OBTAINING RICH NANNOFOSSIL ASSEMBLAGES FROM 'BARREN' SAMPLES: PROCESSING ORGANIC- RICH ROCKS IN NANNOFOSSIL INVESTIGATIONS

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Abstract: Campanian-Maastrichtian carbonate and marl successions in Israel are rich in organic matter (OM) of marine algal origin. Samples from these sections, which were originally processed for nannofossil study by standard techniques, were found to be almost completely devoid of nannofossils. However, removal of the OM by approximately 10 hours of controlled bleaching with sodium hypochlorite (household bleach) yielded extremely rich and diverse nannofossil assemblages. It is suggested that, by selectively bleaching the OM, calcareous nannofossils which were incorporated within the intricate organic debris were released. The fact that, after bleaching, both abundance and species diversity were clearly increased, and the preservation of the nannofossils had not changed significantly, suggests that the bleaching agent did not 'attack' the nannofloras and did not produce a biased assemblage. The proposed preparation method enables rich and diverse calcareous nannofossil assemblages to be recovered from samples which would have provided poor, or even barren, assemblages with the deployment of standard processing techniques.

Introduction

One of the commonly-employed procedures for preparing slides for calcareous nannofossil study involves crushing the rock into a powder, suspending the powder in distilled water, allowing the heavier fraction to sink, and preparing a slide from the remaining suspension (Moshkovitz & Erlich, 1976). For the study of Campanian-Maastrichtian nannofossils in organic-rich carbonates of ocean-phytoplanktonic origin (Bein *et al.*, 1990; Eshet *et al.*, 1994), this processing technique proved to be inadequate: since most calcareous nannofossils were encased within the dominant OM debris, few nannofossils were seen under the light-microscope, even after concentrating them, using the suspension method described above. Some of the samples were even considered to be barren. Herein, a new preparation method is described, which enables rich and diverse calcareous nannofossil slides to be obtained from organic-rich material.

Materials

Fifty-three organic-rich, Campanian-Maastrichtian core-samples from the M-8 core-hole (southern Israel) were processed and analysed in this study. The section belongs to the Campanian-Maastrichtian 'En Zetim Formation. It comprises bituminous marls and chalks, with phosphatic horizons in the lower part (Figure 1). Organic content is high, reaching upto 25% TOM, with an average of 10% TOM (Figure 1, Table 1). In order to examine and demonstrate the effect of the proposed preparation method on the recovered nannofossil assemblages, ten samples were analysed before and after processing (Table 1). The same samples were used previously in an experiment which tested the utility of bleaching in palynological sample-treatment (Eshet & Hoek, in prep.). Samples and slides are stored at the Geological Survey of Israel, Jerusalem.

SAMPLE		ABUNDANCE		SPECIES DIVERSITY		PRESERVATION	
NUMBER	TOM (%)	Before	After	Before	After	Before	After
1	16.5	10	39	18	25	65	71
13	17.0	9	35	11	26	59	54
15	11.0	13	41	12	30	76	75
25	18.8	6	37	8	22	58	64
33	11.5	12	33	9	20	61	60
34	8.5	15	48	12	25	71	70
37	6.7	13	50	10	16	45	48
38	3.8	16	51	11	18	51	49
46	2.0	10	49	13	23	60	62
47	2.0	15	54	14	20	49	53

Table 1: Results of ten hours of bleaching of selected samples. Abundance = number of nannofossils per visual field. Preservation = percentage of complete specimens in the assemblage. The increase in abundance and species diversity, and the small but inconsistent change in preservation, indicate the reliability of the bleaching technique.

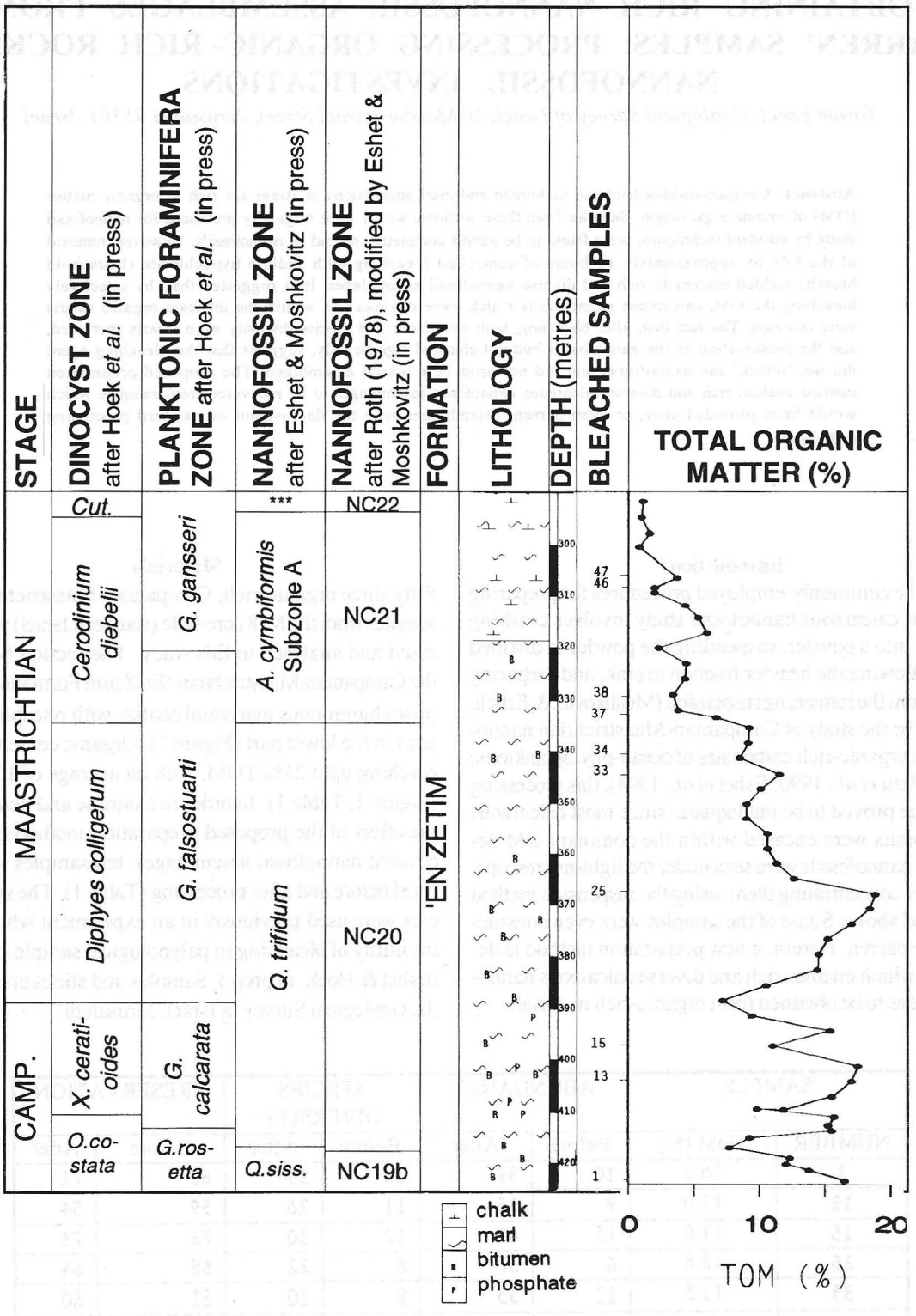

Figure 1: The M-8 sequence: litho- and biostratigraphy, and Total Organic Matter.

Sample processing

All organic-rich samples were treated according to the following procedure:

(1) 2g of rock were crushed to a powder, added to 50ml of distilled water, and stirred thoroughly.

(2) This supension was then subjected to ultrasonic treatment for 1 minute. The suspension was again stirred thoroughly, and allowed to stand for 1 minute so that some settling could take place. Moshkovitz & Ehrlich (1976) suggested that, after 1 minute, most of the settled fraction does not contain nannofossils, thus the suspended fraction was decanted into another beaker and the settled fraction was discarded. This suspension was allowed to stand for 10 minutes. Observation has shown that, after this period, most of the nannofossils have settled out of suspension, and the remaining supernatent liquid contains only very fine, mainly non-coccolithic particles (Eshet *et al.*, 1992).

(3) Bleaching: The amorphous nature of the organic residue, and the high organic-content of the samples, made

it impossible to observe nannofossils which were incorporated into the thick OM debris. Therefore, it was necessary to remove as much of the OM as possible, without affecting the nannofossils. For palaeontological preparations, oxidation of OM is usually performed using either 'Schulz Solution' (a solution of potassium chlorate ($KClO_3$) in nitric acid (HNO_3)) or hydrogen peroxide (H_2O_2). These methods have been described by Brown (1960), Faegri & Iversen (1964) and Doher (1980), among others. In the present study, the use of Schulz Solution produced a harsh chemical reaction which required the use of a fume-cupboard and other safety measures. In addition, in some samples, the nannofossils were either etched or destroyed by the reaction. H_2O_2, on the other hand, was completely ineffective, having no apparent effect on the OM, even after a 12-hour oxidation period. This was probably due to the high organic-content of the samples.

Thus, the suspension obtained in step (2) was mixed with 250ml of a 10% solution of sodium hypochlorite (NaClO). In this case, a commercial brand of household bleach called 'Economica' was used to oxidise the excess OM. Economica was used by Almogi-Labin *et al.* (1993) to remove excess OM in a foraminiferal study of similar Campanian-Maastrichtian sections in Israel. NaClO creates a basic chemical environment (A. Bein, pers. comm., 1995) and therefore does not dissolve calcite. It is thus safe to use for nannofossil preparation, and does not require a fume-cupboard or special safety measures.

For most samples, a period of 10 hours was sufficient to remove most of the OM, and to obtain a rich and diverse assemblage, with no apparent damage to the nannofossils (Table 1, Figure 2). For samples with a particularly high OM-content, a slightly longer oxidation period was required. In order to examine the effect of OM digestion by NaClO, and the process of liberation of nannofossils from the OM, a controlled experiment was conducted on ten samples of different OM-content (Table 1): 5g of each sample were soaked in NaClO for 10 hours. A comparison was then made between the pre- and post-oxidation assemblage, including the abundance (number of observed nannofossils per visual field, NVF), species diversity, and preservation (percentage of complete nannofossils in the assemblage). The results suggest the following: (a) ten hours are enough to oxidise most of the OM, and to expose calcareous nannofossils for microscopic study; (b) nannofossil assemblage 'richness' was increased dramatically: the average abundance increased from 11.9 to 43.7 NVF, and the observed average species diversity increased from 11.8 to 22.5 species per sample; (c) there was no significant change in preservation, thus the bleaching process did not destroy the nannofossils nor considerably alter the assemblage composition.

(4) Because NaClO tends to crystallise on the slide during slide-preparation, it must be removed: after bleaching, the suspension was transferred to a centrifuge-tube, and centrifuged at 2000rpm for 5 minutes. The supernatent liquid was decanted, distilled water added, and the suspension centrifuged a second time. The supernatent liquid was again decanted.

(5) The white, OM-free sediment was diluted with distilled water until the suspension became pale milky-white. One drop was mounted on a slide with Canada Balsam.

Calcareous nannofossil assemblages

The procedure utilised in the present study led to the recovery of well-preserved, rich and diverse assemblages of calcareous nannofossils from most of the samples (Plate 1). The most common fossils were: *Watznauerĩa barnesae*, *Micula decussata*, *Prediscosphaera* spp., *Thoracosphaera* spp., *Eiffellithus* spp., *Ahmuellerella* spp., *Cribrosphaerella ehrenbergii*, *Glaukolithus* spp., *Quadrum sissinghii*, *Q. trifidum*, *Tranolithus* spp., *Lithraphidites* spp., *Vagalapilla* spp., and *Vekshinella* spp.. A complete taxonomic list and a distribution chart for the M-8 sequence are given in Eshet & Moshkovitz (in press).

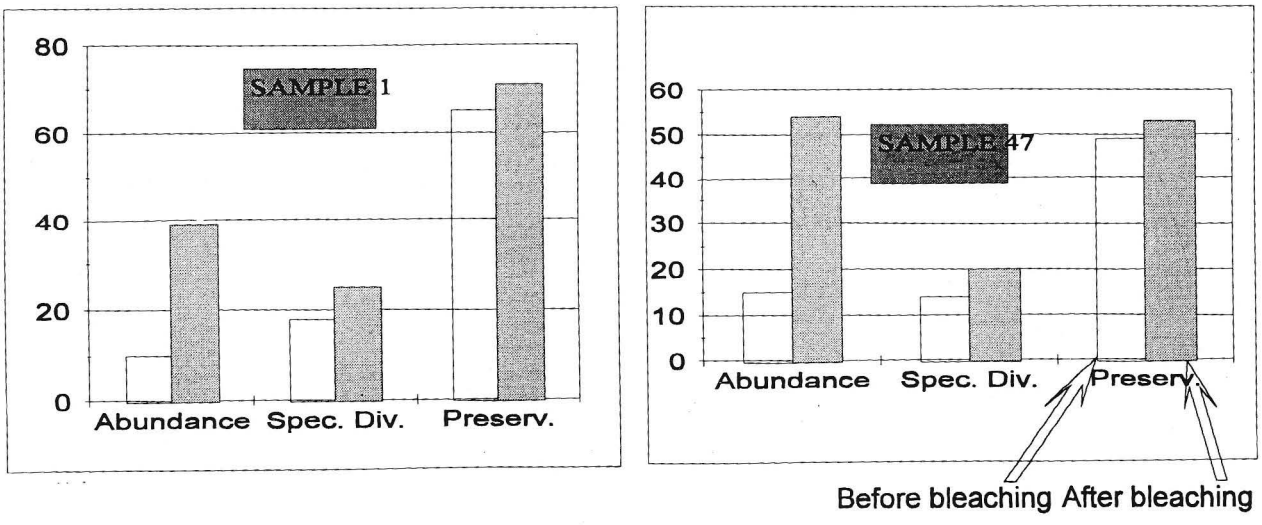

Figure 2: Bar graphs showing changes in abundance, species diversity and preservation after ten hours of bleaching. Note the drastic increase in abundance and species diversity, with no apparent decrease in preservation, suggesting that this method does not damage the nannofloras.

Summary

Bleaching the amorphous organic debris in organic-rich carbonates using NaClO has been found useful in obtaining nannofossil-rich slides. Other preparation techniques were found to be inadequate for processing this type of material. The proposed method is simple, rapid and inexpensive. A bleaching period of ten hours was found to be most effective, although a shorter period will probably be required for samples with a lower OM-content. Bleaching of the OM led to a considerable rise in observed abundance and species diversity, without affecting the preservation of the nannofossils.

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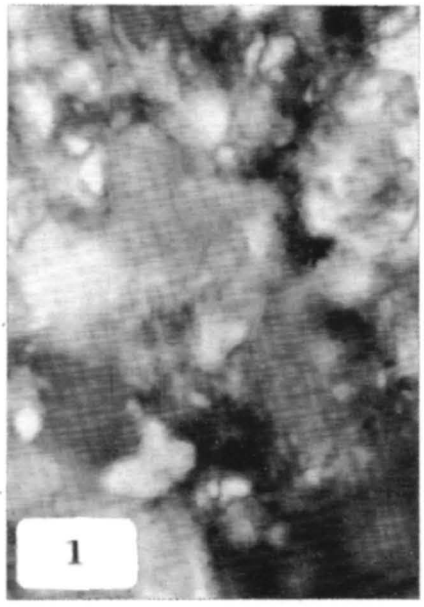
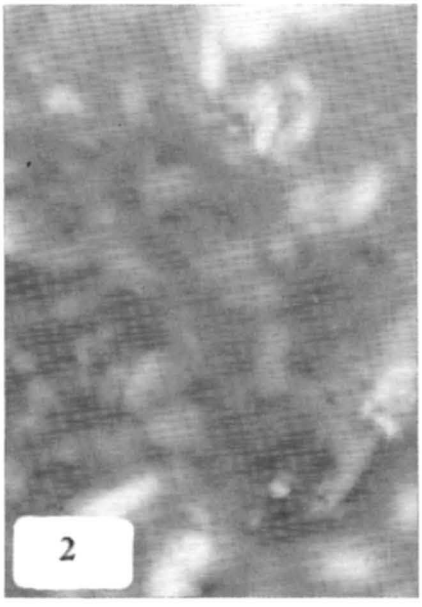
PLATE 1

Nannofossil assemblages before and after bleaching
Example from Sample #47
All magnifications x3750





1, 2: Microscope view before bleaching. Note outline of nannofossils obscured by the thick covering of organic matter.

3, 4: Microscope view after 10 hours of bleaching, which has removed most of the organic matter. Note rich and diverse nannofossil assemblages which were revealed.