features.

Our mechanical preparation techniques were quite traditional, involving a pneumatic pen (air scribe) and reversible adhesives. Sandblasting was not applied in this setting but will be the focus of future projects. For chemical treatments, we would, before all else, recommend the use of Rewoquat® W 3690 PG and only a switch to potassium hydroxide if results with previous chemicals are not satisfactory. Additional use of (semi-)permanent stone deepeners or linseed oil are not mandatory but can enhance certain anatomical features for exhibition purposes. However, in the framework of scientific research we advise using polynomial texture mapping to enhance the contrast of digital photographs, rather than applying physical coatings to specimens (Hammer and Spocova 2013). The untreated and pyrite coated cephalopods have proved fairly stable over the course of five years, but continuous monitoring will be necessary. The same goes for the storage conditions, for which pyrite coated specimens were packed in acid-free paper to avoid acid aerosols

Figure 11. Cross-section of an unidentified orthocone embedded in the compact limestone of the Grand Breux Formation.

	Potassium hydroxide (KOH)	Rewoquat® W 3690 PG
Price	Low	Medium-high
Re-usability	None	Yes
Reactivity	Very high	Medium
Toxicity	Very high	Medium-high
Corrosiveness	High	Medium
Efficiency	Aggressive	Good
Preparation time	3 days	4–5 days
Not Compatible with	Stone deepener	NA
Dissolves the fossil and/or epibionts	Sometimes*	No
Use of water / acetone for rinsing	High	Medium
Safety material requirements	High	Some

Table 1: Comparing different features of Caustic potash (KOH) and Rewoquat® W 3690 as compounds in chemical preparation. *Only when the concentration exceeds more than 1 flake per cm²

from interacting with other specimens. Future X-ray micro-computed tomography analysis, following the method described by Allington-Jones *et al.* (2020), might help us determine if the delicate pyrite-coated cephalopods or the rare and fragile anaptychi present any signs of pyrite decay in the long term under certain conditions.

In 2022, a public exhibition at the Musée du Marbre in Rance (Sivry, Province of Hainaut) will display a large part of the cephalopod collection from the Lompret quarry within a geological, mineralogical and palaeoecological framework. This public outreach program will help increase awareness of the Lompret cephalopod fauna as an important paleontological study collection.

Acknowledgements

We would like to thank quarry supervisor Mr. Jean Bechet of the '*Carrières de Lompret* sa.' company for his wholehearted collaboration over the years. The access he has granted us on a regular basis has proven essential for our research. We would also like to thank the two anonymous reviewers and Dr. Ayla Debaekeleer (VUB-Brussels) for reviewing this paper. Lastly, our gratitude goes out to the Geological Curators' Group of the Geological Society of London for hosting last year's Symposium on Palaeontological Preparation and Conservation (SPPC), where we had the opportunity to present our preliminary work.

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