Diatom Analysis of Cuneiform Tablets Housed in the British Museum

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Abstract   Clay and silt powders taken from Mesopotamian cuneiform tablets and their surface were examined for biological indicators (specifically, diatoms). The tablets are dated to the Third Dynasty of Ur and belong to the category of economic and administrative texts. Three diatom species, *Nitzschia palea, Stephanodiscus minutulus* and *Discostella* sp., were identified, though they were found in quite low densities. These species are very common in polluted/eutrophic water. Further studies are needed, however, to exclude the possibility that these diatoms are a result of later contamination, for instance via the subsequent burial of the tablets.

Key words : conservation, cuneiform tablets, diatom, Mesopotamia.

Introduction

Diatoms are important proxies for palaeoenvironmental reconstruction in archaeological science (Battarbee, 1988; Juggins and Cameron, 1999). For the last few years, we have been studying diatoms in cuneiform tablets with the aim of reconstructing the palaeoenvironment of ancient southern Mesopotamia (Tuji *et al.*, 2011).

In an earlier investigation in 2008 at the British Museum, we examined cuneiform tablets using a metallurgical microscope. On the surface of 16 cuneiform tablets, many diatom-like objects, which seemed to be *Nitzschia* species and centric diatoms, were seen. The tablets were dated to the Third Dynasty of Ur (Ur III Dynasty: 2112–2004 BC), and originated from the sites of Umma and Girsu in southern Mesopotamia (Tuji *et al.*, 2011). There were difficulties in identify-

ing diatom species with the metallurgical microscope because of low resolution and use of reflective light. We realised that we needed to use a biological (transparent) light microscope (LM) and/or scanning electron microscope (SEM) to identify diatoms; sampling from the cuneiform tablets for diatom analysis was essential.

In 2012, we conducted a scientific analysis of cuneiform tablets by inspecting tablet surfaces with a metallurgical microscope and sampling material from cuneiform tablets at the British Museum under the supervision and permission from Museum staff. In this paper, we report the results of this scientific analysis and discuss the ramifications of these results.

Materials and Methods

Direct examination of diatoms and sampling from the cuneiform tablets housed in the British Museum (BM).

At the British Museum on 12–14 March 2012, we started our analysis by inspecting cuneiform tablet surfaces using a metallurgical microscope (BXFM-S, Olympus, Tokyo) with a long working distance objective lens (LMPLFLN, Olympus, Tokyo) to search for diatoms. We examined economic tablets, which were all written during the Ur III period (2112–2004 BC), originating from the sites of Umma and Girsu in southern Mesopotamia. In particular, we looked closely at any cracked surfaces, which might expose intact internal clay and silt. This examination was a follow-up study to clarify the outcome of the earlier investigation in August 2008, when diatom-like remains were found on the surface of 16 Umma tablets. Our investigation started with the re-examination of those 16 tablets and was followed by observations of a further 83 tablets.

In summary, we looked at a total of 99 tablets under the microscope, and diatoms were observed on 33 of those tablets (Figs. 1–3). We sought permission to take samples from eight tablets from those examined in 2008, six new Girsu tablets and 18 new Umma tablets. In the course of seeking permission, we learned that many 'fired' tablets were among those on which diatoms had been observed, except BM 93202, which was apparently not fired for conservation. Permission was eventually granted for seven out of eight 2008 tablets, four out of six Girsu tablets, and 12 out of 18 Umma tablets. Sampling was carried out on 23 tablets in total under the supervision of Museum staff on 16 March.

The samples from the tablets were prepared and analysed at the National Museum of Nature and Science in Tsukuba, Japan, using the methods described below.

a. Direct observation using SEM

Clay and silt powders from six tablets were spread on carbon-coated double-sided adhesive tape (Okenshoji Co., Ltd., Tokyo) on SEM stub
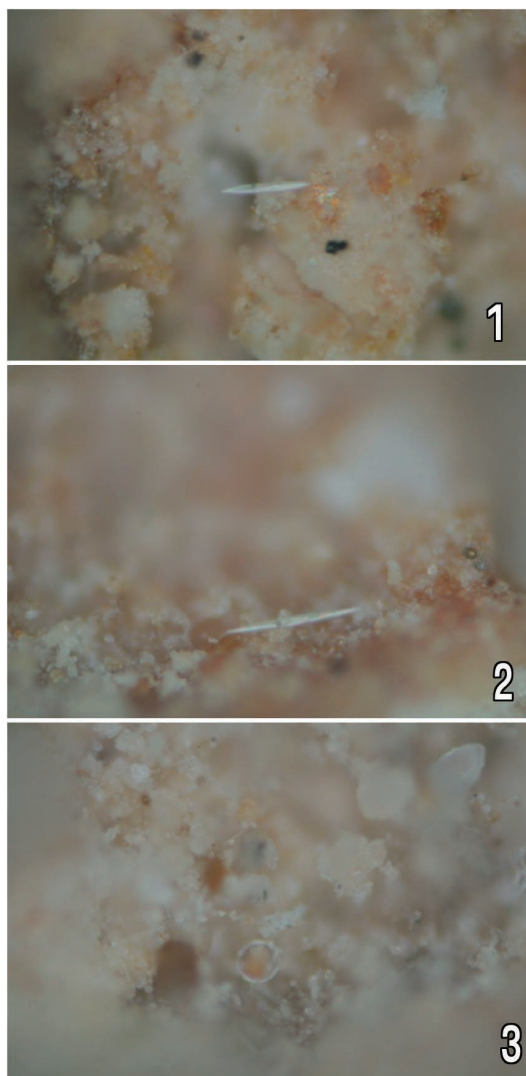
Figs. 1–3. Diatoms observed on the surface of cuneiform tablets under a microscope (Courtesy of the Trustees of the British Museum).

mounts. The stub mounts were sputter-coated with platinum and examined using a SEM (JSM-6390 with LaB6 gun, JEOL, Tokyo).

b. Boiling water treatment of diatom frustules

About 5 mg of the clay and silt powders were put into 50 ml disposable polypropylene centrifuge tubes (Watson Co., Ltd., Tokyo). About 10 ml of filtered tap water was added to each tube. The water was filtered with polypropylene filter cartridges with 5μm and 0.5μm openings

(TCW-5N-PPS and TCW-05N-PPS, Advantec Co., Ltd., Tokyo). This should have removed any diatoms present in the tap water. Tap water was used because distilled water can dissolve diatom frustules. Each tube of disaggregated suspension was heated in a microwave oven at 600 W for 5 minutes.

c. Acid treatment after boiling water treatment

After the boiling water treatment, 0.5 ml of 1.5 ml hydrochloric acid was added to each tube and the tubes were left to stand overnight.

SEM examination of the treated material

3 ml of each sample of material that had undergone boiling water and acid treatment was filtered using a membrane filter with 1.2 μm openings (RTTP02500, Merck Millipore). After filtering the treated material, the filters were washed using 2.5 ml distilled water for a short time. The filters were cut into small pieces and attached to SEM stub mounts using carbon-coated double-sided adhesive tape. The stub mounts were sputter-coated with platinum and examined using SEM.

Strong acid treatment of diatom frustules

About 5 mg of the clay and silt powders were put into 15 ml glass centrifuge tubes. About 5ml of concentrated nitric acid, including 10% concentrated hydrochloric acid, was added to each tube. These tubes were kept at room temperature for ca. two hours to remove any calcium compounds. The tubes were then heated overnight in a water bath at 70 degrees Celsius. After the acid treatment, the treated materials were washed three times with filtered tap water using a centrifuge. After the final washing, the treated materials were kept in 70% ethanol. The disaggregated samples were then mounted in ZRAX (developed by W.P. Dailey) and examined by LM (normal biological microscopy: Axiophoto, Zeiss, Jena).

Results

Since the diatom frustules in cuneiform tablets were thought to be very fragile (Tuji *et al.*, 2011),

we tried using mild treatment on samples from the cuneiform tablets as a first choice. The first treatment to be applied to the samples was the boiling water treatment, but this resulted in no diatoms being found. Because there was a possibility that the diatom frustules might have dissolved, direct observation using a SEM was applied to a sample of the clay and silt powders (no disaggregate treatment) from the tablets. However, no diatoms were found using this method either. It was then determined that calcium compounds might be obscuring or covering the diatoms. As such, the acid treatment was applied to the samples that had undergone the boiling water treatment to eliminate the calcium compounds. These were then examined using the SEM. However, only the following three diatom species were found:

Nitzschia palea (Kütz.) W.Sm.

(Figs. 5–7)

Tablet BM28109 (Fig. 4).
Length: 27 μm, breadth: 4.5 μm, striae 34/10 μm.

Stephanodiscus minutulus (Kütz.) Cleve et Möller

(Fig. 8)

Tablet BM104703.
Diameter: 4 μm.

Discostella Houk et Klee sp.

(Fig. 9)

Tablet BM104703.
Diameter: 4 μm.

The density of diatoms was up to five cells per SEM stub, which was quite low. Since many diatoms were found using a metallurgical microscope, a higher number of diatoms had been expected.

Discussion

The tablets, from which the samples were taken, were chosen based on the microscopic observation prior to sampling. However, samples were not necessarily removed from exactly the
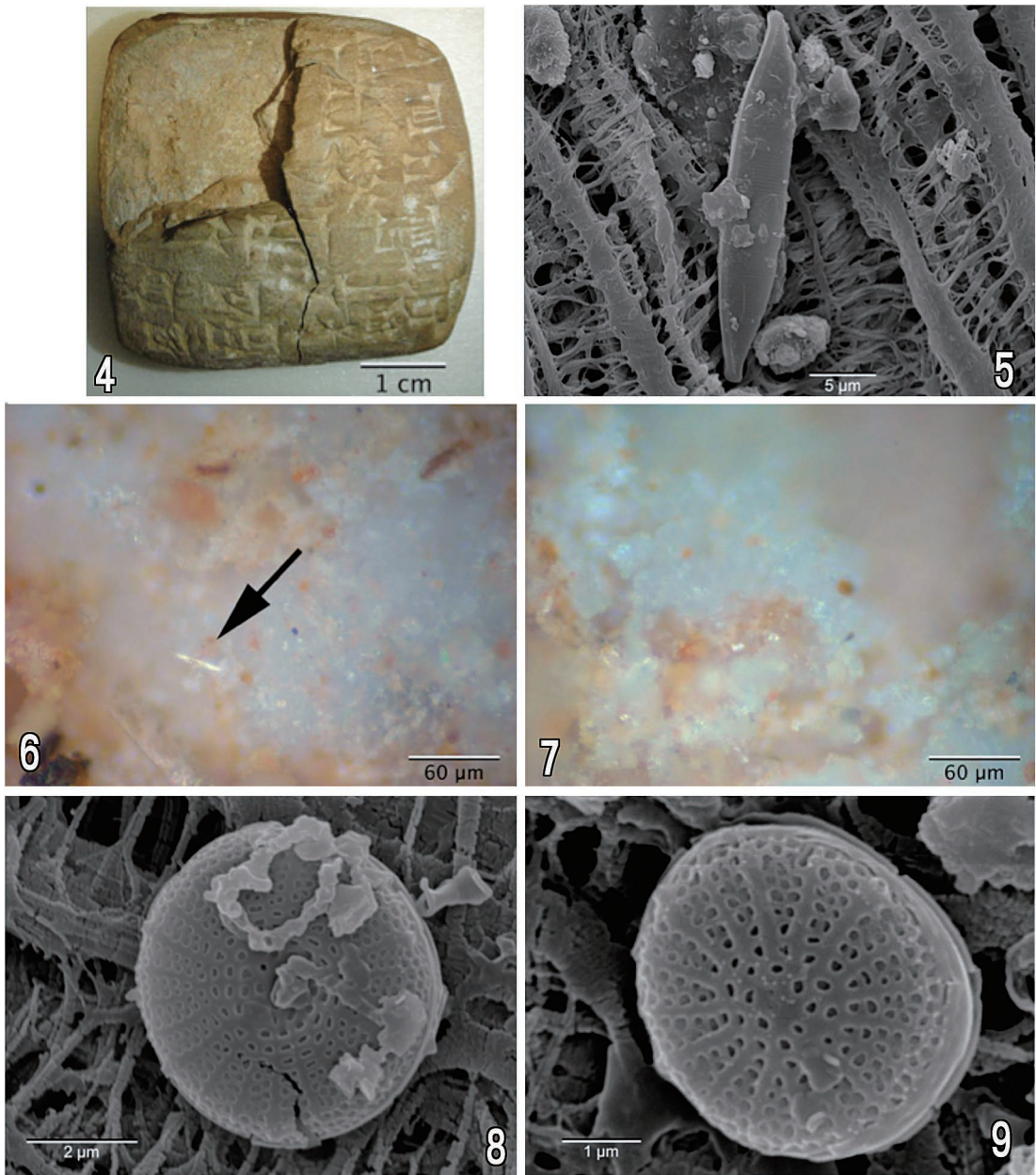
Figs. 4–9.   4. A clay cuneiform tablet BM28109 in BM (Courtesy of the Trustees of the British Museum). 5. Diatoms observed using SEM on tablet BM28109. *Nitzschia palea* (Kütz.) W.Sm. Length: 27 μm, breadth: 4.5 μm, striae 34/10 μm (Courtesy of the Trustees of the British Museum). 6, 7. Diatoms observed using LM on tablet BM28109. 6: *Nitzschia* sp., 7: Small penate diatoms? (Courtesy of the Trustees of the British Museum). 8, 9. Diatoms observed using SEM on tablet BM104703. 8: *Stephanodiscus minutulus* (Kütz.) Cleve et Möller. Diameter: 4 μm. 9: *Discostella* Houk et Klee sp. Diameter: 3.5 μm (Courtesy of the Trustees of the British Museum).

same place (i.e., a cracked surface), where the diatoms were observed, as this might have significantly damaged the tablet. Instead, an extremely sharpened tungsten needle was used to obtain samples from sections which appeared better able to withstand any damage that might be caused by sampling. We expected that tablet clay would include many diatoms throughout, having identified the presence of diatoms in one section. However, if the presence of diatoms was limited to a particular part of the tablet, it would have been necessary to pinpoint that exact section, and obtain samples from there.

Furthermore, diatoms found in this study are those commonly found in freshwater bodies. *Nitzschia palea* is an indicator species for polluted/eutrophic rivers (Watanabe *et al.*, 1990; 2005). *Stephanodiscus minutulus* and *Discostella* species are found in shallow freshwater ponds and lakes (Krammer and Lange-Bertalot, 1991, Tuji and Houki, 2001; Tuji and Williams, 2006a, 2006b). These diatoms are very common species, and it might be that contamination took place after the tablets were buried. However, it is unlikely because these tablets were buried during site abandonment and archaeosediments rarely contain high numbers of diatoms. Another possibility is that contamination occurred during conservation. Freshwater (i.e., tap water) was used in the desalinization treatment, though it is very unlikely that the water used for conservation was polluted as indicated by the species *Nitzschia palea*. Both untreated fresh water and tap water may of course contain freshwater diatoms. In order to clarify the possibility of contamination, it is necessary to investigate the history of the conservation treatments applied to these tablets. We do not know, however, whether the unsuccessful detection of diatoms was due to the firing process of tablet conservation. As diatoms disappeared in ancient firing clay pots made of disaggregated diatomaceous rocks according to Matsuoka and Suzuki (2014), the firing process might be effected the preservation of diatoms in tablets.

Another possibility is that diatoms are present in the samples, but the treatments that have been applied have not been sufficient to reveal them. We considered the possibility that the diatoms were covered with consolidated sediments such as calcium compounds, which made them invisible under the SEM. We tried to rectify this by using the acid treatment described above. Since no diatoms were found after this treatment using light microscopy, the presence of calcium compounds cannot be the reason why we cannot detect diatoms. Another possibility is that the diatoms are being covered with clay particles. However, such clay particles, even if present, would have been washed out through the 1.2 μm filter paper. We have so far applied all available methods to identify diatoms in the very small samples provided. The fact that no significant presence of diatoms was detected could indicate either that there were no diatoms in the original tablet clay or that the concentration of diatoms in the sediment was far lower than expected. In the latter case, there is a method of increasing the concentration of diatoms by using heavy liquids (Meyer *et al.*, 2012). However, this method requires larger amount of examined samples, which is not possible from these high value cultural materials.

It is, however, puzzling why so few diatoms were discovered in the clay samples we examined, especially since the presence of diatoms was confirmed under the microscope on the cracked surfaces of tablets. The lack of diatoms in these tablet clay samples, unfortunately, makes the original purpose of our research (tracing environmental changes) difficult to achieve—we had intended to examine the level of salinity of river water and link it to the precise dates as recorded in the texts on the tablets.

The fact that these samples seem to contain few or no original diatoms, however, may shed new light on some as-yet-unknown aspects of the type of source material chosen for manufacturing clay tablets. Our separate investigation of modern river sediment in the vicinity of ancient Umma revealed recently that fine sediments exposed on a riverbank do not seem to be associ-

ated with diatoms. We examined four different sediment samples taken from different sampling sites along a tributary of the Tigris (Tuji *et al.*, personal information): two were dry sediments and two were wet. The dry sediment samples contained many fine grains, but no diatoms. The wet sediment samples, however, contained diatoms, and these samples seem to have been collected from a site in which the sediment was still in contact with river water. In other studies in the Near East, it was found that phytoliths and other microfossils migrated down sandier strata to the more fine ones, such as clays (Marsh, 2014), and this may be the case with the diatoms here. This provisional study may indicate what type of source material was originally collected. It may be that alluvial sediments dominated with fine sands and silts were deliberately chosen for these economic tablets, which were more disposable and finer clays were used for the more literary texts. In order to establish this, however, we need further study and evidence.

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