

A guide to the extraction of fossil diatoms from lithified or partially consolidated sediments

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ABSTRACT: Based on experience gained from processing samples from the Cenozoic basins of south-west Ecuador, a composite procedure for the processing of fossil diatoms from lithified or partially consolidated sediments is presented. The procedure is divided into four stages: (I) initial sample breakdown. (II) cemented rock breakdown and removal of carbonate component. (III) organic matter removal. (IV) clay removal. These are presented in the form of annotated tables linked by an accompanying flow chart.

INTRODUCTION

Marine Oligo-Miocene sediments from the Manabi and Progreso basins of south-west Ecuador were examined for diatoms. Lithologies encountered varied from partially consolidated clay-rich bentonites of the Portoviejo Formation, to lithified "chocolate-brown" shades and siltstones of the Tosagua Formation. These units contained a mixed marine fossil assemblage of foraminifera, (Whittaker 1988), radiolaria, diatoms and silicoflagellates. A biostratigraphy for the area has been erected using foraminifera (Whittaker 1988) and radiolaria (Robinson 1992); diatoms and silicoflagellates have also proved essential for palaeo-ecological interpretation and a refined biostratigraphy (Hinchey 1992).

Diatomaceous sediments from Ecuador differ considerably in their lithology and therefore react differently to conventional diatom processing techniques (Lohmann 1972, Schrader 1973). Samples from the bentonitic Portoviejo Formation broke down fairly easily, but problems were encountered in the removal of clays from these sediments. Lithified samples from the clay-rich Tosagua Formation, however, proved very problematic; conventional processing techniques were ineffective in most stages of breakdown and the soaking of samples in petroleum spirit (Abelmann 1988, Bodén 1991) also failed to disaggregate the samples satisfactorily.

OBJECTIVES

As standard diatom processing techniques failed to breakdown many of the samples collected from Ecuador, a different approach was clearly required. A literature review was conducted on previous techniques and experienced workers were asked for their comments (see acknowledgments). Techniques suggested were applied to material from Ecuador and the most effective methods noted. The result of this work has produced a composite processing procedure which can be generally applied to most types of diatomaceous sediments, including those which are lithified or partially consolidated.

The procedure is presented in the form of a flow chart (text-fig. 1) and a series of annotated tables (1-4), each table representing a particular aspect of processing. The procedure is designed to be "user friendly" and emphasizes the use of wet mounts between certain stages (e.g. removal of clays and organic matter; text-fig. 1, Tables 3 and 4) to evaluate whether or not further processing is required. The tables are designed to work independently of each other and therefore should allow workers to initially assess the

lithology of the sediments they are studying and to then determine which of the tables apply to that particular type of sediment.

ADDITIONAL NOTES TO TABLES

Table 1

Mechanical breakdown of the sediment should be done with as little damage to frustules as possible. Not all stages listed will be necessary. Physico-chemical breakdown (Table 2 (2a) and Table 3 (1a-d)) can be used for more resistant samples.

Table 2

Warming of sample + HCl (1b) is not always necessary. Heat only if very little reaction occurs.

Table 3

Addition of a few drops of methanol to a hydrogen peroxide solution (1b) prevents strong foaming in organic rich samples.

Table 4

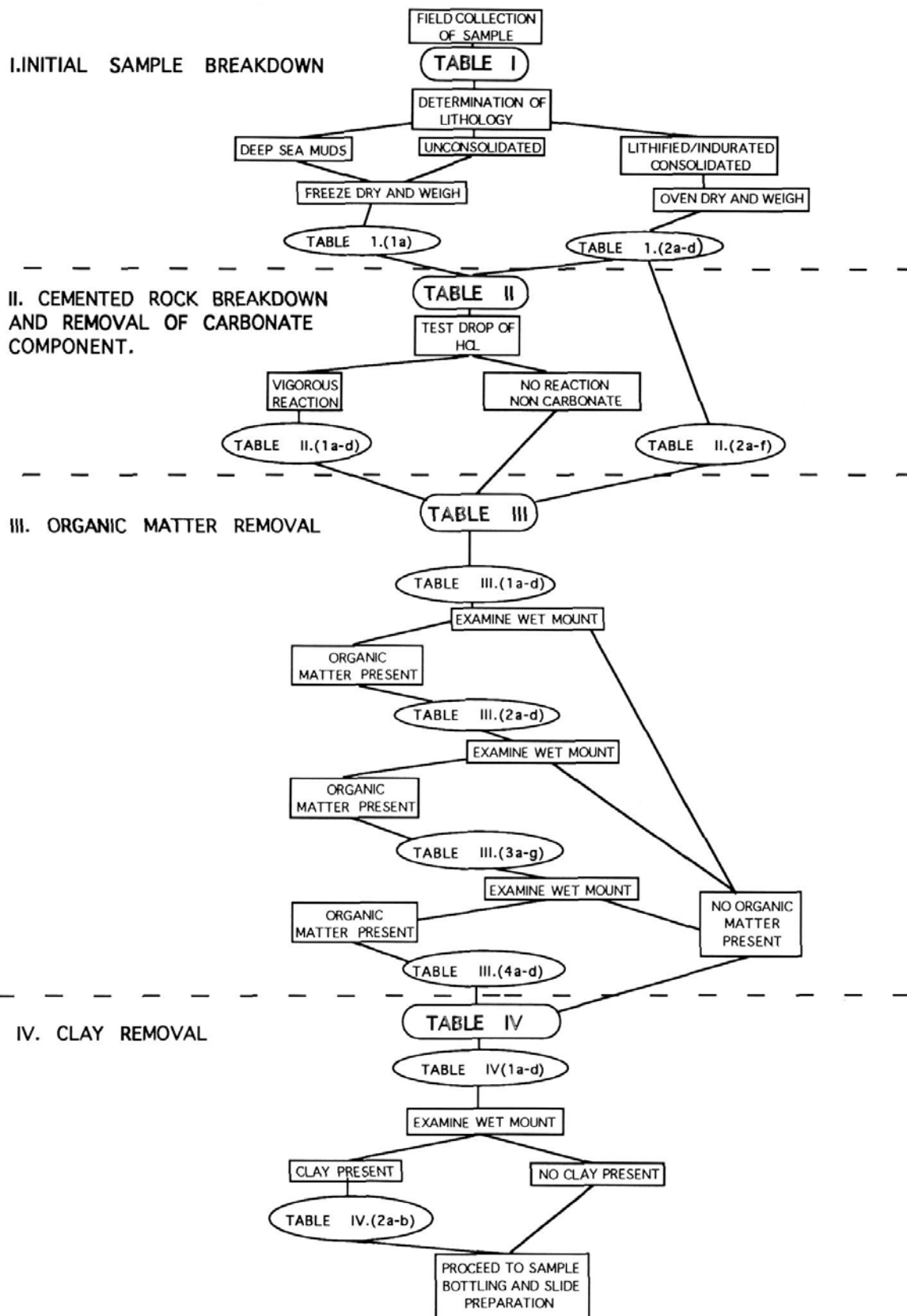
In some samples, all clay cannot be removed. As a consequence the worker must assess the residue (using wet mounts) to evaluate what is an acceptable amount of clay in their particular study.

Note on the use of wet mounts.

Tables 3 and 4 advocate the use of wet mounts at various stages of the cleaning procedure to check whether further processing is required. It must be noted that the removal of material for wet mount preparation will bias any quantitative examination (P. Bodén, pers. comm.). Therefore, if high resolution quantitative studies are to be attempted it is suggested that either 1) wet mounts should not be used, or that 2) two splits of the same sample be processed. One split being used to assess whether further processing is necessary and the other used for quantitative work. The wet mount procedure forms an essential stage in the cleaning of diatomaceous lithified sediments from Ecuador. However, it has proved unnecessary for DSDP deep sea muds from the equatorial Pacific. Therefore the use of wet mounts is wholly dependant on the nature of sediment and the purpose of study.

SLIDE PREPARATION

The method of slide preparation utilized will depend on the type of study proposed. The pipetting method of Schrader and Gersonde (1978) proves suitable for biostratigraphic studies, while the "ran-



TEXT-FIGURE 1
Flow chart illustrating the major stages in obtaining a clean diatom residue. For details see text and tables.

Table 1

The disaggregation of sedimentary rocks to component parts prior to stages of chemical treatment.

GENERAL PROCEDURE

PRIOR TO TREATMENT LITHIFIED SAMPLES SHOULD BE THOROUGHLY OVEN DRIED AND WEIGHED. DEEP SEA MUDS AND UNCONSOLIDATED SAMPLES SHOULD BE FREEZE DRIED AND WEIGHED. FREEZE DRYING IS ESSENTIAL IN AVOIDING AGGREGATE FORMATION (P. BODÉN, PERS. COMM) AND MAY ALSO AID IN DIATOM FRUSTULE CLEANING (ABELMANN 1988).

SAMPLE	PRE-TREATMENT	MECHANICAL BREAKDOWN
1) UNCONSOLIDATED OR PARTIALLY CONSOLIDATED SEDIMENTS	1a) SOAK IN DISTILLED WATER UNTIL SAMPLE IS COMPLETELY DISAGGREGATED	
2) LITHIFIED AND CONSOLIDATED SEDIMENTS		2a) FREEZE THAW: ALTERNATE SOAKING OF FOILED WRAPPED SPECIMENS IN LIQUID NITROGEN AND WARM WATER
		2b) SOAK IN PETROLEUM SPIRIT, DECANT, ADD BOILING WATER (ABELMANN 1988, BODÉN 1991)
		2c) ULTRASONIC TANK: PROLONGED EXPOSURE OF SAMPLE TO THIS TREATMENT MAY WELL RESULT IN PHYSICAL DAMAGE TO FRUSTULES AS NOTED IN OTHER MICROFOSSIL GROUPS (HODGKINSON 1991)
		2d) PERCUSSION DISAGGREGATION:
	ACTION INCREASING IN SEVERITY	i) PESTLE AND MORTAR
		ii) HAMMER AND MALLET
		iii) PNEUMATIC AND ELECTRICAL ROTARY AND RECIPROCAL TOOLS
		iv) ROCK SPLITTER, JAW CRUSHER AND MILLS
		SAMPLE AGGREGATES 10MM IN DIAMETER

AVOID EXCESSIVE USE OF ANY MECHANICAL STAGE EMPLOYED. CRUSHING TECHNIQUES ON WET SEDIMENTS (2d i) WILL CAUSE LESS DAMAGE TO MICROFOSSILS THAN THOSE ON DRY SEDIMENTS (2d ii-iv). GRINDING ACTIONS WILL DESTROY MICROFOSSILS. ONCE THE SEDIMENT HAS BEEN PHYSICALLY DISAGGREGATED (AGGREGATE SIZE GRADE 10MM), PROCEED TO TABLES II, III OR IV, DEPENDING ON LITHOLOGICAL COMPOSITION AND CONSOLIDATION.

Table 2

The removal of carbonate components and silicified rock breakdown.

CARBONATE REMOVAL

CHEMICAL PROCESS	THERMO-MECHANICAL PROCESS	MECHANICAL PROCESS
1a) SAMPLE + 20ML 15% HYDROCHLORIC ACID (HCl)	1b) WARM GENTLY AT 60°C FOR 2 HRS UNTIL EITHER SOLUTION IS LEMON YELLOW OR REACTION CEASES.	
	1c) ADD DISTILLED WATER, ALLOW TO SETTLE.	
	1d) DECANT, REPEAT UNTIL NEUTRAL	

REMOVAL OF DOLOMITE AND SILICIFIED ROCK BREAKDOWN (LAGLE 1984)

2a) SAMPLE + 30ML 0.5% HYDROCHLORIC ACID (HCl)	2b) HEAT SOLUTION AT 40°C FOR 4 HRS OR UNTIL REACTION CEASES. ADD DISTILLED WATER, DECANT	2c) CENTRIFUGE AT 1200 R.P.M FOR 2 MINS RESUSPEND SAMPLE AND REPEAT UNTIL SUPERNATANT LIQUID IS CLEAR. IF SUPERNATANT LIQUID WILL NOT CLEAR RETURN SAMPLE TO BEAKER CONTAINING FRESH 0.5% HCl SOLUTION AND REPEAT PRE-ACID DIGESTION PROCEDURE 2b
		2d) ONCE SOLUTION CLEARS ADD DISTILLED WATER AND DECANT. REPEAT UNTIL SOLUTION IS NEUTRAL. PROCEED TO COMPLETE ACID DIGESTION STAGE 2e
2e) SAMPLE + 40 ML 15% HCl. LEAVE TO STAND FOR 5 HRS AT ROOM TEMPERATURE. ADD DISTILLED WATER. ALLOW TO SETTLE THEN DECANT. REPEAT DECANTATIONS UNTIL NEUTRAL		
2f) REPEAT STEPS 2a-e IF INSUFFICIENT RESIDUE IS PRODUCED. PROCEED TO ORGANIC OXIDATION STAGE (SEE TABLE III)		

Table 3

The removal of organic matter from sediments.

CHEMICAL PROCESS		THERMO-MECHANICAL PROCESS	
1a)	SAMPLE + 20ML 30% HYDROGEN PEROXIDE (H ₂ O ₂) SOLUTION	1b)	HEAT GENTLY FOR 2 HRS AT 90°C
		1c)	ADD DISTILLED WATER - ALLOW TO SETTLE FOR 8 HRS
		1d)	DECANT. REPEAT 1c UNTIL NEUTRAL
2a)	SAMPLE + 20ML 25% NITRIC ACID SOLUTION (HNO ₃), (SETTY 1966)	2b)	HEAT GENTLY 1 HR
		2c)	ADD DISTILLED WATER. ALLOW TO SETTLE (8 HRS)
		2d)	DECANT. REPEAT UNTIL NEUTRAL
FOR LARGER AMOUNTS OF ORGANICS			
3a)	SAMPLE + SOLUTION OF 10ML SATURATED POTASSIUM PERMANGANATE (KMNO ₄) + 0.5 ML DILUTE (10%) SULPHURIC ACID (H ₂ SO ₄)	3b)	BOIL GENTLY 1 HR AT 120°C
		3c)	ADD DISTILLED WATER, ALLOW TO SETTLE 8 HRS
		3d)	DECANT. REPEAT UNTIL NEUTRAL
THEN TAKE NEUTRAL SOLUTION FROM (3d)			
3e)	ADD 1-2 ML H ₂ SO ₃ OR SMALL AMOUNT OF POWDERED DI-SODIUM DISULFITE (Na ₂ S ₂ O ₅) TO REMOVE BLACK RESIDUE OF MANGANESE DIOXIDE (MNO ₂)	3g)	IF EXAMINATION OF WET MOUNT REVEALS CLUMPS OF ORGANIC MATERIAL REMAINING
3f)	ADD DISTILLED WATER, ALLOW TO SETTLE. DECANT. REPEAT UNTIL NEUTRAL		PLACE BEAKER CONTAINING SAMPLE IN ULTRASONIC TANK FOR 3 SECS. THIS WILL DISAGGREGATE CLUMPS
FOR STUBBORN ORGANICS			
4a)	SAMPLE + 20ML CONC (30%) SULPHURIC ACID (H ₂ SO ₄) (LOHMANN 1972)	4b)	BOIL SOLUTION AT 150°C UNTIL WHITE FUMES OF ANHYDROUS SULPHURIC ACID LIBERATED
4c)	ADD SMALL CRYSTALS OF SODIUM NITRITE (NaNO ₂) TO OXIDIZE ORGANIC MATTER		
4d)	ADD DISTILLED WATER, ALLOW TO SETTLE. DECANT. REPEAT UNTIL NEUTRAL		

Table 4

The disaggregation of colloidal clay aggregates.

CLAY REMOVAL		
CHEMICAL	THERMO-MECHANICAL	MECHANICAL
1a) SAMPLE + ALKALINE SOLUTION (SODIUM HEXAMETAPHOSPHATE (NaPO ₃) ₆ + SODIUM CARBONATE (Na ₂ CO ₃) 38G/100ML (G. ANDREWS 1992, PERS COMM)	1b) HEAT GENTLY (80°C) FOR 2 HRS. ALLOW TO COOL	1c) CENTRIFUGE 1000 R.P.M FOR 3 MINS
		1d) DECANT. REPEAT 5 TIMES OR UNTIL SOLUTION CLEAR. ENSURE ALL SAMPLE RE-SUSPENDED EACH TIME BY GENTLE STIRRING WITH PIPETTE
FOR MORE STUBBORN COLLOIDAL CLAY (LOHMANN 1972)		
2a) SAMPLE + 20ML DILUTE (1%) AMMONIA SOLUTION (NH ₃) STAND FOR 4 HRS. DECANT. REPEAT 6-8 TIMES		
2b) ADD DISTILLED WATER, ALLOW TO SETTLE (4 HRS). DECANT. REPEAT 2b UNTIL NEUTRAL		

dom settling method of Moore (1973), and Bodén (1991) is preferred for quantitative work. The reader is referred to these papers for further information. The toluene-based high resolution diatom mountant "Naphrax" proved a satisfactory mounting medium.

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