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EXTRACTION OF CALCAREOUS MACROFOSSILS FROM THE UPPER CRETACEOUS WHITE CHALK AND OTHER SEDIMENTARY CARBONATES IN DENMARK AND SWEDEN: THE ACID-HOT WATER METHOD AND THE WATERBLASTING TECHNIQUE

Jan Kresten Nielsen and Sten Lennart Jakobsen

ABSTRACT

Improved methods in extracting macrofossils from the Upper Cretaceous White Chalk and other partly lithified calcareous sediments in Denmark and Sweden are presented. Bulk sediment samples may be disaggregated in highly concentrated acetic acid (99-100%) to provide assemblages of cleaned fossils. Because the concentrated acetic acid does not react with carbonates (providing they do not contain any water), the calcareous fossils remain unetched. Adding boiling water, the acid-saturated sediments become rapidly disaggregated by mechanical pressure from the production of carbon dioxide. The procedure is time saving in comparison with the classical Glauber's Salt method. The acid-hot water method is efficient for large bulk samples of limestones that have a high permeability and porosity.

A waterblasting technique is introduced as a cleaning agent in fossil preparation. The pressurized water even leaves fragile skeletal fossils undamaged. Because the technique leaves the fossils in their original position within outcrop surfaces and samples, the technique is readily applicable for biofabric studies. Combined with the acid-hot water method, the waterblasting technique has been employed effectively in preparation of Campanian brachiopods and Maastrichtian bivalves and bryozoans as well as Danian octocorals, asteroids and regular echinoids.

Jan Kresten Nielsen. Geological Museum, University of Copenhagen, Øster Voldgade 5-7, DK-1350 Copenhagen K, Denmark. bioerosion@yahoo.dk
Sten Lennart Jakobsen. Geological Museum, University of Copenhagen, Øster Voldgade 5-7, DK-1350 Copenhagen K, Denmark. slj@savik.geomus.ku.dk

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INTRODUCTION

Extraction of fossils from carbonates is affected by using different techniques depending on the composition and preservation of the fossils and the hardness of the enclosing sediment (matrix). Preparation consists of the following steps: 1) breaking up the sample (disaggregation) by chemical and physical means; 2) removing the fine fraction through sieving; 3) drying the residue; and 4) hand-picking the fossils. The harder the sample is, the harsher the preparation treatment required (Green 2001). For uncemented, slightly consolidated sediments, treatment by wet sieving is generally sufficient. If the fossils are embedded in more firm sediments, such as chalk, sieving will be useless without proper pre-treatment. The sample must be subjected to a procedure of disaggregation (without damaging the fossils) prior to wet sieving. The simplest way of disaggregating indurated calcareous sediments, such as the Upper Cretaceous White Chalk, is by saturating the samples with water and then placing them in a freezer. The formation of ice crystals in the pore system breaks up the matrix (Hanna and Church 1928; Pojeta and Balanc 1989). This freeze-thaw method can be repeated several times until the sediment (matrix) is fully disaggregated. Another freeze-thaw method is Glauber's Salt method (Na_2SO_4 $10H_2O$, hydrated sodium sulphate) that also requires repetition of the procedure to function properly (Surlyk 1972).

The purpose of this paper is to document the combination of two methods that are more efficient and less time-consuming than freeze-thaw methods but have received little attention. These are called the acid-hot water method and the water-blasting technique. They have been applied separately and in combination on samples from two localities: Lower Campanian skeletal sands at Ivö Klack, Sweden; the Maastrichtian White Chalk at Stevns Chalk Quarry, Denmark; the Danian Bryozoan Limestone at the sea cliff Stevns Klint, Denmark; and the Danian Bryozoan Limestone at the Fakse Limestone Quarry, Denmark.

HISTORY OF CHEMICAL DISAGGREGATION METHODS

One method of splitting up calcareous sediments is Glauber's Salt method (Franke 1922; Wicher 1942; Herrig 1966; Surlyk 1972; Schmid 1974; Wissing and Herrig 1999). Following an idea from A.G.W. van Riemsdyk, Harting (1866) introduced the use of a saturated solution of crystalline sodium sulphate as a substitute for ordinary water. The method, using a solution prepared at about 40°C, was successfully used by Keilhack (1917) and Franke (1922) for extraction of foraminifers and other microfossils from chalk. The sodium sulphate crystals grow in the pore system initiated by placing the samples in a deep freezer (or a water-cooled container), and was an improvement in the water-based freeze-thaw process and decreased the cycles of treatment to 16 to 18 times (Wicher 1942; Surlyk 1972). Moreover, Wicher (1942) modified the Glauber's Salt method to involve heating of samples covered with anhydrous sodium sulphate and subsequent boiling in a concentrated solution of sodium sulphate. The method is destructive to the microfossils, and thus, Kirchner (1958; Green 2001) revised the method by maintaining the temperature of the solution below the boiling point. Although still widely used, the Glauber's Salt method has some disadvantages: 1) The samples have to be reduced to smaller pieces of walnut size prior to treatment with Glauber's Salt; 2) The method is time consuming; 3) The fossils are not generally free of adhering sediment particles, and therefore need to be cleaned in an ultrasonic bath.

Non-calcareous fossils are usually extracted from chalk and limestones by dissolving the matrix with a solution of acetic acid (Reid 1958; Müller 1962; Zankl 1965; Rudner 1972; Jeppsson et al. 1985, 1999). Because this process destroys the calcareous fossils, other methods must be used to extract such fossils. Improving the procedure by Bourdon (1956; 1962; Lethiers and Crasquin-Soleau 1988), Nötzold (1965) described a method for disaggregating calcareous and clayey calcareous sediments used to extract charophytes and gyrogonites. A sample is submerged partly (2/3) in a solution of 96% acetic acid (CH_3COOH) and anhydrous copper II sulphate ($CuSO_4$) for 12 to 20 hours. Then the sample is removed from the solution and covered by at least 10 litres of cold water. The formation of calcium acetate ($(CH_3COO)_2Ca$) and absorption of water within crystals leads to the breakdown of the sample (Nötzold 1965; Hansch 1994; Reich 1997; Wissing and Herrig 1999). The method requires the use of highly toxic components. Another drawback is that further treatment with hydrogen peroxide (H_2O_2) may be required to clean fossils (Wissing and Herrig 1999). Our acid-hot water method resembles a modified and much improved version of the method given by Nötzold (1965). However, there is a fundamental difference: the disaggregation is caused by carbon dioxide formation and not crystal-bound water as in Nötzold's (1965) method.

THE ACID-HOT WATER METHOD

A faster and more efficient method for disaggregating coccolithic White Chalk is presented below. The method involves the use of concentrated acetic acid (99-100% pure acid) and must be employed with great care in a well-ventilated fume hood. Because the acetic acid does not completely dissociate into its component ions when dissolved in an aqueous solution, it is classified as a weak acid. A solution of acetic acid is a dynamic equilibrium between acetate ions (CH_3COO^-), hydronium ions (H_3O^+) and neutral molecules. The dissociation constant for acetic acid is 1.8 x 10^{-5} at 25°C. The essence of the method is that owing to the low dissociation, concentrated acetic acid, when applied to calcareous sediment, does not affect the carbonate until mixed with water. The key step is to cover acid-saturated samples with plenty of boiling water. This may subsequently lead to complete disaggregation of the samples. Accordingly, the method described here involves the use of concentrated acetic acid prior to disaggregation in boiling water.

The White Chalk samples used in our experiment are 60 x 60 x 60 mm in size and weigh about 190 g. The samples must be thoroughly dried overnight in a ventilated oven prior to immersion in acid. To avoid hardening of the sediment, the temperature should be less than about 50°C. After soaking for 40 minutes (normally sufficient for a complete saturation of the sample), the acid is decanted, and the sample is covered with boiling water containing a buffer solution of sodium carbonate (i.e., soda ash). When the acid is mixed with the water, an immediate chemical reaction takes place. The formation of carbon dioxide causes breakdown of the sample into finer particles. Although some of the fossils may be damaged during this process, sedimentary rocks generally disaggregate at their weakest points, which are the contacts between the fossils and the matrix. After 30 minutes, the sample is almost entirely broken down and can be sieved through a set of mesh screens that retain the larger fossils and other particles. The residue is now thoroughly washed in cold running water. The fossil rich residue consisting mostly of foraminifers, ostracods, brachiopods and remains of larger biogenic fragments, is oven dried at 90°C.

The dried residue is covered with concentrated acetic acid until saturated. Treatment is renewed with the addition of boiling water containing a buffered solution of sodium carbonate (Na_2CO_3) in order to keep the solution neutral. A few minutes later, the residue is entirely disaggregated by the action of carbon dioxide formation. After this second treatment, the residue is washed in cold water and separated into different size fractions. Although the formation of carbon dioxide implies etching of carbonate, the fossils are free of dissolution features at 40x magnification. The extent of etching on microfossils and nannofossils remains to be examined.

Safety notice: as the fluid and vapour of concentrated acetic acid are toxic, the method may be hazardous. The procedure should be carried out within a fume hood. Because concentrated acetic acid is very corrosive and can cause painful burns, safety gloves and glasses must be worn.

HISTORY OF BLASTING TECHNIQUES

One of the basic requirements for studying fossils is to remove obscuring matrix. Various mechanical methods for surface cleaning include tools such as hammer and chisels, air-operated tools such as air scribes, rotary grinders, wire brushing, ultrasonic cleaning and sandblasting (air abrasive). The earliest record of sandblasting utilized in palaeontology dates back to the 1890s. At that time existing commercial sandblasting equipment was not designed for palaeontological work. In his study of Wenlockian trilobites, Bernard (1894) tried to remove the matrix of their underside in order to reveal hidden appendages. As the marly limestone was unfavourable for preservation, the attempt was unsuccessful. Together with W.I. Last (South Kensington Museum), however, Bernard (1894) succeeded in constructing a tool for future applications in palaeontology. Despite the usefulness of this cleaning equipment for fossils, the popularisation of the sandblasting technique first came with the development of the micro sandblaster for use in dentistry in the late 1940s (Osborn 1904). The micro sandblaster was marketed as Airdent by the firm S.S. White. However, it was not until the 1960s that the sandblasting technique was re-introduced in preparation laboratories and subsequently led to a wide range of applications in palaeontological research (e.g., Stucker 1961; Spreng 1962; Stucker et al. 1965; Aichinger 1969; Kuhn-Schnyder 1969; Lanooy 1970; Rensberger 1971; Gunther et al. 1979; Lörcher 1984; Hannibal et al. 1988; Hannibal 1989).

The use of high-pressurized water, known as waterblasting, is a common industrial procedure for removing stubborn deposits such as paint and rust, from various kinds of surface materials. The waterblasting technology has been known for about 30 years, but has so far only been used in palaeontology on a limited scale by Jakobsen (2003). By blasting, the water creates an abrasive spray that is as effective, but not as damaging, as sandblasting. The waterblasting technology offers a new option in mechanical treatment of fossils.

THE WATERBLASTING TECHNIQUE

The waterblasting process delivers water as an abrasive material under high pressure. By using an ordinary pressure washer (Alto Dynamic X-tra, Kew Technology, Type P406) with adjustable water pressure up to 150 bars, surfaces of uncemented and weakly cemented sedimentary carbonates are cleaned rapidly with outstanding results. Different nozzles can be mounted onto the pressure washer. Some nozzles fan out the water to cover a wide area resulting in a gentle cutting action. The angle of spray should be kept at 90 degrees to avoid grooves within the sediment surface. By moving the nozzle back and forth over a surface, matrix is removed and macrofossils may be exposed *in situ*.

Other nozzles have tight spray patterns suitable for deep cutting. Prolonged cleaning of the samples can result in a complete disintegration of the sediment, so the fossil remains can be extracted for study without any evidence of abrasion. Cutting by the pressure washer can be varied by adjusting the water pressure, thus raising or lowering the velocity of the water stream, or by changing nozzles, thus altering the spray pattern of water emitted.

Subtle cleaning of macrofossils may require lower water pressure than provided by the ordinary pressure washers. This type of cleaning may be facilitated by using a hand-held watergun (Wagner Model W 400 SE) with adjustable water pressure up to 180 bars at 10 mm from its orifice. For even more delicate cleaning, the Paasche airbrush (Model VL #3; http://www.paascheairbrush.com) is useful because of its smaller size when delivering a water stream at about 8 bars. By adding carborundum powder (1000 grit) to the water, the effect of blasting may be increased by a factor 10 to 20. Adding a few drops of detergent may ease the suspension of the powder. Because the suspended powder is covered by a water film, the technique is much less abrasive than sandblasting. The water film functions like a cushion around the powder grains.

Safety notice: never point the blasting equipment toward people. Safety glasses and gloves must be worn. The sample should be secured tightly before blasting.

EXAMPLES

Because no single tool or method is universally suitable for preparing fossils, the initial step in any preparation is to consider the best choice of method. A combination of different methods may work best. Treatment of calcitic encrustations such as worm tubes and bryozoans with gentle waterblasting and the acid-hot water method may provide further information about the fossil record. Prepared fossils are deposited in the Geological Museum, University of Copenhagen (DK and GM numbers).

Danian octocorals from the Fakse Limestone Quarry, Zealand, Denmark

Together with the Stevns Klint section, the Fakse Limestone Quarry constitutes the type locality of the Danian Stage, which is the lowermost stage of the Paleogene. At Fakse, the Bryozoan Limestone is interbedded with Coral Limestone within a large coral-bryozoan mound complex (Floris 1980; Willumsen 1995; Surlyk 1997). The Bryozoan Limestone contains abundant octocorals in places. Surfaces of blocks containing the octocoral, *Moltkia isis* Steenstrup, 1847, were cleaned by the large-scale waterblasting technique (Figures 1, 2). To remove the matrix surrounding the octocorals, an ordinary pressure washer (Alto Dynamic X-tra, Kew Technology) was applied.

Lower Campanian brachiopods from Ivö Klack, Scania, Sweden

Marine skeletal sands, which may be characterised as shell banks consisting in the main of oysters and other sessile bivalves, occur on the Swedish island Ivö Klack. An uppermost Lower Campanian age is indicated by belemnites (Christensen 1975). Large specimens of the inarticulate brachiopod *Crania stobaei* Lundgren, 1885 (up to 30 mm in size) are common within the sands. The shells of *C. stobaei* are usually filled with consolidated sediment that covers internal morphological features such as muscle scars. Clearing by using air scribes, scrapers and other mechanical aids has hitherto been the only way of exposing the inner shell surfaces. Preparation by combining the acid-hot water method and waterblasting has produced fine results, even over short time periods (Figures 3, 4).

Procedure:

1. Before preparation, the specimen must be impregnated with a suitable lacquer on the outer shell surface. Otherwise, the thin shells disintegrate during processing. When the lac-

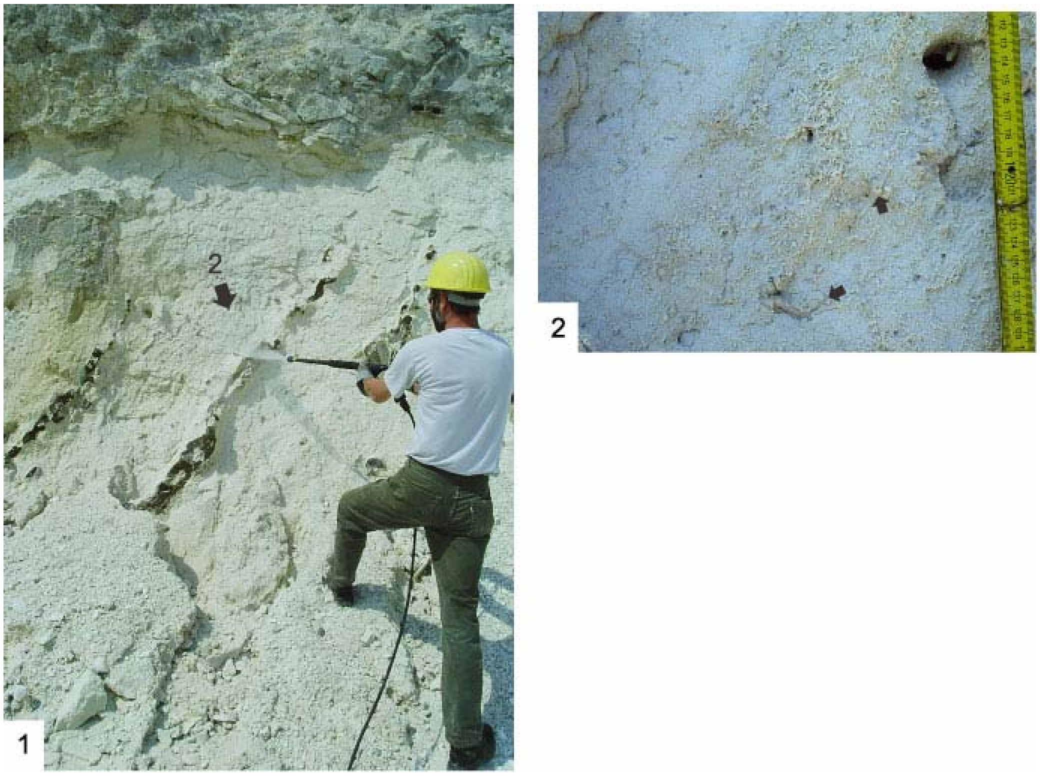

Figure 1. Large-scale waterblasting of an exposure of the Middle Danian Bryozoan Limestone, Fakse Limestone Quarry, Denmark. **1.** The angle of spray is maintained at 90 degrees. **2.** The waterblasting creates rapid artificial weathering exposing numerous bryozoans and octocorals (*Moltkia isis*) (arrows). To the left untreated Bryozoan Limestone is seen. Scale on the folding rule is in cm.

quer is dry, the specimen is treated with concentrated acetic acid.

2. Concentrated acetic acid is poured over the specimen. A wash bottle may be used to apply the acid locally on matrix. Application of acetic acid should be done repeatedly until the matrix has absorbed sufficient acid.
3. After 10 minutes, the acid-treated specimen is transferred to a container of suitable size, and plenty of boiling water is poured over the specimen. A vigorous effervescence of carbon dioxide is caused by the mixture of the acid and the water. When bubbles of carbon dioxide can no longer be observed in the solution (about 20 to 30 minutes), the specimen is removed and cleaned with a Wagner watergun at maximum working pressure. The treatment with acid and boiling water softens the matrix, allowing high-pressure washing to completely remove it.
4. The specimen is then carefully washed in water to avoid the formation of calcium acetate crystals, which may be destructive. The specimen is then dried in an oven for about three hours at 50°C.
5. The procedure is repeated until all matrix is removed. The specimen illustrated in Figure 4 had two treatments following the described procedure (Figure 4.1-4.3). The complete process took about six hours.

Remarks: Boiling water may damage shells by exploiting pre-existing shell weaknesses. These weaknesses probably originate from earlier sediment compaction, and the treatment may exacerbate them.

Middle Danian echinoderms from Stevns Klint, Zealand, Denmark

Bryozoan wackestone and packstone mounds are well exposed within the Stevns Klint section, Zealand (Surlyk 1997; Surlyk and Håkansson 1999). The mounds, which are Danian in age, consist of fragmented bryozoans within chalk-like debris of coccoliths, thoracospheres, foraminifers and invertebrate shells. Brachiopods, serpulids, echinoids, crinoids and octocorals are common (Surlyk 1997). Recently, a complete specimen of the asteroid, *Metopaster kagstrupensis* Brünnich Nielsen, 1943, was collected from Middle Danian wackestone at Stevns Klint. The specimen needed preparation to uncover morphological features of the obscured side of the asteroid (Figure 5).

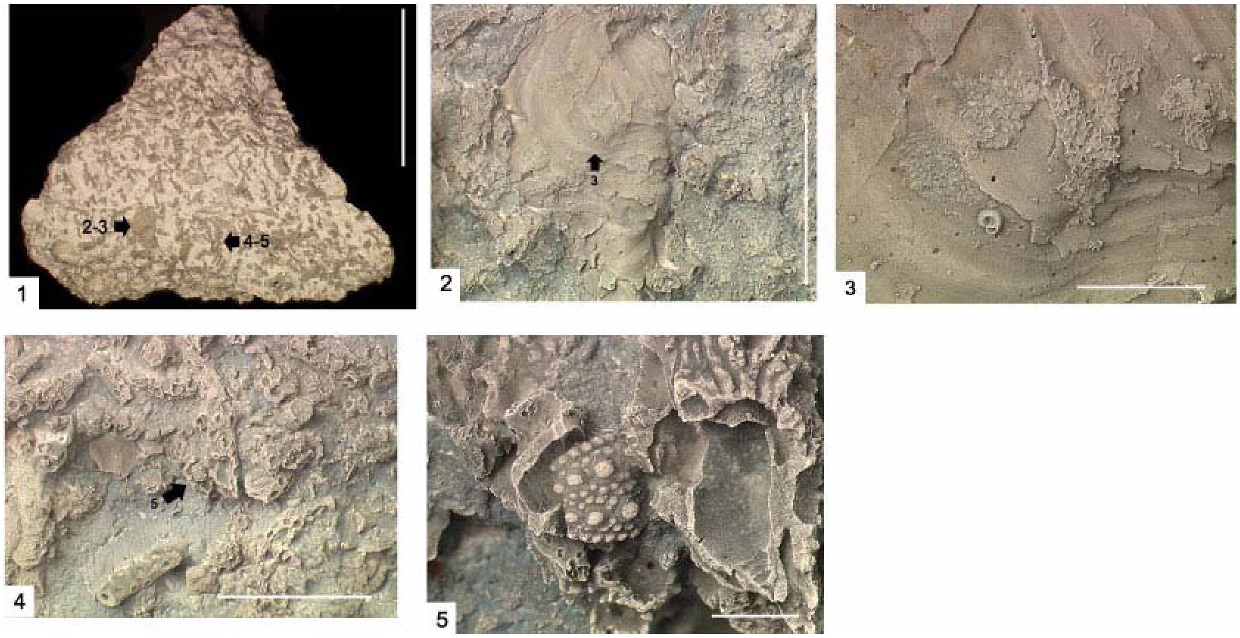

Figure 2. The result of large-scale waterblasting of a block of Danian chalky limestone, Fakse Limestone Quarry. Sample GM 2004.919. **1.** Bedding plane with abundant octocorals and oysters. The cleaning took about one minute. Scale bar 150 mm. **2.** Close-up of a large oyster with scattered encrustations of cheliostomate bryozoans and single serpulids. Scale bar 25 mm. **3.** Further close-up section of the oyster showing that the waterblasting provides a superior cleaning effect without harming encrustations and boring openings (*Entobia* isp.). Scale bar 5 mm. **4.** Close-up of numerous fragmented stems of octocorals and a fragment of an echinoid test, *Cidaris* sp. (arrow). Scale bar 20 mm. **5.** Further close-up of the echinoid and the octocorals. The octocorals have been heavily bioeroded by *Cliona* sponges. Scale bar 2 mm.

Figure 3. The Wagner watergun, suitable for medium-scale waterblasting.

Procedure:

1. The preparation of the asteroid was more complex than the cleaning of inarticulate brachiopods. The specimen and the enclosed matrix were pre-treated with Synocryl 9122x (acryl-polymer) (Cray Valley Products Ltd., http://www.crayvalley.com/) to withstand possible disaggregation.
2. Additional strengthening of the specimen was obtained by covering one side of the specimen with plaster of Paris (Figure 5.2). The plaster jacket provided a base against which the uncovered side could be cleaned without breaking the skeletal plates (Pojeta and Balanc 1989).
3. To loosen the matrix covering the specimen, concentrated acetic acid and boiling water were applied locally. Then the specimen was dried overnight at 50°C. The acid-hot water method was repeated four times (Figure 5.3-5.6).
4. After cleaning, the plaster jacket was removed mechanically, and the underside of the specimen became visible (Figure 5.7). The duration of the preparation was about two hours. In similar cases, hydrated tri-sodium citrate ($Na_3C_6H_5O_7$ $2H_2O$) may be used to break down the plaster of Paris (Pojeta and Balanc 1989).

In addition, the Stevns Klint section yielded Middle Danian specimens of regular echinoids with attached skeletal plates. A regular echinoid recovered from the bryozoan wackestone was successfully prepared by the aims of needles and

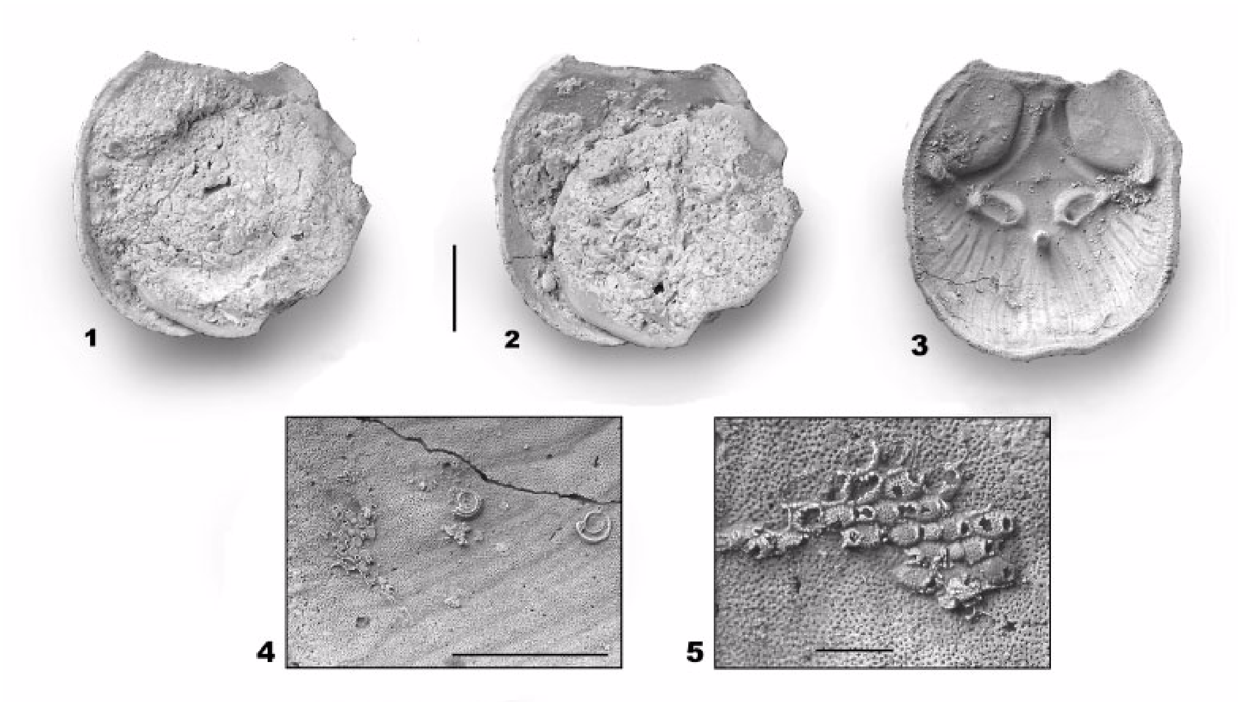

Figure 4. Combined application of the acid-hot water method and medium-scale waterblasting technique (Wagner watergun). Lower Campanian inarticulate brachiopod, *Crania stobaei*, from Ivö Klack, Sweden. GM 2004.920. **1-3.** Successive stages of removal of surface matrix. Scale bar 10 mm. **4.** Close-up of encrustations of bryozoans and serpulids. Scale bar 5 mm. **5.** Close-up of bryozoan colony. Scale bar 1 mm.

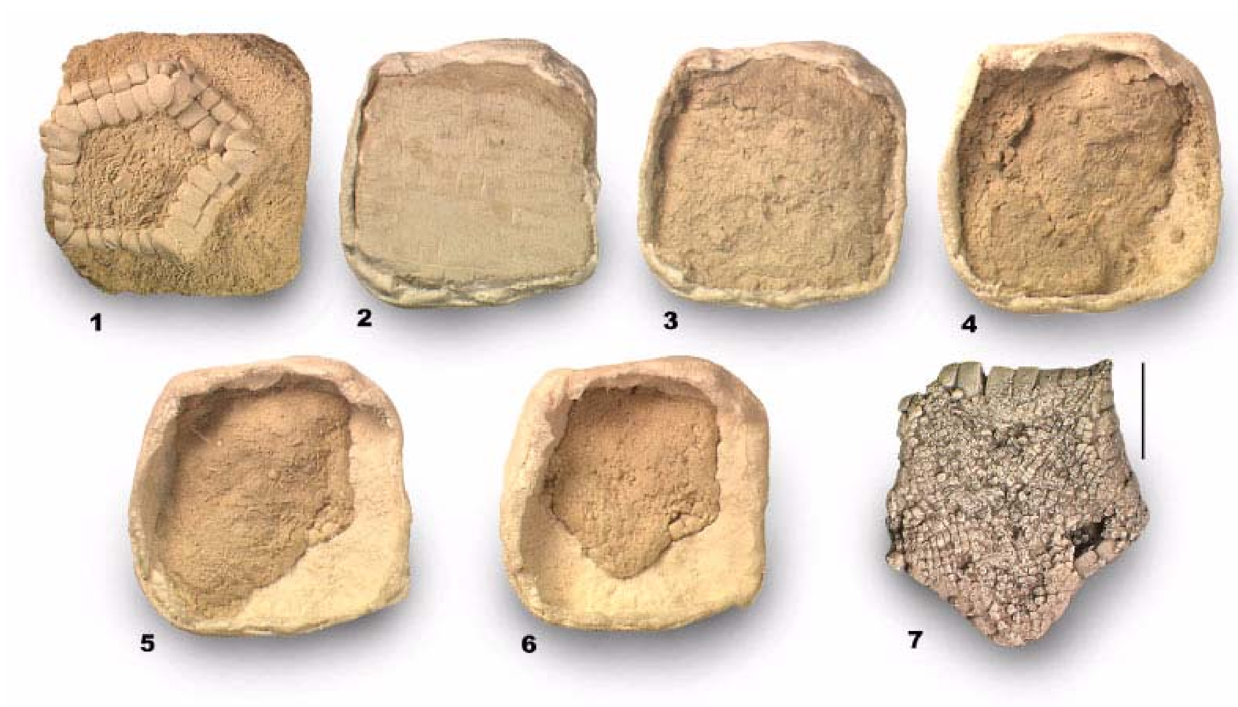

Figure 5. Application of the acid-hot water method on an articulated asteroid, *Metopaster kagstrupensis* Brünnich Nielsen, 1943. The asteroid was recovered from the Middle Danian Bryozoan Limestone (*Tylocidaris abildgaardi* Biozone) at Stevns Klint, Denmark. DK 283. Scale bar 25 mm. **1.** Dorsal side of *M. kagstrupensis*. **2.** The specimen enclosed in plaster jacket before treatment with the acid-hot water method. **3-6.** Successive stages in preparation. **7.** Ventral side of the prepared asteroid, airbrushed with black ink prior to coating with ammonium chloride.

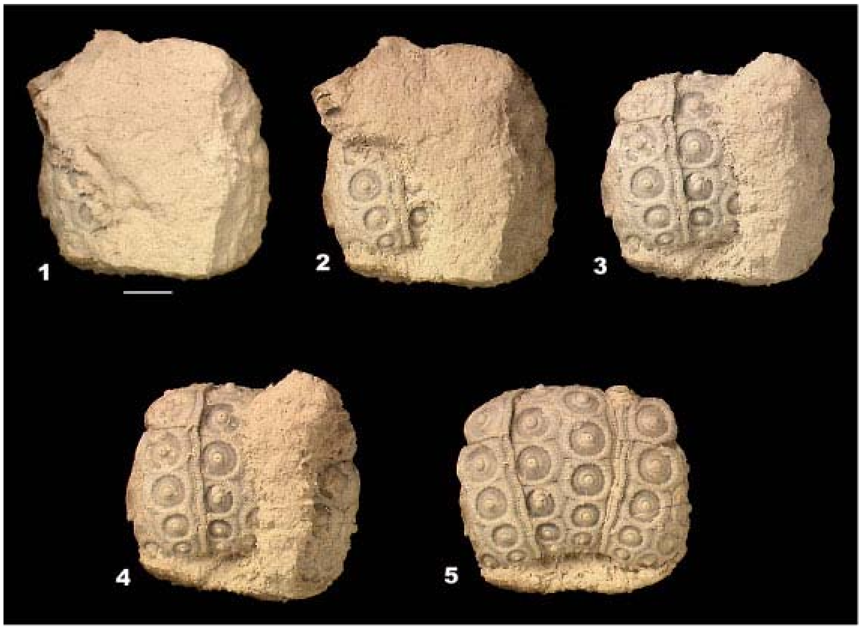

Figure 6. Medium-scale waterblasting and needle preparation of a regular echinoid, *Stereocidaris* sp., from the Middle Danian Bryozoan Limestone (*Tylocidaris abildgaardi* Biozone) at Kulsti Rende, Stevns Klint. GM 2004.921. Scale bar 10 mm. **1.** Before preparation. **2-5.** Successive stages in preparation.

waterblasting technique (Figure 6). The Wagner watergun proved suitable for cleaning the specimen, which can be referred to *Typocidaris danica* Ravn, 1928. According to Smith and Jeffery (2000), *T. danica* is a junior synonym for *Temnocidaris* (*Stereocidaris*) *arnaudi* (Lambert 1909).

Maastrichtian bivalves from Stevns Chalk Quarry, Zealand, Denmark

The Upper Maastrichtian White Chalk is well exposed at the Stevns Chalk Quarry, which is adjacent to Stevns Klint. Calcitic shells of bivalves are commonly well preserved within the White Chalk. Shells of different species were carefully prepared by the use of small-scale wetblasting with carborundum powder (Paasche airbrush) (Figures 7, 8). To avoid damage of the shells during preparation, their inner surfaces were embedded in epoxy ("Strong Epoxy, Rapid," product no. 2806, company Casco). The preparation, which took a few minutes for each shell, revealed subtle details of external morphological features such as radial ribs and scales. Encrustations of bryozoan colonies were also preserved unaffected by the wetblasting (Figure 8.6-8.7).

Figure 7. The Paasche airbrush, suitable for small-scale wetblasting.

CONCLUSION

The main advantages of the acid-hot water method are: 1) A significant reduction of time compared to the Glauber's Salt method; 2) Large-sized samples can be treated, which makes it easier to extract fossils from bulk samples; 3) Fossils are cleaned of adhering sediment particles, which makes the method more reliable within a quantitative analysis of the sediments; 4) The method is particularly useful in examining drill cores, which have obvious sample size limitations; 5) The method has numerous applications in both the field and the laboratory in combination with waterblasting cleaning.

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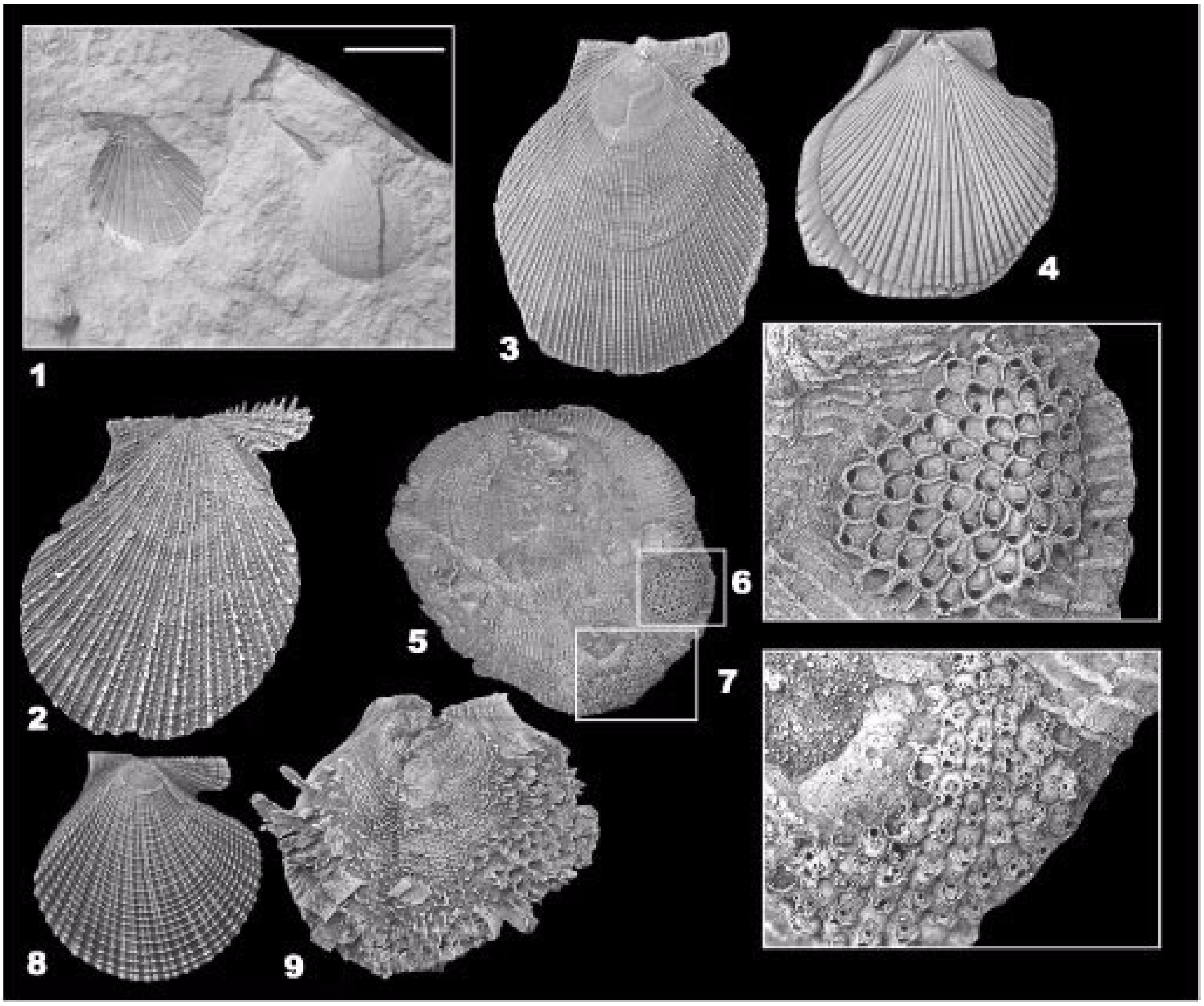

Figure 8. Small-scale wetblasting with carborundum powder (Paasche airbrush) of bivalves from the Upper Maastrichtian White Chalk at Stevns Chalk Quarry adjacent to Stevns Klint, Denmark. These calcitic bivalves may be encrusted by fragile bryozoans. **1.** *Merklinia variabilis* (von Hagenov, 1842). The ornamentation of their outer shell surface is commonly covered by matrix. GM 2004.922. Scale bar 10 mm. **2.** *Merklinia variabilis*, right valve. GM 1987.67. Scale bar 5 mm. **3.** *Mimachlamys cretosa* (Defrance in Brongniart, 1822), right valve. GM 2004.923. Scale bar 5 mm. **4.** *Dhondtichlamys pulchella* (Nilsson, 1827), both valves. GM 2004.924. Scale bar 5 mm. **5.** *Paranomia* sp. GM 1987.53. Scale bar 5 mm. **6.** Bryozoan colony, unknown taxon. Scale bar 2 mm. **7.** *Tricephalopora* sp. Scale bar 2 mm. **8.** *Dhondtichlamys campaniensis* (d´Orbigny, 1847), right valve. GM 1987.71. Scale bar 5 mm. **9.** *Spondylus dutempleanus* d´Orbigny, 1847, left valve. GM 2004.925. Scale bar 5 mm.

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