

Impregnation method for detecting annual laminations in sediment cores: An overview

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Abstract

Annually laminated sediments can provide an absolute time scale (by varve counting) and a high-resolution palaeoclimate information (from varve thickness). Both types of information may be directly measured from sediment core surfaces. In this paper, we stress that varve counting and varve thickness measurements derived from fresh core surfaces could not systematically reveal the internal sedimentary structure, even if assisted by high resolution image analysis. We present an example of a homogeneous sediment core for which the varves were only observable after core impregnation and polishing steps. Because the impregnation methods are not yet standardized, the aim of this paper is to give an updated review of the methodology. In this review, we present the major critical points during impregnation steps. In particular, we focus on all of the post-treatment sediment disturbances that can alter the laminated micro-structure and, consequently, varve measurements. Finally, we propose a modified impregnation protocol, especially adapted for tracking varved intervals in long cores.

Keywords: laminated sediments ; polymerization ; thin-section ; varve dating ; palaeoclimate analysis

1. Introduction

High resolution palaeoclimatological data reconstructed from annually banded archives (e.g., tree rings, speleothems, corals, ice cores, varves) constitutes the basis for inter-comparison of past climate variability (e.g., Holivar programs: Battarbee, 2003). Of these approaches, the marine and lacustrine varved cores sequences represent some of the highest-resolution materials useful for tracking short-term climate changes calibrated with absolute time scales (cf., Kemp, 2003). Because such archives are wet and unconsolidated, counting the varves directly from the fresh core surface may underestimate the correct number of varve years (Lotter and Lemcke, 1999). One particularly attractive method consist of impregnating the sediment core with polymers in order to more easily prepare thin-sections (Kemp et al., 2001). This step is essential for being able to count each "varve year" (e.g., Lamoureux, 2001). Since the 1960s, the evolution of impregnation methods has been strongly linked to the development of innovative technique (e.g., sampling devices, cryogenic and vacuum technologies, polymers, etc.). Originally, sediments were simply covered by resins, with impregnation assisted by gravity (Heezen and Johnson, 1962; West, 1966; Shannon and Lord, 1967; Moreland, 1968; Orlansky, 1968). Later, total impregnation techniques developed by pedologists for soil micromorphology (Altmuller, 1962) were adapted in order to emphasize the muddy material (Ashley 1973). At the present there are still two embedding methods in existence, but they have not been standardized (Lotter and Lemcke, 1999). In this paper, we first review the available protocols for resin impregnation of varved sediments. We then propose a revised method that may be useful for tracking the internal sediment structure in long marine cores. The modified criteria are discussed in terms of efficiency for varve identification, especially in clayey material.

2. Review of the impregnation methods

Impregnation methods require three major steps, namely sampling, sediment dehydration, and polymerization. Descriptions of the protocols are presented in the accompanying literature review (Table 1). Note that this review does not include the sediment disturbances related to the different drilling systems (see Lamoureux, 2001).

2.1. Core sampling

Core sampling is an important part of this procedure because the cohesion of the muddy material must be maintained. Conventional sampling methods (i.e., syringes, spatulas, plastic bags) cannot be used because they are destructive. Published methods to collect undisturbed samples from the cores used overlapping aluminium foil boxes pushed down the working sections. The boxes are designed to accommodate the dimensions of thin-sections, or longer (e.g., 180 mm long \times 20 mm wide \times 7 mm deep; Francus and Asikainen, 2001). The sub-sampling boxes are easily extruded from the cores without damage to the sediment material by using a monofilament, a square of sheet metal (Lamoureux, 1994), or by using an electro-osmotic core cutting device (Chelmik, 1967; Bouma, 1969; Sturm and Matter, 1972; Schimmelmann et al., 1990; Francus and Asikainen, 2001; Kemp et al., 2001).

Table 1 Review of published methods for sediment core impregnation

Impregnation step	Literature protocol	References
Core cutting	Electro-osmotic core cutting	Schimmelmann et al. (1990)
	Frozen core cutting	Lotter and Lemcke (1999)
	Sub-sampling boxes	Francus and Asikainen (2001)
Dehydration	Acetone in liquid phase	Conway (1982), Clark (1988)
	Acetone in vapor phase	Camuti and McGuire (1999)
	Freeze-drying	Pike and Kemp (1996)
Impregnation	Acetone-resin-exchange	Lamoureux (1994)
	Under vacuum	Lotter and Lemcke (1999)
Polymerization	Oven (20-40 °C)	Ashley (1973)

See text for explanations.

2.2. Dehydration techniques

Core material composed of fine clays with unstable physical properties cannot be air-dried in the same way as can sandy sediments. Conventional drying procedures (e.g., oven-drying) are responsible for shrinkage and distortion of the clay matrices (e.g., Tanner and Leong, 1995). One solution for preserving the sediment microfabric during the drying phase is to decrease the water content with successive solvent baths, e.g., by water/acetone removal (e.g., Pusch, 1999). The acetone exchange method may be done in liquid-phase (Conway, 1982) or vapor-phase (Camuti and McGuire, 1999). In the use of liquid phase exchange, sediment is soaked in a container with acetone. One sample requires eight consecutive acetone baths over a period of one week, while measuring the baths specific gravity in comparison with pure acetone (Clark, 1988). In the case of vapor phase methodology, sediment is placed under acetone vapors in a dry chamber for three weeks (Camuti and McGuire, 1999).

The other solution to the shrinkage problem is the lyophilization (freeze-drying) technique that evacuates the interstitial water by sublimation (Bouma, 1969; Merkt, 1971; Crevello et al., 1981; Kuehl et al., 1988; Francus, 1998; Kemp et al., 2001). First, the liquid phase is converted into a solid phase (i.e., ice). The sediment is floated over liquid nitrogen at -196 °C by using, for example, styrofoam plates (Lotter and Lemcke, 1999). Second, the ice is sublimated in a freeze-dryer chamber at a pressure level below the triple point. The produced H₂O vapors are trapped into a condenser until the end of the drying cycle (12-24 h).

2.3. Saturation with polymers

Two standards protocols exist for this important step. The first method is applied on acetone dried material, in which the sediment is impregnated by acetone-resin-exchange (Clark, 1988). The acetone is progressively exchanged for a low viscosity polymer (Lamoureux, 1994; see also the appendix in Dean et al., 1999). Polymer resin penetrates by capillary action into the sediment in several stages (i.e., 25%, 50%, 75%, and 100% of resin-acetone concentration) to avoid osmotic shock (Jim, 1985).

The second method may be applied on acetone or freeze-dried sediments. The sediments are impregnated under vacuum to avoid bubbles. The samples are placed within a solvent resistant mould in a vacuum chamber in order to be progressively saturated with polymers (e.g., resin added in four steps). The sediment is impregnated under vacuum conditions (e.g., 800 mbar, Camuti and McGuire, 1999). Pumping and venting cycles may be used to achieve the desired saturation (Tiljander et al., 2002).

2.4. Polymers

Numerous impregnation substances have been used by laboratories specializing in the preparation of unconsolidated material. For example, Carboax (Renberg, 1981), Vestopal H (Altmuller, 1962; Ashley, 1973), methyl methacrilate (Palle et al., 1985; Murphy, 1986; Pusch, 1999), Crystic (Conway 1982), and LR hard white resin (Ferrow and Roots, 1989) have all been employed. The standard resins most used for sediment impregnation are Epoxy products (Martin et al., 1979; Crevello et al., 1981; Jim, 1985; Clark, 1988; Lamoureux, 1994; Francus, 1998; Kemp et al., 2001).

3. Revised method

We use long plastic U-channels (100 cm length \times 5 cm wide \times 1.5 cm deep) to extract a one meter core sequence at one time (Fig. 1). The U-channels are slowly pushed down the mud by hand and are then extruded with a monofilament. The cores are inclined 90° to facilitate the extrusion. The samples are immediately frozen in a deep-freeze with the initial water content. Once the interstitial water is frozen (after few hours), the sediments may be sectioned without risk of collapse. The samples are dried by the lyophilization (freeze-drying) methods proposed previously by Pike and Kemp (1996) or Francus (1998), but with a modified sublimation rate.

Lyophilization cycles consist of three phases: (1) freezing, (2) primary drying, and (3) secondary drying. (Phase 1) U-channel are frozen in a deep-freeze at a temperature between -80 to -20 °C only, depending on the freeze dryer aptitude (see discussion paragraph) and water content (high-water content samples must be frozen quickly). (Phase 2) The interstitial ice is sublimated according to the vapor pressure above ice, as depicted in Fig. 2. For example, for a sample frozen at -40 °C, the vapor pressure above ice is 10^{-1} mbar. (Phase 3) During the secondary drying, water sublimates and sample temperature decreases. To compensate for the loss of energy, the heat supply is increased proportionally. After 12 to 24 h the sample is dried (sublimation rate is controlled from an ice-track). Because freeze-dried clayey sediments are soft and sensitive to air-humidity, we always treat them without delay for consolidation by impregnation. The samples are placed into a vacuum chamber within a solvent-resistant mould. We use a vacuum apparatus similar to that used for the drying step, i.e., a vacuum chamber equipped with a rotary pump ($5 \text{ m}^3 \times \text{h}^{-1}$), and a condenser to trap the organic vapors (polymers and acetone), that may destroy the pump (Fig. 1). The vacuum level is applied according to sediment textures: a low vacuum level close to atmospheric pressure is used for the dense clay, whereas up to 700 mbar pressure is used for the coarser textures (empirically determined, see discussion section). The sediments are saturated with low-cost resins like Norsodyne S 2010 V (supplier: Cray Valley) or TRA (supplier: Finres group) polymers under vacuum. We mix TRA or Norsodyne with 0.2% of catalyst (Interox), 0.1% of accelerator (Octoate Cobalt 49), and less than 10% diluents (acetone). The polymer is progressively added in four steps (after 30 min and successively at 1, 2 and 4 h), as the resin rises drop by drop from the bottom to the top. During the polymerization, the temperature increases and the polymerization rate decreases. The saturated samples may be placed in a dry-oven at 40 °C to for compensate the energy lost and to accelerate the polymerization rate. The accuracy of the varve measurements is improved if the treatment is performed on fresh core samples, i.e., having an initial water content.

Fig. 1. Sediment pre-treatments for core polishing step. The numbers correspond to the different preparation step: sampling (1-2), dehydration (3), impregnation (5), and polymerization (6). 1 and 2: Core cutting with a fisher wire and sampling with a U-channel (100 cm). 3: Freezing within a deep-freezer at selected temperature. 4: Ice sublimation in a dry chamber, H_2O vapor is trapped into a condenser. 5: Dried sample saturated with resin under vacuum, acetone is trapped into a condenser. 6: Polymerization in a dry-oven at 40 °C. A: Vacuum chamber equipped with a manometer; B: Condenser at -50 °C; C: cooler and rotary van vacuum pump ($5 \text{ m} \times \text{h}^{-1}$); D: Thermic probe. For long sediment core preparation, note large size chambers will avoid time-consuming sub-sampling.

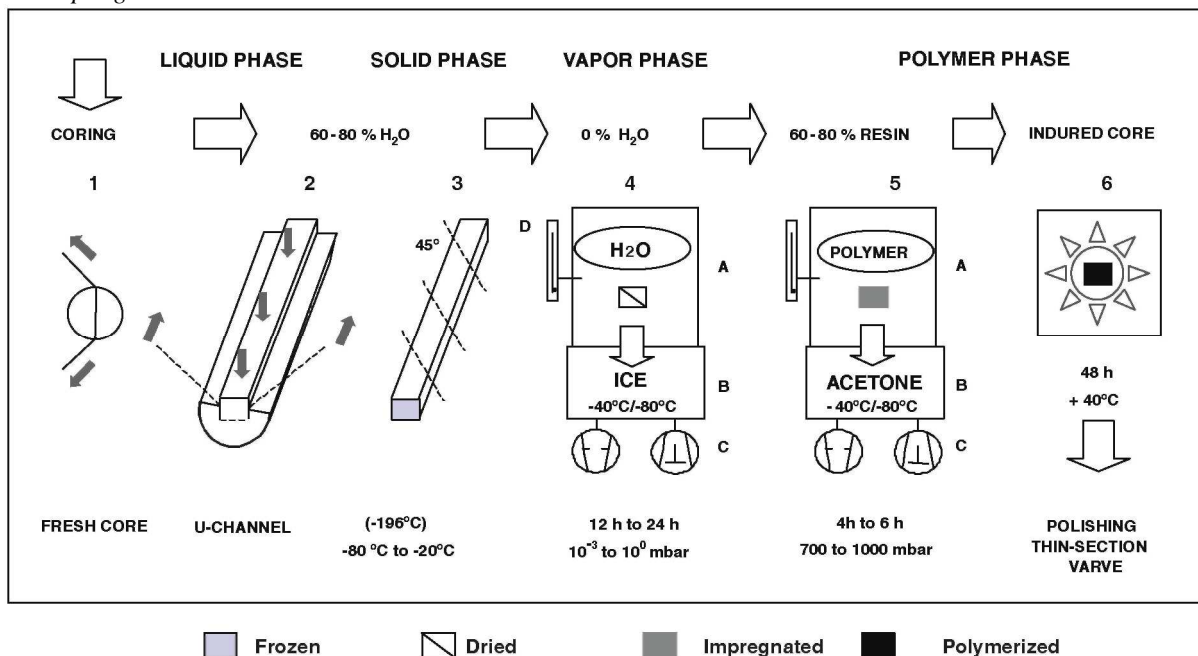


Fig. 2. Diagram for determination of the vapor pressure above ice to adjust the drying chamber. Note that the pressure level should be selected as high as possible, but not higher than the pressure corresponding to ice temperature.

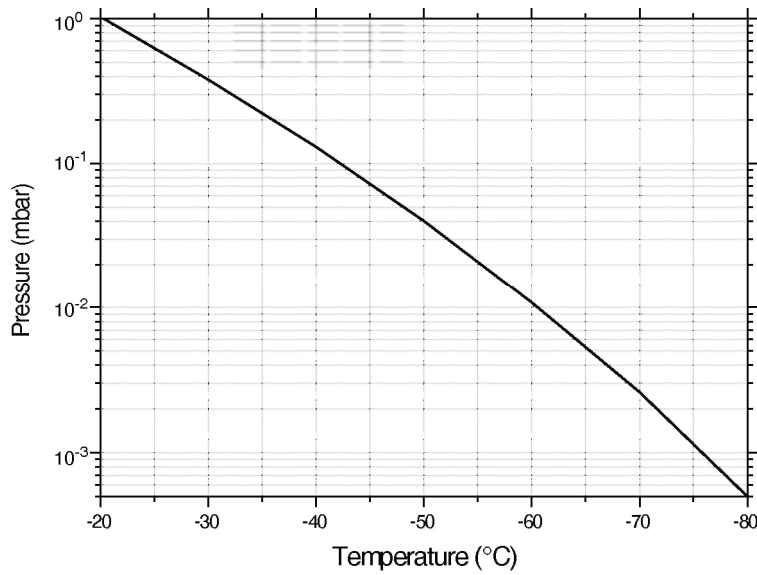
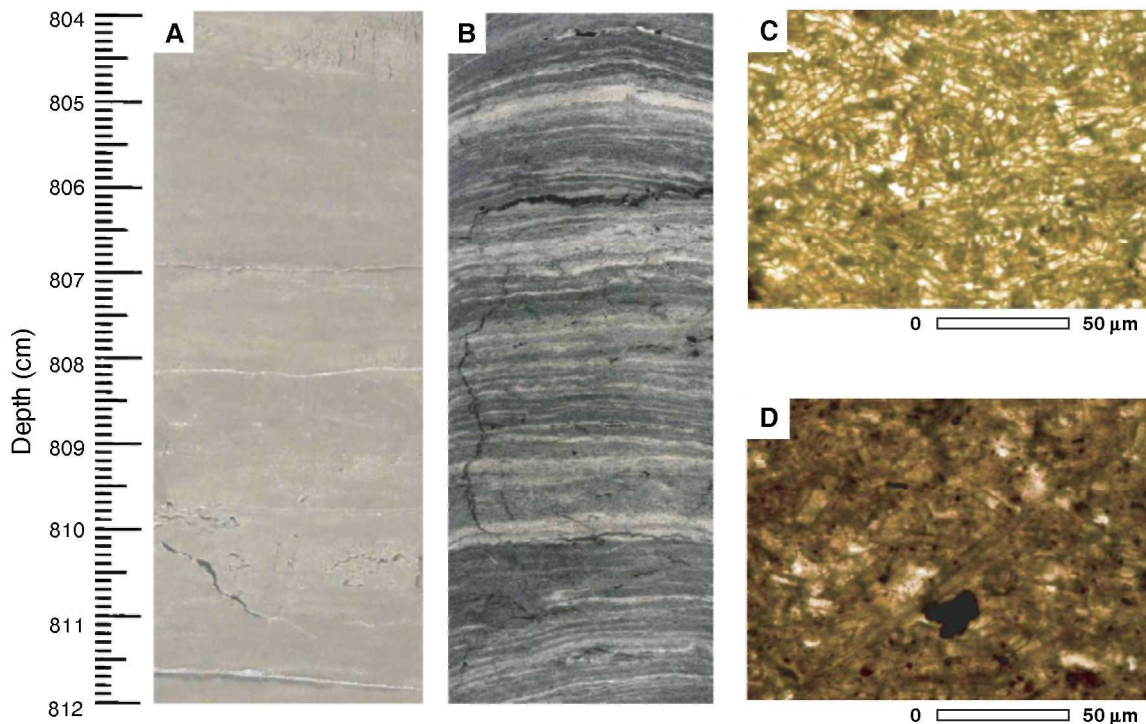


Fig. 3. Core scan of biogenic lacustrine sediments (Lago Puyehue, Late Glacial, (PUII station at 804-812 cm, Chili, 40° S). The pictures on the left show different quality of sedimentary resolution according to cut surface of the core before (A), and after (B) the pre-treatment by polymerization. Macroscopically the sediment does not present apparent seasonal laminations. The enlarged pictures on the right are thin-section images of typical seasonal doublets formed by : a light diatom-rich layer (C), and a dark layer enriched with fine-grained organic material. It would have not be possible to apply varve-counting methods directly from the fresh core.

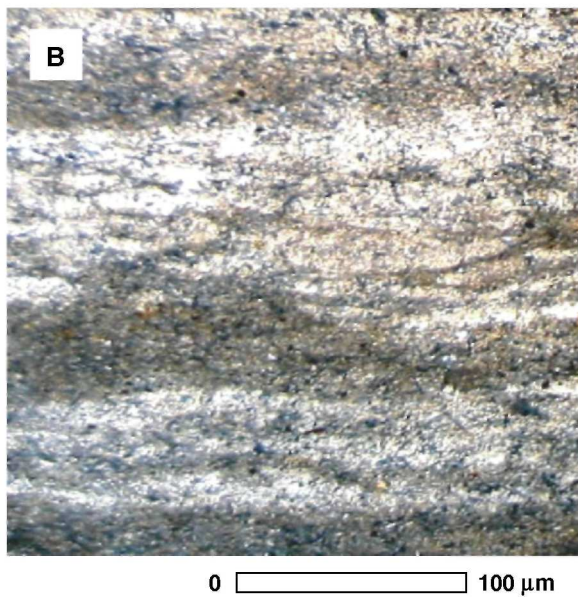
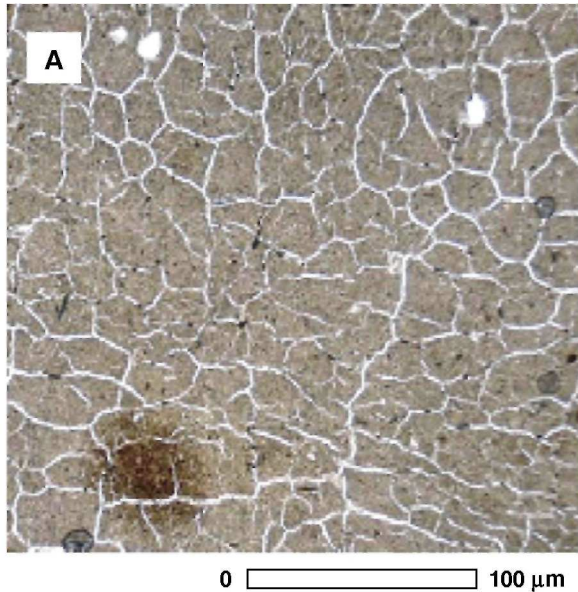


4. Discussion

The revised method described herein has been employed on lacustrine clayey sediments sampled from Southern Chile (OSTC-ENSO-Chile project) and from Lake Baikal (CONTINENT project) sediments. The method, when applied on sediments assumed homogeneous at a macroscopic scale, revealed internal finely laminated structure

(see Figs. 3 and 4). The varved sections from the Chilean lake were determined only after the total impregnation/polishing of the core length (Fig. 3). The thin layers in Lake Baikal sediments were only evident after thin-section preparation (Fig. 4). In the discussion below, we explain our impregnation strategies.

Fig. 4. Optical microscope pictures taken from Lake Baikal thin-section of clayey material. (A) Note the local effect of liquid nitrogen shock on the micro-structure, (B) thin laminations tracks are observed from a thin-section prepared with the revised protocol. A: VER-98-1-3, Academician Ridge (290 cm depth); B: CON01-603-5, Continent Ridge (29 cm depth).



4.1. The U-channel sampling strategy

The sub-sampling of a varved sequence is the most difficult step to perform; and it may cause irreversible deformation of the material. One disadvantage of the sub-sampling box method is the large number of sediment transfers and hand manipulations, which increase risk of collapse. In addition, the preparation of sub-sampling boxes is a time-consuming and a fastidious operation for long cores that increases the risk of air-drying/oxidation of the core surface (i.e., varve alteration). The U-channel, similar to those used for paleomagnetic studies, allows the sampling of a one meter core section at once, reducing the total number of sediment transfers and hence reducing the chances of disrupting the primary sediment fabric within the cores. Once frozen, the hardened sediment may be continuously sub-sectioned per 45° edge if necessary (Fig. 1). Smaller U-channels should be

used inside a small gravity corer, or sediment can be taken within the plastic corer without sub-sampling.

4.2. Selection of the dehydration technique

The use of acetone methods in organic-rich sediments may modify the volume of the samples by extraction of the soluble organics. For the preparation of long cores, the acetone methods are time-consuming (several weeks), and they require a large volume of volatile, noxious and inflammable solvent. For example, four hundred liters of acetone are necessary for a 10 m long core in liquid phase, requiring adapted laboratories with powerful extraction hoods. In liquid phase the acetone may cause sediment collapse. In contrast, the lyophilization method is rapid and safe (no particular safety precautions). The sediment is dried without dissolution risk of organic phase, and without liquid phase, as the water content is frozen and then sublimated.

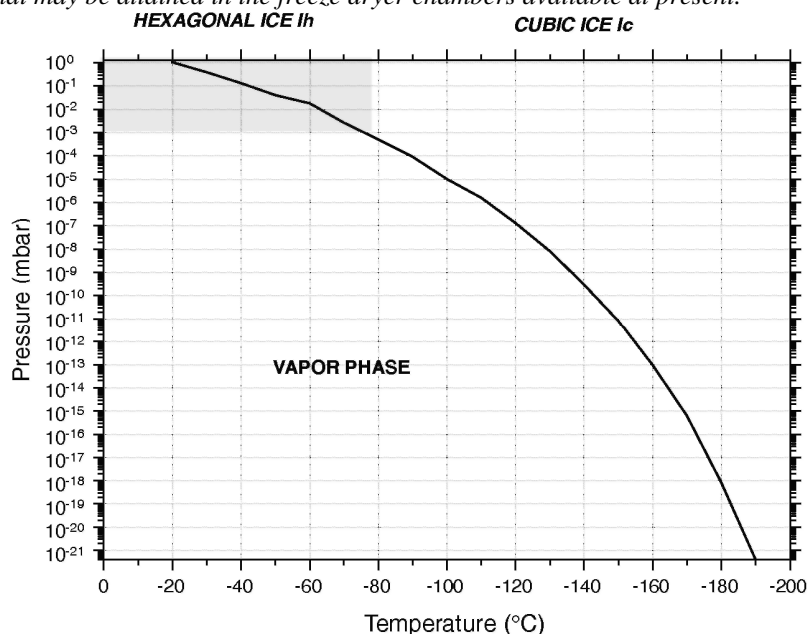
4.3. Selection of the freezing technique

With standard techniques, the water content is quickly frozen at low temperature in liquid nitrogen (-196 °C). The quick decrease of the temperature allows for the growth of small cubic ice in the sediment structure, rather than larger hexagonal ice crystals (Francus, 1998). However, we retain two problems with this technique: (1) liquid nitrogen shock, and (2) sublimation at very low temperatures. (1) The shock produced by liquid nitrogen may involve development of microscopic cracks over the sediment matrix that partly erase the laminated microstructure (Fig. 4A). Although the "shock freeze-drying" method in liquid nitrogen is a rapid procedure, we do not recommend its use as it is a dangerous product to manipulate (e.g., anoxia risk). (2) During lyophilization, the temperature has to be kept constant over the primary drying phase in the freeze-dryer chamber (e.g., Zhai et al., 2003). However, the temperature of the condensers (-40 to -80 °C) is higher than -196 °C, thereby leading to a re-crystallization.

4.4. Temperature and freeze dryer performances

The highest frozen temperature is determined according to the collapse/eutectic point, e.g., -22 °C in salt water. Lowest temperature is determined according to the condenser and vacuum equipment performances (e.g., Beckett, 1951). The temperature of the condenser determines the constant temperature in the chamber (e.g., -50 °C with Lyovac GT3). The pump rate determines the ultimate sublimation pressure above ice that may be reached (e.g., 10^{-2} mbar with Lyovac GT3). The deepest vacuum level attained by most of the available freeze-dryers chambers is no more than 10^{-3} mbar. This limit corresponds to the highest pump efficiency and pressure resistance of the chamber. For instance, the maximum pressure above ice for sublimation at -196 °C (cubic ice) is $2.2 \cdot 10^{-24}$ mbar, which is an unrealistic high vacuum unattainable with existing equipment. At present, the optimum of sublimation rate is obtainable only for temperatures between -20 to -80 °C (i.e., above the cubic ice phase — see Fig. 5). Freeze drying devices with liquid nitrogen used in the condenser for trapping moisture may be used for lyophilization at very low temperature (Rindler et al., 1998), but vacuum chamber aptitude still controls the ultimate sublimation pressure. A 10^{-3} mbar freeze dryer performance corresponds to ice at -80 °C.

Fig. 5. Sublimation curve for low ice temperatures. The shadowed area represents the vapor pressure above ice that may be attained in the freeze dryer chambers available at present.



4.5. Vacuum conditions for the impregnation

The impregnation step has to be performed under strictly controlled pressure conditions. Based on our experiments, the pressures to apply vary according to the sediment textures, and mainly porosity (Fig. 6):

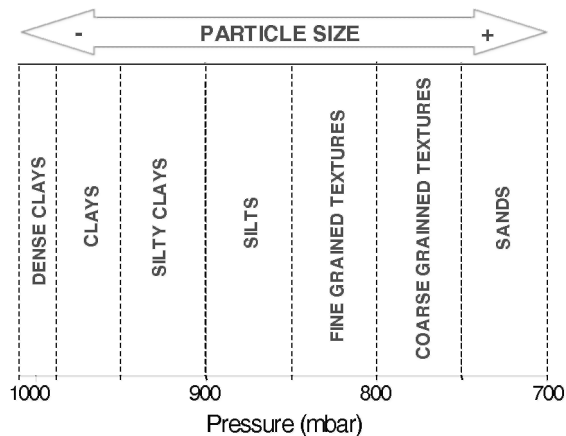
- for clayey texture, a pressure level close to atmospheric conditions is sufficient;
- for coarser material, the pressure level has to be increased up to 700 mbar.

For dense clayey structures, atmospheric pressure allows for satisfactory impregnation to be performed.

The sediment is saturated only by capillarity, but the polymer must be low viscosity (Lamoureux, 1994). A lower pressure will produce a faster resin flux that could damage the sediment fine texture, due to the low porosity. For silts and fine grained materials, the vacuum level has to be lower than 1013 mbar. Indeed, the vacuum level must produce a slow flux of resin able to saturate the higher sediment porosity. Coarser material requires the highest vacuum levels, as the resin flux must fill the largest pores.

The rate of the resin flux may be visually controlled and is based on the boiling point of the resin mixture under vacuum conditions. The pressure level should not exceed the boiling point otherwise bubbles will destroy the matrix (i.e., the resin flux is too high for the pore dimensions). Note that the boiling point varies according to the acetone-resin ratio in the mixture, and also to temperature-pressure conditions in the chamber. For resin mixed with 10% acetone at 15 °C, we empirically observed that the boiling point is reached at around 700 mbar (Fig. 6).

Fig. 6. Diagram for determination of the vacuum level to apply according to the different sediment textures. Note that the limits are determined from a 700 mbar boiling point of the acetone-resin mixture.



4.6. Polymer mixtures

One of the most widely used Epoxy resins is probably Spurr, which is actually one of the most available fluid resins for the saturation of dense clayey material (Lamoureux, 1994; Pike and Kemp, 1996; Kemp et al., 2001). For the preparation of the polymer mixture, the most crucial step is the catalyst/accelerator ratio adjustment. A high catalyst/accelerator percentage ratio results in a fast rate of polymerization, and a bad quality impregnation (see also Ashley, 1973). Moreover, increasing acetone diluents above 10% result in cracks due to solvent evaporation after polymerization is over (note that styrene monomers as diluents should be avoided due to cancer risk for the operator). Increased temperature above 40 °C leads to fast polymerization rate and involves cracks (see also Ashley, 1973).

5. Conclusions

Sediment impregnation is the first step necessary for investigating laminations in cored sediments. This paper provides an outline of the different procedures in order to prepare continuous polished cores sections. For each step, we emphasize minimizing post-treatment sediment disturbances. Here are the main comments:

- (1) The sampling of long sections at once (one meter U-channels) reduces the risk of disturbances.
- (2) Frozen samples may be cut without risk of collapse.
- (3) The dehydration step is the most important step for lamination thickness preservation. Acetone gives good results but necessitates hundreds liters of acetone for long cores, and use of acetone is dangerous (noxious, inflammable). Moreover, acetone also dissolves the organic material (core shrinkage). The sublimation method is

a fast method that should be adjusted according to actual equipment performances (i.e., condenser temperature, pump rate). The freezing step at low temperatures requires unrealistic vacuum conditions for sublimation. Even if LN₂ is used in the condenser for trapping moisture, the maximum vacuum chamber aptitude is 10⁻³ mbar, which correspond to ice at -80 °C.

(4) The frizzing step should be done according to condenser temperature, which controls the temperature in the chamber.

(5) For the impregnation step, the vacuum level has to be adjusted according to sediment texture. We note the vacuum conditions increase with increasing particle size and porosity. Dense clayey material may be impregnated at ambient condition, whereas coarser sediments require deeper vacuum.

(6) Finally, thin-sections are prepared with standard protocols such as those used for rock samples. We prefer large thin-sections (140 × 65 mm) that allow a better visual control of lateral variations of the laminated sediments.

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