Swiss Journal of Palaeontology

REPORT Open Access

A report on palaeontological excavations and sampling in mudrocks: some guidelines

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Abstract

More than 60% of the world's sedimentary rocks are mudrocks (Potter et al., 1980; Schieber, 1998; Potter, 2003; the term mudrock is favored here over mudstone because the latter term was used to characterize a limestone texture; Dunham, 1962). From a palaeontological perspective these are, compared to sandstones and limestones, heavily undersampled. The main reason for this is that mudrocks decay in surface exposures to small chips, which develop with sun/heat and rain into an awkward pulp. The decay of the mudstone concomitantly destroys all macrofossils which are not durable. This comprises fossils with an aragonitic or delicate calcitic shell unless they are preserved as pyritic or internal calcitic molds or preserved within calcareous concretions. Therefore, most of the fossils are not recorded in surface exposures. In addition, sedimentologic investigations of mudrocks are hampered because (i) compaction makes sedimentary structures hardly recognizableand (ii) good thin sections of mudrocks are exceedingly difficult to manufacture. For micropalaeontological investigations, mudrocks rich in organic material are especially difficult to process. Standard treatments with boiling water, sodium carbonate solution, or peroxide H_2O_2 generally fail to dissolve much of the sediment so that the fine fraction (and in mudrocks we usually need the 63µm-fraction) largely consists of clay particle aggregates. Yet there are methods to dissolve these aggregates. Otherwise, picking the microfossils would become extremely laborious. In this paper, some guidelines for successful palaeontological work in mudrocks are outlined. These are based on the author's personal experience. Examples from Jurassic mudrocks of Switzerland/Europe show that such excavations can be very rewarding.

Keywords Exceptional preservation, Systematic excavations, Conservation of macrofossils, Thin sections, Microfossil preparation

Introduction

Palaeontological investigations of mudrocks have a big potential. Mudrocks are notoriously under-sampled, and it is therefore not surprising that new and hitherto unknown fossil finds can be made with thorough surveys. Especially mudrocks with high content of organic carbon may show excellent fossil preservation that is reminiscent of the pattern in famous black shales like the Posidonia Shale Lagerstätte (Seilacher, 1990; Urlichs et al., 1994). This is illustrated here with examples from the Lower Jurassic Schambelen Member (Hettangian, northern Switzerland; Fig. 1) and the Middle Jurassic Opalinuston Formation (Aalenian, northern Switzerland; Fig. 2).

Yet this big palaeontological potential can only be retrieved with adequate sampling methods and elaborate preparation and conservation techniques. This will be outlined in detail in the following.

Getting the fresh rock

When comparing the fossil yield from surface sampling in mudrocks and shales with that from, for instance calcareous marls, it becomes obvious that the results from mudrocks/shales are disappointing. The sediment decayed on the surface to small chips (Fig. 3) Fossils

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appear to be almost absent with the exception of some internal molds ("steinkerns") or fossils being preserved in concretions. Grayish bioturbated mudrocks contain some organic matter (e.g., Berner, 1981; Myrow, 1990) but accumulated under aerobic/oxic conditions. In this case, oxic oxidation of organic matter takes place that produces CO_2 that in turn forsters the dissolution of carbonate (e.g., Morse et al., 2007). Aragonitic shells are mostly dissolved even in dark, laminated mudrocks, leaving only molds and perhaps the periostracum of mollusks. Delicate calcitic, and, if present, aragonitic, shells are usually broken into small fragments. The notion of "barren" mudrocks and shales is quite often only the consequence of inadequate sampling (e.g., Simões et al., 2016). Massive shells cannot be expected in mudrocks and shales as these would have sunk into the soupy substrate (e.g., Wignall, 1993).

While there is some literature about the sampling design (random versus systematic; Krumbein, 1965; resampling methods; Kowalewsky & Novack-Gottshall, 2010), there are only sparse references on collecting techniques (e.g., Fisher, 1965) and techniques specific to mudrocks are not covered. Giving here some guidelines seems therefore appropriate. Collecting/sampling fossils in mudrocks requires systematic excavations in which the topmost weathered layers of the mudstone are removed. Then the now exposed fresh rock can be systematically quarried and the fossils be sampled in a quantitative manner. Most mudrocks are not very rich in macrofossils, therefore quarrying bed by bed on an area of 1–2 m^2 is recommended.

Systematic excavations are of course most easily done in a still active clay pit or quarry, and sometimes only a few centimeters of decayed mudstone need to be removed. Yet such systematic excavations can theoretically be undertaken anywhere. But depending on the weathering, the plant cover, and the penetration depth of plant roots (see Gregory, 2022), this can mean removing well over one meter of decayed sediment material (Fig. 4).

The fresh mudstone underneath is in most cases quite stiff and hard. Heavy tools (large chisels, crowbar, and heavy hammers) will be needed for its quarrying. If the mudstone is homogeneous and does not show natural bedding, it is strongly advised to quarry the rock in arbitrarily defined beds of equal thickness such as 15–20 cm (Fig. 4). Thickness and all observable sedimentologic features should be recorded. For thin sections, geochemistry, x-ray investigation (e.g. micro- and cryptobioturbation; Pemberton et al., 2008), and microfossil processing, raw samples are collected that must be kept moist (see below).

Sampling and recording the fossils

Blocks of the 15–20 cm-thick bed can now be removed and then split along the bedding planes as fine as is technically possible. This minimum splitting thickness varies according to the fissility of the rock but appears to be in most instances somewhere around 0.5–1.0 cm. Splitting is done with hammers and broad flat chisels (Fig. 5) or palette-knifes/putty-knifes in which the handle is internally reinforced by steel.

The split surfaces are now closely inspected by the naked eye, the smaller objects with a hand lens. For every bed, all the occurring macrofossils including trace fossils are recorded. For as yet undetermined fossils, sketches and photos are helpful. Along with the information about abundances, the size and preservation of the fossils should be recorded as well. Patterns of reorientation must be measured with the compass but appear to be not common in mudrocks and largely confined to intercalated more durable silt- and sand-beds.

A selection of the best, most representative and not yet identified fossils will have to be wrapped and transported to the lab. It is now of utmost importance that these fossils as well as the raw samples collected never dry out. The following procedure has proven to be most effective: wrap the slab/sample in newspaper, wrap around an adhesive stripe, label it with waterproof marker, then soak it in a bucket of water, and put it in a large impermeable plastic bag. A normal trash bag will do.

When a bag contains enough wet samples, it is sealed and labelled. These bags can now be transported to the lab where they can remain untouched for several months, unless they are leaking in which case the samples have to be sealed in a new bag.

Preparation and conservation of the macrofossils

The first step upon unwrapping the fossil slabs is washing. This can be done under a soft shower, and soft brushes might be used to clean a slab but all of this must

(See figure on next page.)

Fig. 1 Exceptional preservation of fossils in mudrocks. Examples from the Lower Jurassic (Hettangian) Schambelen Member of northern Switzerland. **a** Compressed ammonites (*Laqueoceras laqueous* (Quenstedt, 1856)) with the shell dissolved but the organic periostracum preserved; **b** small compressed ammonite (*Psiloceras* sp.) with a preservation as in a; **c** ammonite (*Waehneroceras* sp.), preservation as in a; **d** organic anaptychus; **e**: organic lower jaw of a cephalopod; **f** beetle; **g** forewings (elytra) of a beetle; **h** echinoids (*Diademopsis heeri* Merian in Desor 1858) with articulated spines; **i** ophiuroid (*Ophioderma escheri* Heer 1865)

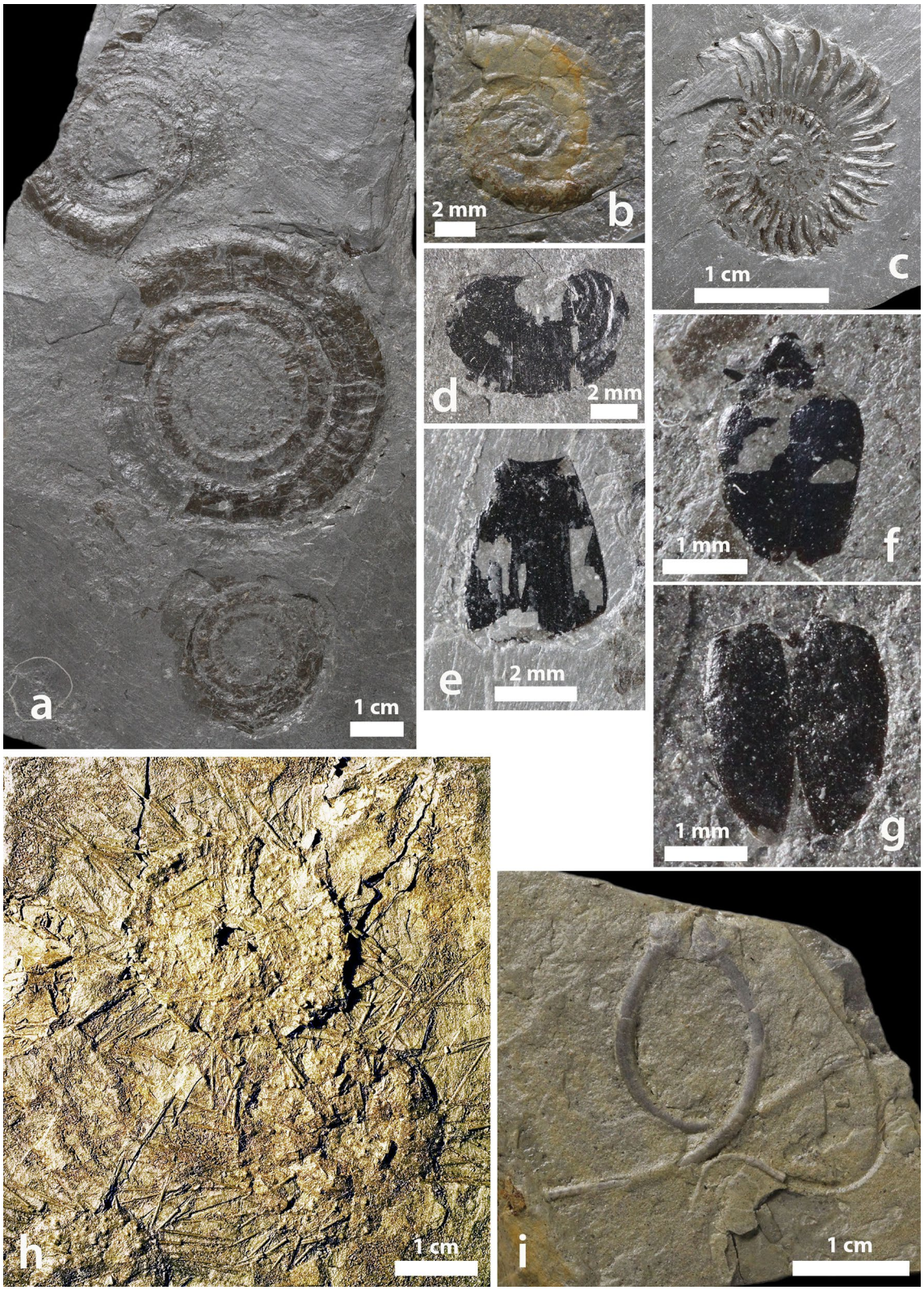

Fig. 1 (See legend on previous page.)

be done carefully. Note, however, that when a slab has incidentally dried out, it should not be washed because that would result in the disintegration of the slab and the fossils.

After washing, the specimens are allowed to dry surficially for a short time. But preparation of the fossils is best done when the slabs are still moist (Fig. 6). In this condition the rock is still quite soft and not as splintery as in the dry condition. Preparation is done mechanically with small chisels and sharpened needles, when necessary, under a binocular lens.

In this condition, the slabs can also be formatted: thinned with knifes or putty-knifes and reduced in size with nipper pliers.

For the final conservation of the fossils/the slabs it has proven best to let them surficially dry for another 1–2 h. This duration depends strongly on the humidity of the air in the lab you are working in. After that time, a thin varnish is applied to all the surfaces of the slab and the fossil, and the specimens are put on a coarse metal grid for drying. The varnish does not completely seal the rock, but the still remaining moisture in the rock can then diffuse slowly out of the slab.

Best results were obtained with zapon varnish (=cellulose varnish) diluted with acetone in the proportion of 1:1 (Fig. 7). This gives the rock an appearance similar to the one in fresh moist condition, and it is also removable again with acetone, in case further preparation becomes necessary. Slabs treated in this manner proved to be much more stable than untreated slabs, and they even survive an accidental drop of water without damage.

The raw samples are of course not treated with varnish. Those destined for geochemistry and microfossil preparation are washed, dried, and stored in plastic bags or containers. Those which will be used for thin sections are kept moist.

Manufacturing thin sections

Manufacturing thin sections of mudrocks is difficult because argillaceous rocks, once dried, react with water destroying the sample surface and ultimately turn the sample into a useless pulpy chunk. The processing comprises the following steps:

- A moist prism (standard size 48×26 mm or larger; Lazar et al., 2015) is cut using a stone saw with diamond blade.
- This sediment slab is carefully dried.
- A large glass slide is grinded with F600 grit (grit size 10 µm) and then dried. After this, all successive steps must be carried out under entirely dry conditions.
- One face of the dried sediment slab is dry polished using very fine abrasive paper (P1500).
- The sediment sample is then glued onto the glass slide using a standard epoxy resin.
- With a scroll saw with diamond blade, the sediment is cut dry to a thin slab.
- The sample is then dry polished by hand on ever finer sand paper/abrasive paper, up to P1500, to a standard thickness of 30 µm or less, constantly examining the result. Make sure that before changing to a finer grit the thin section is thoroughly cleaned as only one grain of coarser grit can ruin the polished section (Wells, 1989).
- The finished thin section (Fig. 8) might be covered with a cover slide, but this is optional. Note, however, that thin sections not covered with a glass slide are very delicate and susceptible to scratching.

An older, alternative method for the preparation (A. Wetzel, personal communication July 2023) of thin sections that may work in more fissile, shaly mudrocks is (i) to replace pore water with acetone, (ii) evaporate acetone (=drying the mudrock), (iii) to impregnate the mudrock with low-viscosity resin under vacuum and (iv) to prepare thin section.

For the replacement of pore water, the mudrock is submerged in acetone in a vessel wherein also dry beans are placed. When the acetone starts to replace water, the latter is taken up by the beans. When the beans are swollen, they are taken out of the vessel and dry ones are added—until the beans do not swell any longer. This method can work well in some sediments but is, unfortunately, rather time consuming.

(See figure on next page.)

Fig. 2 Exceptional preservation of fossils in mudrocks. Examples from the Middle Jurassic (Aalenian) Opalinuston Formation of northern Switzerland. **a** ammonite (*Leioceras opalinum* (Reinecke 1818)) with preserved apophyses, shell dissolved but the organic periostracum preserved; **b** ammonite (*Leioceras opalinum* (Reinecke 1818)), preservation as in a, and aptychi preserved in the body chamber; **c** lucinid bivalve with preserved ligament; **d** compressed trace fossil (*Planolites* isp.) preserved as thin pyritized film; **e** small fully articulated tanaidacean crustacean; **f** upper jaw of a belemnite; **g** articulated decapod crustacean (*Mecochirus eckerti* Frentzen 1937); **h** large arm hook (*Onychites*) of a belemnite; **i** delicate trace fossil (*Spirodesmos spiralis* Geinitz 1867) preserved as thin pyritized film; **k** as yet undescribed, fully articulated multi-armed sea star

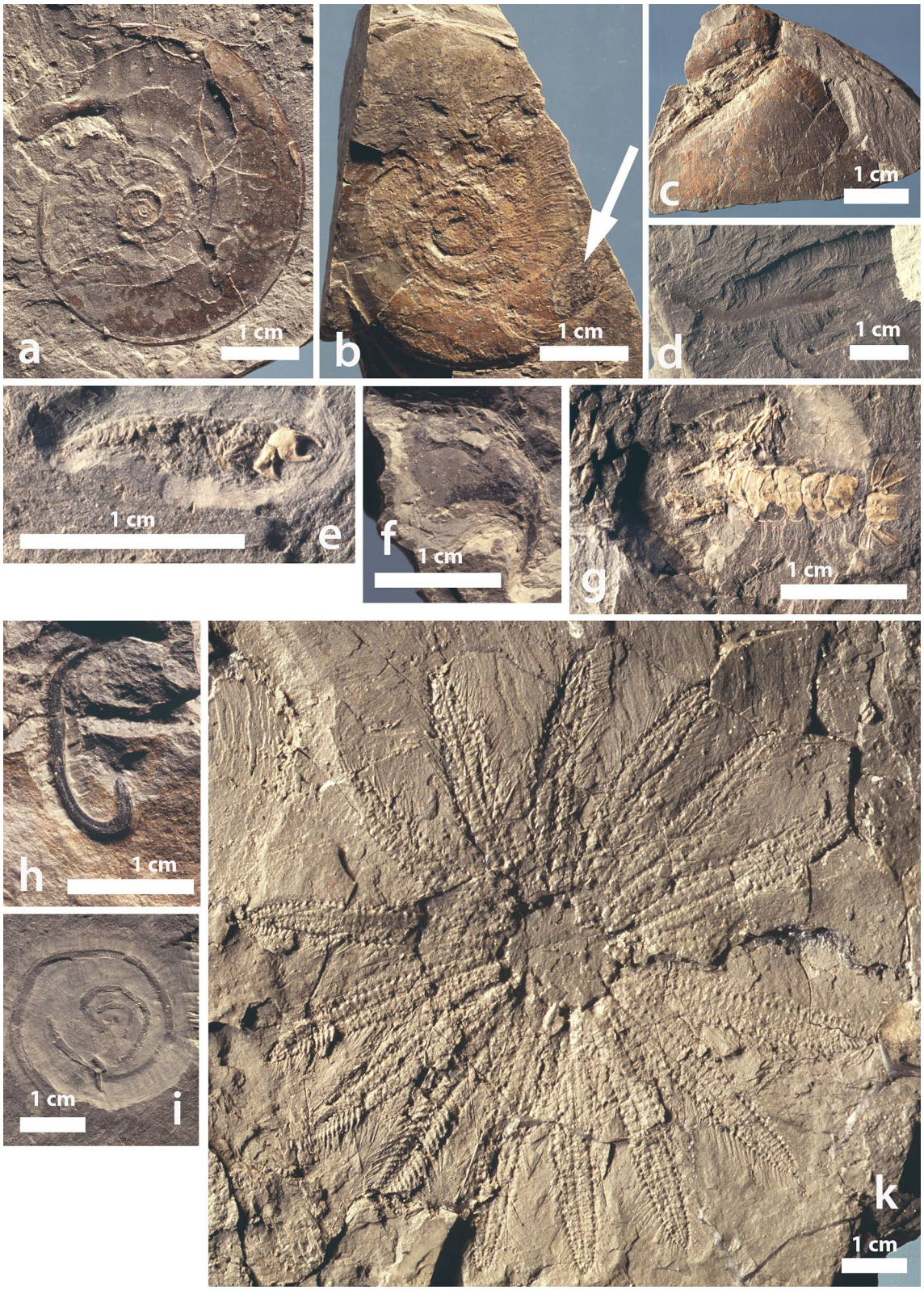

Fig. 2 (See legend on previous page.)

Fig. 3 Surface appearance of weathered mudrocks of the Pliensbachian Amaltheenton Formation at Buttenheim (see Keupp et al., 2022). Only calcitic fossils and concretionary parts of other fossils survived, others decayed together with the mudstone to small chips

Fig. 4 Fieldwork in an abandoned clay pit of the Opalinuston Formation (see Etter, 1990). Left: removing around 1 m of weathered rock. Right: quarrying 1.5 m^2 of a bed 20 cm thick

Recording micro- or cryptobioturbation

In addition to recording the larger trace fossils (Wetzel & Uchmann, 1998) it might be worth looking for micro- or cryptobioturbation (Pemberton et al., 2008). If the minute trace fossils have low contrast to the enclosing rock, they can be checked in thin sections. Yet quite often, they are pyritized. In this case thin slabs of the still wet samples can be manufactured with the stone saw and then be dried in a manner that the slabs stay intact, e.g., covered with a thin layer of epoxy resin.

Fig. 5 Splitting and formatting slabs of the Opalinuston Formation

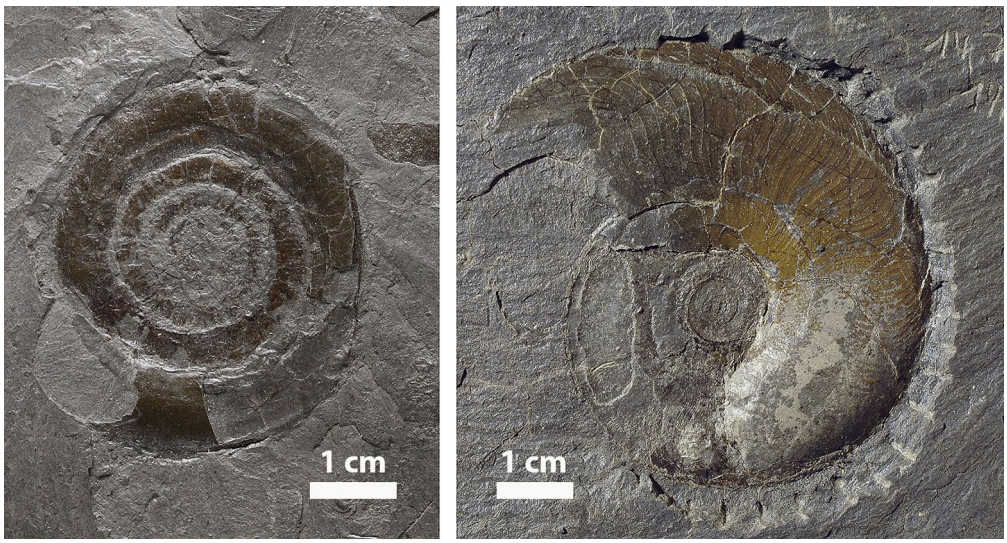

Fig. 6 Ammonites from slabs of unweathered mudrocks. Compare this splendid preservation with that of Fig. 3. From *Laqueoceras laqueous* (Quenstedt 1856) in the left picture, preserved with the periostracum but with the shell dissolved, nothing would remain in a weathered surface exposure. The body chamber concretion of *Leioceras opalinum* (Reinecke 1818) in the right picture would survive on a weathered surface as isolated fragment

Alternatively, dry samples can be manufactured into rectangular prisms using a scroll saw with diamond saw blade. These slabs are then examined with X-rays and might yield very interesting and meaningful results (Fig. 9).

Fig. 7 Applying diluted zapon varnish (= cellulose varnish) to an ammonite (*Laqueoceras laqueous* (Quenstedt 1856)) on a sediment slab. The shell of the ammonite was dissolved, but the delicate periostracum is preserved

Microfossil preparation

There is a confusing large array of methods to disintegrate/break down indurated mudrocks and shales to obtain calcareous microfossils. Obviously, no standard procedure is appropriate for all the rocks (Slipper, 2005; see Harris & Sweet, 1989 for detailed protocols for different rock types). Correspondingly, there is a perplexing number of publications dealing with the different methods.

The standard procedure (Green, 2001; Müller, 1992; Todd et al., 1965) includes the following steps: 200–250 g of dried sample are soaked in 7–15% H_2O_2 peroxide solution overnight, with occasional gentle stirring. There are, however, reservations about using H_2O_2 in marls or clays containing pyrite. It oxidizes the pyrite producing sulfuric acid which will affect the calcareous microfossils especially when they are infilled by pyrite, as is often the case for foraminifera in mudrocks (Riegraf, 1985; Kennedy & Coe, 2014; see also below: Freeze–thaw processing technique). Yet in the mudrocks investigated here (which also contain pyrite), no corrosion of foraminifers was observed. Furthermore, the oxidation of pyrite can be avoided through the addition of a few drops of ammonia (e.g., Keupp, 2022).

A 5% solution of sodium hypochlorite NaClO can be used as a substitute for peroxide (Green 2001; Harris & Sweet, 1989; Sohn et al., 1965). Sodium hypochlorite appears to be effective for breakdown of black shales (Harris & Sweet, 1989). A gentler method for only weakly lithified sediments involves soaking in pure water, perhaps gently boiling to hasten the process of breakdown, or

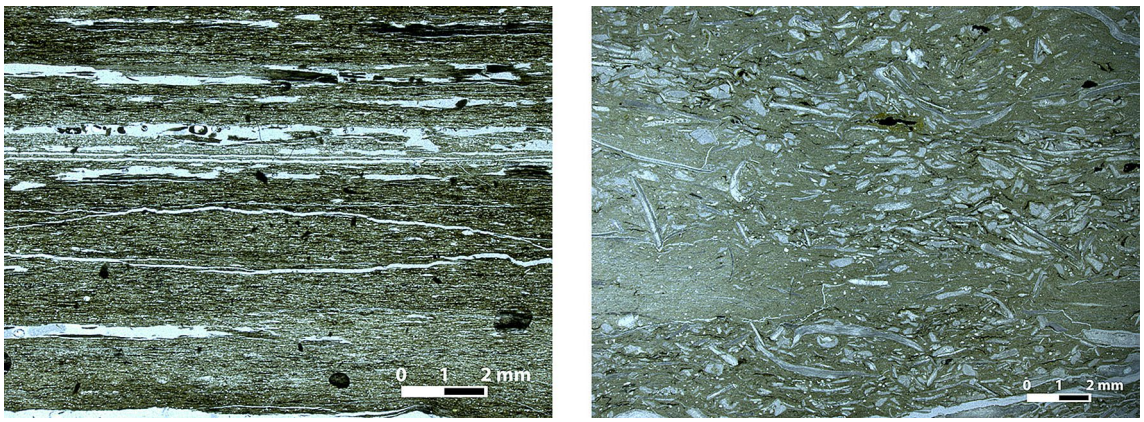

Fig. 8 Two examples of splendid thin section from the Lower Jurassic Schambelen member of northern Switzerland (see Etter & Felber, 2018). Thin sections manufactured by Willy Tschudin, University of Basel

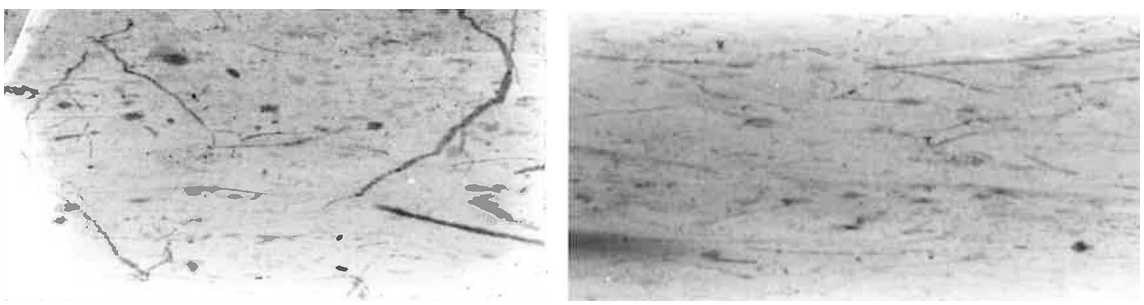

Fig. 9 X-ray images of two slabs from the Opalinuston Formation showing small pyritized traces (microbioturbation) and mottled background (cryptobioturbation). Width of each slab is 2.6 cm

soaking in water with a small amount of soda (Sohn et al., 1965), again boiling to speed up the process of breakdown. This procedure can be repeated several times.

Additional methods involves rupturing the rock through crystallization of salts, the Glaubersalz = sodium sulfate = $Na_2SO_4 \cdot 10\ H_2O$ method (Müller, 1992; Riegraf, 1985). The dry sample is crushed into small pieces then covered with saturated solution of sodium sulfate. Repeated cooling and heating leads to crystallization and therefore breakage of the rock. The resulting pulp must be washed with hot water. Sodium thiosulfate = $Na_2S_2O_3 \cdot 5\ H_2O$ (Saraswati & Srinivasan, 2016) or sodium acetate = $NaCH_3COO$ (Sohn et al., 1965) were used as substitutes.

When rupturing the rock with the freeze-thawing method, the following steps are required: The rock sample is crushed to fragments no larger than 1 cm diameter, dried and then covered with either tap water, a solution of sodium acetate or hydrated sodium sulfate. Only metal or plastic containers should be used because glass beakers and porcelain containers may break when the solution freezes (Pojeta & Balanc, 1989b). The sample is then slowly heated until all the salt crystals are melted, then cooled, and placed in deep freeze. This can be repeated several times until the rock sample is completely disaggregated. After every run it should be controlled if shell material is broken and proceeded only when this is not the case. A very detailed instruction for the freeze–thaw method is given by Hesemann (2020).

Indurated mudrocks and shales and those rich in organic material will very often not fully disintegrate with the gentle, the H_2O_2 and other standard methods, and the amount of residue is large, making picking an extremely arduous task. For these samples additional methods may be applied.

The surfactant Rewoquat which is widely used for the preparation of macrofossils in mudrocks (Strick, 2007) proved to be an efficient agent for the dissolution of excess clay aggregates (Lierl, 1992). It has successfully been used for the extraction of microfossils in mudrocks (Jarochowska et al., 2013) and appears to be most effective after the sample was treated with the standard H_2O_2 method. The sample is submerged without heating with occasional stirring for at least 20 h in Rewoquat. This removes up to 90% of the clay aggregates that were present after the H_2O_2 treatment. A severe disadvantage is that Rewoquat is detrimental for the environment, therefore, it should be used only in small amounts. After treatment the excess Rewoquat should be decanted and used for renewed processing of additional samples.

For the Gasoline–Kerosene-method (Müller, 1992), the rock is mechanically crushed to fragments no larger than 5 mm, and then the dry sample is covered over night with gasoline or kerosene. Boiling in a reflux apparatus (Müller, 1992) is not necessary and also dangerous because of the fumes. The Kerosene is then decanted and immediately thereafter the sample must be covered with boiling water (personal communication Michael Knappertsbusch, July 4th 2023).

Regardless of the disintegration method, the decayed sediment is then wet washed over a sieve stack with 250, 125, and 63 µm mesh sizes (Fig. 10). Only small amounts of sediment should be treated at one time because otherwise the sieves will clog. If the Gasoline–Kerosene-method was applied, the lab sink must include a container that holds back the washed pulp and the Kerosene, preventing the latter to reach the sewage.

The residues of each size fraction are then carefully washed into clean metal bowls (Fig. 10). After letting these stand for several minutes excess water can be decanted. Here extreme care must be taken as some microfossils might float on the surface.

The residues are dried overnight in an oven at 60 °C, and then transferred to small labelled plastic boxes. If the residues still contain a large amount of clay particles, treatment with Rewoquat is recommended (see above), and then again they are washed over a sieve stack and dried. The dried residues can now be picked for microfossils and other remains (e.g., sclerites of macrofossils).

The standard technique for picking the microfossils and the remains of macrofauna uses a picking tray, fine needles or very fine soft brushes, and microfossil slides (Todd et al., 1965; Fig. 11). The screened samples are thinly scattered on a picking tray which is preferably black and has on the inside fine rulerlines. Picking under the microscope (Fig. 12) is done either with a fine needle that is slightly greased from the wing of the nose, or with a very fine soft brush that is dampened with plain water (Todd et al., 1965). The microfossil, which sticks to the needle or the brush tip, is then transferred to a microfossil slide.

Remains of macrofossils like echinoderm ossicles, can be cleaned with ultrasonics before imaging. The ossicles are placed in a water-filled beaker and placed on an insert tray in the water-filled ultrasonic apparatus. This treatment will remove any contamination from the surface of the ossicles. Ultrasonic treatment is not recommended for delicate calcareous microfossils because they easily break (Pojeta & Balanc 1989a).

Conclusions

The big potential of mudrocks for palaeontological research may now be undisputed. Yet adequate methods must be employed to take full advantage of this potential. The first and most important step is the mining of fresh rock. This

Fig. 10 Washing disintegrated sediment over a sieve stack with 250, 125 and 63 µm. The residues are then gently washed into stainless bowls

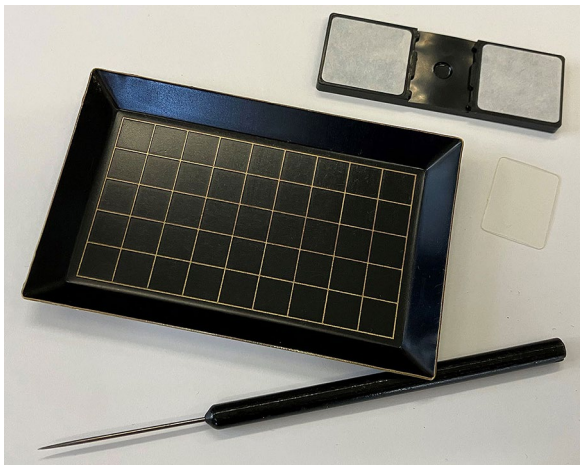

Fig. 11 The material needed for picking microfossils from the dried residues: black picking tray, fine needle and microfossil slides

Fig. 12 Picking under the stereomicroscope

is achieved through systematic excavations in unweathered mudrocks. Only then can delicate fossils be recovered which have their aragonitic shell dissolved but the organic material preserved. We can make these fossils durable with appropriate methods. Manufacturing of thin sections of mudrocks is challenging and must, because mudrocks disintegrate upon contact with water, be done entirely with dry methods. If small trace fossils are pyritized, this microbioturbation can be made visible through X-ray investigations. A plethora of methods exists for the extraction and investigation of microfossils. The disintegration of

mudrocks with a high content in organic carbon is especially difficult. In this case, immersion in the cationic surfactant Rewoquat has proven to be most effective.

Acknowledgements
Willy Tschudin, Basel, provided all the necessary details on how to manufacture thin sections of mudrocks. The manuscript was critically read and corrected by Michael Knappertsbusch, Basel; Daniel Marty, Basel; and Andreas Wetzel, Basel. The corresponding author thanks all of them for their valuable comments.

Author contributions
The author read and approved the final manuscript.

Data availability
Not applicable.

Published online: 15 February 2024

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