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SEPARATION AND ^{14}C DATING OF PURE POLLEN FROM LAKE SEDIMENTS: NANOFOSSIL AMS DATING

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ABSTRACT. We have developed and tested a practical device for manually separating pollen from pollen concentrates in sufficient quantity for AMS ^{14}C dating. It is a combination of standard, commercially available equipment handled in a clean room by an individual trained to recognize pollen. A typical example requires about 15–20 h of hand-picking under the microscope. We show the usefulness of this procedure with results on a mid-Holocene segment from a core from Mono Lake. Sediments from this hardwater lake contain pollen and finely disseminated organic matter, but no macrofossils. The pollen dated *ca.* 1000 yr younger than the bulk sediment. The sediment "date" is most likely affected by incorporation of limestone-derived carbon, and is erroneously old.

INTRODUCTION

The long-standing problem with ^{14}C dating of lake sediments is that events of interest, such as sedimentological and botanical changes, may not have the same apparent ^{14}C age as the bulk organic carbon fraction most often dated. Common reasons for this difference include: 1) carbon in the organic fraction derived from old groundwater or dissolution of limestone (the "hard water" effect, which yields ^{14}C dates older than correct age); 2) atmospheric carbon incorporated through fungal growth, which yields ^{14}C dates younger than correct age. Most ^{14}C dating of lake sediments occurs with analysis of terrestrial pollen, with the goal of inferring times of botanical changes. The most direct approach to ^{14}C dating these changes is by direct dating of terrestrial plant fossils. In many instances, pollen is the most abundant plant fossil adequately preserved. As typical pollen grains range from 0.01 to 0.2 $\times$ 10^{-6} g, at least 10^2 to 10^3 grains (depending on species) are required for an acceptable AMS ^{14}C date. Previous work (Brown *et al.* 1989) employed chemical and physical concentration procedures. Although a clear improvement, their pollen enrichments still contained chemically resistant non-pollen organic material, such as algal bodies, whose carbon derives from water rather than from the atmosphere. As AMS technology has decreased the amount of carbon necessary for dating without compromising precision, analysis of 100–200 μg samples has become possible. Thus, we have begun testing the practicality of manual pollen isolation.

APPARATUS AND PROCEDURES

We selected sediments at 200–215 cm depth at Mono Lake, California (Davis & Kailey 1990). This core interval contained anomalously high pine percentages and low sagebrush percentages, above a layer barren of pollen. Davis and Kailey (1990) attribute this layer to the obliteration of local vegetation by a volcanic eruption. However, the demise of local vegetation might also have resulted from a very high lake level, such as the Dechambeau Ranch High Stand (3679 $\pm$ 188 BP; Stine 1990). Mono Lake is isolated, and is fed by springs and runoff that contain terrestrial carbon. Hence, organic carbon from the lake dates *ca.* 1000 yr too old (Broecker & Walton, 1959).

The pollen in a 320-g sample from 200–215 cm was concentrated by acid digestion and screening (*cf.* Brown *et al.* 1989). Carbonates and silicates were removed with concentrated HCl and HF, cellulose and related compounds were removed by acetolysis, and humates were removed with 5%

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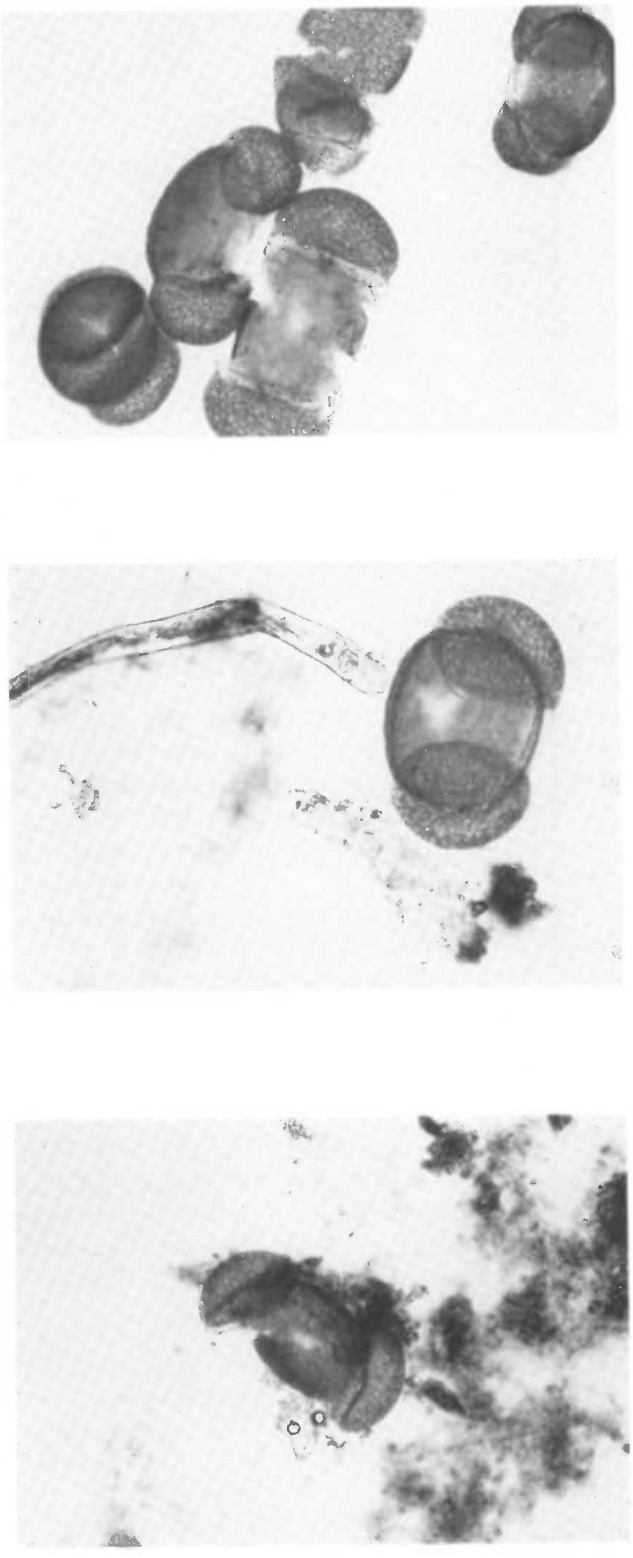

Fig. 1. Photomicrographs showing pine pollen before and after final mechanical purification. Pine pollen grains are typically 60–90 μm in longest dimension. A. Before final mechanical purification – note presence of organic detritus. B. Before final mechanical purification – note plant part. C. After final mechanical purification.

KOH. Each step was followed by repeated washing with deionized water, so soluble organic compounds also were removed. The pollen-rich residue was then filtered. The >84-μm fraction contained charcoal and plant and animal matter, the <63-μm fraction contained non-conifer pollen and colloidal material, and the 84–63 μm fraction was nearly pure pine (99%) and fir (1%) pollen. All non-pollen material was mechanically removed. A date for the 200–215 cm interval was obtained for the core bulk sediment (4605 ± 60 BP; A-6267).

The apparatus for the manual pollen purification consists of a dissecting microscope (Nikon SMZ-2T) with a 2 × objective adapter, and a special mechanical stage. The pollen-rich residue is placed on the mechanical stage in a petri dish. The microscopic contaminants then are sucked into a glass "needle" fabricated by heating and stretching a capillary tube. The needle is controlled by a micromanipulator (Narishige MO 155) with a "joystick." Under 90 × magnification, the hollow tip of the needle (inner diameter *ca.* 200 μm) is positioned next to the non-pollen material using the joystick, and suction is applied with a stationary microsyringe (Narishige 1H 5B). The chemical extraction, screening and mechanical purification were done in the University of Arizona Palynology laboratory with 10 μm-filtered air. Figure 1 consists of three photomicrographs which illustrate the pollen extract before (A, B) and after (C) mechanical purification. Note the presence of organic matter and plant parts present before final purification.

The sample was centrifuged, excess water was decanted, and the sample was dried over silica gel. Then, 500 μg of pollen were combusted in a sealed tube with CuO, and the CO_2 was reduced to graphite (Slota *et al.* 1987) for AMS dating (Donahue, Jull & Zabel 1986). In cases of minimal amounts of pollen, a slurry of purified pollen can be pipetted directly into the combustion tube, frozen and the water sublimed. The ^{14}C date on pure pine and fir pollen is 3730 ± 60 BP (AA-6837); this date is 875 yr younger than the bulk sediment result, but contemporaneous with the Mono Lake high stand (Stine 1990).

Discussion and Conclusions

We have developed procedures of physically removing non-pollen contaminants from pollen concentrates, using a micromanipulator under a dissecting microscope in a clean-room environment. The procedure requires less labor than we expected. One person can produce 2 to 3 samples of pure pollen in one week. The additional labor cost of manual purification ($100–$200 per sample) is less than the cost of one AMS ^{14}C analysis. The procedure greatly reduces the uncertainty in the interpretation of the result. In the example from Mono Lake, the bulk sediment date is, most likely, too old due to the "hard-water" effect. Lakes situated in limestone terrains receive bicarbonate derived partly from limestone dissolution. If this bicarbonate does not equilibrate with atmospheric CO_2 before aquatic plants growing in the lake utilize the bicarbonate as a source of carbon, the living plants will have a ^{14}C content between that of the limestone and the atmosphere, and consequently have an "apparent ^{14}C age." Organic matter from the remains of aquatic plants and organisms feeding on aquatic plants becomes part of the lake sediment. Macrofossils, such as wood fragments, pine cones and charcoal are often not present in core material in sufficient abundance for AMS dating, but pollen, a nanofossil, is usually present in suitable quantity. Thus, ^{14}C dating of pure pollen has the potential of being a valuable approach to assigning high-confidence dates to botanical changes observed from pollen analysis.

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