Technical Note

Ramachandran Dhavamani, Golej Marián, Starek Dušan, and Pipík Radovan*

Dehydration and stabilization of unconsolidated laminated lake sediments using gypsum for the preparation of thin sections

https://doi.org/10.1515/geo-2020-0211
received July 10, 2020; accepted November 05, 2020

Abstract: An improved Lamoureux method for subsampling of unconsolidated laminated sediment is described. Here, we describe a new methodological approach that changes the Lamoureux method in four steps of which the most important change concerns dehydration and stabilization. In this step, we adopted gypsum embedding of the subsample, which took about 1 h to harden and keeps the sediment partially moist. After drying of the gypsum, the subsamples are impregnated with Epoxy 2000 resin under room temperature. This method requires commonly available equipment and can be implemented cost-effectively within 3–4 days.

Keywords: paleolimnology, annually laminated deposit, dehydration, micro-facies analyses, varve counting

1 Introduction

High-resolution paleolimnological and paleoclimatological data can be reconstructed from the annually laminated (varved) sediments of lacustrine and marine environments [1–4].

The microscopic investigation and microfacies analyses are essential activities for the understanding of varve formation [4–6]. The polished thin sections are effective for high-resolution microscopic studies of unconsolidated sediments [7]. They are widely used by the paleolimnologists and paleoclimatologists for the (1) characterization of annually laminated sediments [8,9], (2) study of the grain size variation [5,10,11], and foremost, (3) varve counting [8,9,12]. This conventional method of investigation can be integrated with a high-resolution micro-XRF study, which provides the seasonal indicators recorded in varve deposits [3,4]. However, the preparation of the thin-sections from the unconsolidated deposit, which preserves varve structures, is always a big challenge for the sedimentologists and laboratory technicians. It is a delicate procedure in all steps, and several techniques and protocols have been developed for the sampling and permanent storage, dehydration, impregnation, and polymerization of unconsolidated deposit [13–15]. Especially, the appropriate dehydration and impregnation method allows to decrease the water content and prevent a shrinkage and texture destruction of a fine clastic sediment and its later usage for various sedimentological and geochemical analyses [14]. Freeze-drying [12,16] and acetone in the liquid or vapor phase [13,14] are principal methods of dehydration followed by embedding with high-viscous polymers to preserve sediments permanently [8,13,17].

This article presents a modified approach of ref. [13] for impregnating the unconsolidated clastic sediment found in the Tatra Mts. lakes. Our work focuses on the preparation of thin sections with easily available and accessible tools, easy to perform as well as a low-cost method. Detailed steps are described with the illustrations for subsampling of the core sediment, dehydration, and impregnation of subsamples.

2 Material

2.1 Sedimentary material

- Sample – fresh laminated limnic clastic deposit in the plastic tube used for coring. The deposits come from the lakes
Popradské pleso (1,494 m asl), Batizovské pleso (1,884 m asl), and Zelené pleso Kežmarské (1,546 m asl). All deposits have similar mineralogical and grains size composition

- Subsample – limnic sediment removed from split core using subsampler.

2.2 Tools for the preparation of the thin sections

- Subsampler – consists of a brass insertion tray of the U form with aluminum plate and thin silicon sheet (Figure 1).
- Brass sheet for the preparation of insertion tray – a brass sheet of 160 × 45 mm and thickness of 0.3 mm folded to an interior width of 25 mm and height 8 mm. At least, three holes of 6 mm in diameter are made in the insertion tray to push out the inserted aluminum plate.
- Aluminum plate – 160 × 25 mm and thickness of 2 mm; it is used for the successful removal of the wet sediment from the brass insertion tray.
- Thin silicon sheet – 160 × 25 mm, 1 mm in thickness. It helps in easy removal of the aluminum plate from the subsample [13] without disturbing and sticking of sediment to the aluminum plate surfaces. Silicon sheet is chosen due to its flexibility, reusability after proper sterilization/washing process and at least does not stick on the fresh surface.
- Large silicon sheet – 200 × 75 mm, 1 mm in thickness for placing the deposit from subsampler to the plastic container.
- Plastic container (suitable material is PP, PE, food storage boxes) – container for the embedding of the subsample with gypsum; container dimensions: length 20 cm, width 4.5 cm, and height 4 cm.
- Embedding box (suitable material is PP, PE, food storage boxes) – for Epoxy resin impregnation of the sediment; should be slightly larger than gypsum embedded subsample.
- Paper label – of size 16 × 2.5 cm (120 g heavy) for marking the core orientation and sampling depth to cover the entire insertion tray. It also helps to minimize crack formation during the drying process.
- Cardboard plate – 2 mm thick
- Diamond saw – for cutting off the plastic tube and to cut the impregnated subsample into appropriate size.
- Razorblade – for removal of the uneven sediment surface
- Vacuum chamber
- Needle

2.3 Chemical components

- Epoxy 2000 (resin + hardener) – 75 g of the mixture per subsample for sediment impregnation
- Gypsum – 250 g per subsample

3 Result

3.1 Core cutting and subsampling of the core sediment

Initially, after the splitting of the sediment core on the core splitter, the disturbed sediment surface is cleaned and leveled with a razorblade (Figure 2a). We make sure that the material is undisturbed and appropriate reference numbers and directions are marked on the paper label and attached to the fresh core surface before the subsampler insertion. After that subsampler (inserted with aluminum plate and thin silicon sheet) is pushed carefully into the wet sediment surface, which will hold the sediment for preparing thin section (Figure 2b). Since the diameter of the core is 6 cm, it allows us to insert two subsamplers at once (Figure 2b). Then, the wall of the sediment core (plastic tube) is cut from both sides using a diamond saw, and the sediment in subsampler is separated from the rest of the core using a clean razorblade (Figure 2c). The separation of the subsampler from the main core is done by slow slicing with razorblade and the excess sediment on the sides of the subsampler is removed (Figure 2d–f). Next, we put the subsample on a clean large silicon sheet placed over a cardboard (Figure 3a). We hold the subsampler with one hand, lift it upward and through the hole in subsampler using a needle, the aluminum plate and the thin silicon sheet with the sediment is carefully pushed downward on a large silicon sheet (Figure 3b and c). After this step, we turn the subsample together with aluminum plate and the thin silicon sheet in a plastic container (Figure 3d). And finally, the subsample is ready for embedding with gypsum.

3.2 Sediment stabilization and dehydration

The sediment subsample should be dehydrated before epoxy impregnation. Our alternative method uses the
gypsum to embed the fresh (wet) subsample (Figure 4). Because we prepared two subsamples from each core (Figure 2b), we used both for embedding in a single step. We prepare the gypsum in semiliquid consistency following the gypsum producer instructions and pour it to the plastic container with the subsample and the space is filled (Figure 3e). The thickness of the gypsum layer should be 10–15 mm to avoid deformations during the drying and cutting. The hardening time in the plastic container is about 1 h. Once the gypsum is hard, the gypsum embedded subsamples are extracted from the plastic container and separated by a saw into two parts for faster drying (Figure 3f). In this step, we remove the aluminum plate and thin silicon sheet (Figure 3g). The paper label remains attached to the core sediment. After 2 days of drying at room temperature (Table 1), paper label is removed from the surface and we slice the gypsum wall to a maximum of 3 mm thick for a better epoxy impregnation (Figure 3h).

Figure 1: (a) Brass insertion tray and its dimensions and (b) subsampler with aluminum plate, thin silicon sheet, and paper label; modified from ref. [13].

Figure 2: Subsampling steps (a) split-core; (b) insertion of the subsamplers into the wet unconsolidated sediment; (c) cutting of the plastic tube wall; (d) subsamplers and sediment separated from the split core; (e) removal of the excess sediment from the top of the subsampler; and (f) subsample before removal from subsampler; modified from ref. [13].
3.3 Impregnation and polymerization

Dry- and gypsum-embedded subsample is arranged in a rectangular embedding box and then impregnated with low viscosity Epoxy 2000 resin in two stages (Figure 3i). Epoxy mixture is prepared from two components (resin and hardener) and mixed in a ratio of 100 g of Epoxy-2000 resin and 48 g of Epoxy-2000 hardener. The epoxy solution is added to the embedding box leaving the top of the sediment uncovered with epoxy inside the vacuum chamber (~0.8 bar) for about 15 min. This allows the air bubbles better to escape through the top of the sediment while the epoxy resin penetrates the sediment from the sides through gypsum. Then, the sample is completely covered by the rest of the prepared resin to achieve a 3 mm thick layer above the sediment surface, again in the vacuum chamber for 15 min, and then left to cure for about 24 h at room temperature. The higher...
4 Discussion

The impregnation method for the study of laminated deposits has a long history, and several protocols to achieve high-quality thin sections for the study of annual cyclicity were described (for review see ref. [14]). The last very sophisticated protocol was proposed by ref. [18] which modified Lamoureux’s protocol [13] in subsampling and dehydration steps using a cheese cutter for better separation of subsample from core and freeze-drying to remove water from the subsample by sublimation. Using our alternative gypsum method, we successfully prepared over 80 polished thin sections, and here, we described approach changes method of ref. [13] in four steps (Table 1).

4.1 Core cutting

Instead of the plastic wrap, we use the silicon sheet which is one of the best alternatives for separating the aluminum plate from the sediment subsample (Figure 5a). We see its advantage in easy cleaning, reusability, and non-adhesiveness to the sediment surface. The second change is in the separation of subsampler from the core. We do not use the cheese knife [18], metal jig [13], or fishing line [19], but for removing the subsample, we first cut the plastic tube wall and then the razor separates the subsample from rest of the deposit. Our practice in most cases do not disturb the subsample, and the rest of the sample can be used for other analysis (Figure 2). Even though resampling is impossible and the procedure needs more manual manipulation than U-channel sampling, it works well with short part of the cores [19].

Table 1: Steps in impregnation of the unconsolidated limnic sediment used for the preparation of the thin sections compared to ref. [13]

<table>
<thead>
<tr>
<th>Impregnation steps</th>
<th>Lamoureux [13]</th>
<th>Gypsum method (this article)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Core cutting</td>
<td>Manual core splitter</td>
<td>Manual core splitter</td>
</tr>
<tr>
<td></td>
<td>Subsampling boxes with plastic wrap</td>
<td>Subsampler with silicon sheet</td>
</tr>
<tr>
<td></td>
<td>Separation using jig</td>
<td>Separation using saw</td>
</tr>
<tr>
<td>Dehydration</td>
<td>Acetone in the liquid phase</td>
<td>Gypsum embedding</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Separation using saw</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Natural dehydration (1–2 days)</td>
</tr>
<tr>
<td>Impregnation</td>
<td>Acetone-resin exchange under vacuum</td>
<td>Embedding with epoxy resin under vacuum chamber</td>
</tr>
<tr>
<td>Polymerization</td>
<td>Oven (20–40°C)</td>
<td>At room temperature (20–24°C)</td>
</tr>
</tbody>
</table>
4.2 Dehydration

Subsample dehydration process is an important step for the preparation of thin-section and to achieve sediment without cracks and the deformed surface is very crucial. Various dehydration methods have been developed through time and have different properties (Table 2): freeze-drying \([10,17,20,21]\), acetone in the liquid phase \([13,21–24]\), and critical point drying \([17,25]\). Repeated exchanges of acetone \([13]\) are a time-consuming procedure and require a large quantity of solvent \([14]\). Our first attempt to produce the thin sections followed the acetone method \([13]\), but it caused the large cracks in the laminae and the samples were unusable. So to avoid such cracks, we adopted a process of the subsample stabilization using the ordinary gypsum, which took about 1 h to harden (Figure 5b). We tested the gypsum of two different producers. To obtain the semiliquid consistency, we followed the producer instructions and the gypsum with longer time of hardening was more suitable for work. The gypsum embedding keeps the sediment partially moist, and later, the Epoxy resin impregnation

![Figure 5: Steps and results of gypsum method: (a) subsamplers pushed lengthwise into the wet sample; (b) subsamples embedded with gypsum; the cracks in the middle formed before cutting and embedding; (c) impregnated and un-polished subsample slab; (d) subsamples after the first epoxy resin impregnation showing the penetration depth; light places still non-impregnated; (e) polished thin-sections prepared from unconsolidated laminated sediments from Tatra Mts. lakes.](image)

<table>
<thead>
<tr>
<th>Method</th>
<th>Advantages</th>
<th>Disadvantages</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>Freeze-drying</td>
<td>Fast and low recurring costs; minimal cracks</td>
<td>Cracks in sediments with high contents of organic matter</td>
<td>[10,17,20,21]</td>
</tr>
<tr>
<td>Acetone in liquid phase</td>
<td>Gentle dehydration; rare cracks; high quality samples</td>
<td>Collapsing of coarse sediments; expensive due to acetone consumption; time consuming (7–10 days)</td>
<td>[13,21–24]</td>
</tr>
<tr>
<td>Critical point drying</td>
<td>Gentle dehydration; minimal cracks</td>
<td>Changes in the physical properties of pore solutions; possible damage due to fluid movements</td>
<td>[17,25]</td>
</tr>
<tr>
<td>Gypsum embedding</td>
<td>Gentle dehydration; easy manipulation with soft and fresh sediment rare cracks; fast curing time; suitable for acetate peel replicas</td>
<td></td>
<td>This article</td>
</tr>
<tr>
<td>Embedding material</td>
<td>Catalyst</td>
<td>Advantages</td>
<td>Disadvantages</td>
</tr>
<tr>
<td>---------------------</td>
<td>-----------------------------------</td>
<td>----------------------------------------------------------------------------</td>
<td>-------------------------------------------------------------------------------</td>
</tr>
<tr>
<td>Spurr low-viscosity resin</td>
<td>Dimethylaminoethanol</td>
<td>Can be performed without special equipment; easily adaptable in laboratory</td>
<td>Hydrophobic; suspected to be carcinogenic; toxic; suspected to provoke allergic reactions</td>
</tr>
<tr>
<td>Araldite</td>
<td>Accelerator 964 C DMP-30</td>
<td>Moderate costs; fast hardening</td>
<td>Suspected to be carcinogenic; suspected to provoke allergic reactions</td>
</tr>
<tr>
<td>Palatal</td>
<td>Cyclohexanon peroxide</td>
<td>Good quality; applicable to dry sample; can easily be thinned with acetone and the curing time is long enough to allow complete impregnation</td>
<td>Suspected to be carcinogenic; prolonged ‘floating’ of the sediment over palatal; slow, requires at least 2 weeks; requires dehydration by acetone</td>
</tr>
<tr>
<td>Castolite resin</td>
<td>Oxydation</td>
<td>Undisturbed soil samples; low viscous for easy penetration</td>
<td>—</td>
</tr>
<tr>
<td>Methyl methacrylate Crystic</td>
<td>Benzoyl peroxide</td>
<td>Suitable for the embedding of porous material</td>
<td>Lack of autopolymerisation</td>
</tr>
<tr>
<td>Epoxy 2000 resin</td>
<td>Epoxy 2000 hardener</td>
<td>Fast hardening; can be performed without special equipment; easy preparation; curing at room temperature, low water content within the sediment does not influence the hardening process</td>
<td>Distortion and wraping occurs on curing; anaerobic polymerization is advised for curing</td>
</tr>
<tr>
<td>LR white hard resin</td>
<td>Benzoyl peroxide</td>
<td>Requires a minimum of equipment</td>
<td>Needs thinning with acetone</td>
</tr>
<tr>
<td>Epoxy 2000 resin</td>
<td>Epoxy 2000 hardener</td>
<td>Requires a minimum of equipment</td>
<td>Needs thinning with acetone</td>
</tr>
<tr>
<td>LR white hard resin</td>
<td>Benzoyl peroxide</td>
<td>Requires a minimum of equipment</td>
<td>Needs thinning with acetone</td>
</tr>
</tbody>
</table>
could be carried out. The dry gypsum embedded sub-sample can be used to produce acetate peels. In the gypsum procedure, the crack formation in sediment is remarkably reduced and the time for dehydration can be reduced. To minimize the crack formation, the paper label is left on the fresh sediment surface which acts as “patch” against cracking, prevents the sample surface from quick drying and holds the sediment during the drying (Figure 3g and h).

4.3 Impregnation

The use of polymers in impregnation of the unconsolidated deposit is a standard practice, and each polymer has its own properties (Table 3). Our laboratory has the best experiences with the Epoxy 2000 resin for the preparation of the petrological and paleontological thin sections, and we used this resin for impregnating the laminated deposits. To gain the total impregnation, an
embedded subsample is left in the highest possible vacuum; in our case, the vacuum chamber reaches pressure conditions \(-0.8\) bar. The vacuum condition produces a slower flux in resin and saturates the porous sediment due to the capillarity of the sediment and the low viscosity resin embeds the unconsolidated sediment \([13]\). The embedding box can be reused for next sample.

When impregnating the unconsolidated laminated deposit, the resin penetrates the sample only 2 mm from the surface \((\text{Figure 5d})\), but the depth of impregnation depends on porosity and sediment material composition (resin goes deeply in gyttja). Therefore, during the lapping of the surface before gluing the sample to glass slide, we have to repeat the impregnation 1–2 times accompanied by gentle lapping to preserve sedimentary structures of the impregnated subsample.

### 4.4 Polymerization

Embedded subsample is left to cure under room temperature, which is easy but little time-consuming whereas there are other methods work with higher temperatures \([13]\) that could lead to bubbles development in the resin. This process requires the maximum time of 24–36 h, depending on the amount of polymer used to impregnate the sediment.

The impregnated sediment (Figure 5c) is diagonally cut into standard \(2.5 \times 4.5\) cm slabs (Figures 5d, e, and 6), but also larger thin-sections could be produced (it depends on the laboratory equipment). For grinding of the surface before gluing, we use the SiC powder of the grain sizes F320, F600, F800, and for the finishing SiC powder F800, F1200. For final polishing, we use the water-based 1-micron polycrystalline diamond suspension. Thin-sections prepared by this method can be used for elemental analysis using micro-XRF (Figure 7) and X-radiographic images \([10,17,32]\) to study the paleoclimate and seasonal indicators in these laminated deposits.

### 5 Conclusion

Gypsum can effectively help in dehydration and stabilization of the fine-laminated clastic deposit. The gypsum
embedding keeps the sediment partially moist, and the sample is ready for impregnation with resin. After impregnation, the slab of $10 \times 2.5$ cm is prepared for elemental mapping on micro-XRF, X-radiographic, image analysis, or production of the thin sections. The method needs a minimal infrastructure and makes the entire process of preparation of polished thin sections faster and can be done within 3 to 4 days. The gypsum dehydration was performed on laminated clastic deposits and its use on biochemical- and organic-rich laminated sediment should be tested.

Acknowledgments: This work was financially supported by the project APVV-15-0292 and by the projects: Centre of Excellence for Integrated Research of the Earth’s Geosphere (ITMS: 26220120064) and Completion of technical infrastructure for research of geodynamical processes and global changes in Earth’s history (ITMS: 26210120013), which had been co-financed through the European Regional Development Fund.

Author contributions: Ramachandran Dhavamani – Cooperation in the process of preparation of the thin-sections and study of the laminated deposits. Golej Marián – Preparation of the polished thin-sections, technical support. He proposed the gypsum for the dehydration process and the Epoxy 2000 resin for impregnation process. Starek Dušan – Expertise in sedimentology and microscopic studies of the clastic deposits. Pipík Radovan – supervising the technical work, project leader.

References


