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Non-destructive fabric analysis of prehistoric pottery using high-resolution X-ray microtomography: a pilot study on the late Mesolithic to Neolithic site Hamburg-Boberg

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ABSTRACT

The characterisation of prehistoric pottery fragments presents a quite complex task. In provenance studies, petrographic and chemical analyses of the ceramic materials are employed to investigate potential production areas in respect to the geological background. Moreover, also the production technology of the firing process, as well as the forming techniques used by the prehistoric potters are of great interest. Their investigation is most often accompanied by a destructive preparation of the samples. In this paper, we want to present high-resolution X-ray microtomography (μ -CT), a non-destructive and non-invasive method, as a supplementary research tool in the study of prehistoric pottery.

Ceramic fragments from the Endmesolithic–Neolithic site Hamburg-Boberg 15 (northern Germany) were analysed by X-ray microtomography. μ -CT inspection combines quantification and shape analysis of fabric components by means of computer aided image processing. As the μ -CT method is sensitive to material densities, qualitative and quantitative analyses of different temper materials are possible. Furthermore, the μ -CT method permits the characterisation of the connectivity within the porous system, as well as the analysis of the orientation of the pore structures, which are indicative for vessel forming techniques. Although limited by the resolution of the reconstructed images, distribution analysis of heavy minerals in the clay matrix can offer distinctive features to discriminate various clay sources.

Moreover, X-ray microtomography can be used to infer the nature of organic temper even with all plant remains completely burnt out during the firing process. The visualisation of the high-resolution true volume renderings and their detailed morphometric characterisation enables new avenues in the study of ceramic technology.

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1. Introduction

Pottery-making can be considered as a complex activity combining technical and social constraints. The study of archaeological ceramic materials bears a cornucopia of insights into behaviour and environment of the people involved into both production and use of pottery. To facilitate subsequent interpretations, e.g., concerning organisational aspects of pottery production such as craft specialisation, or implications of provenance studies for regional trade and interaction, a thorough reconstruction of the production technology as well as scientific investigation of the

desired use of the ceramic products (e.g., storage vs. cooking pots) is essential. In this respect, geochemical, petrographic and spectroscopic techniques have proven to contribute substantially to our understanding of the technological aspects such as raw materials, manufacturing techniques and vessel function.

Both provenance studies and investigations of production technology involve the determination of compositional characteristics in terms of chemical or petrographic attributes of the bulk ceramic or its constituent compounds. Analyses of major and trace element bulk chemistry can be obtained e.g. by X-ray fluorescence (XRF; e.g., Cultrone et al., 2011; Maggetti et al., 2011; Maritan, 2004), neutron activation analysis (see Tite, 2008, and references therein) or inductively coupled plasma spectrometry (Tite, 2008; ICP-OES and ICP-MS). More recently, also laser ablation ICP-MS has been utilised in archaeometric research (LA-ICP-MS; e.g., Speakman and Neff, 2005). Chemical characterisation using Scanning Electron

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Microscope/Energy Dispersive Spectrometry (SEM/EDS; e.g., Spataro, 2011) permits compositional area mappings on a smaller scale or point-focused analyses of temper compounds. Petrographic analyses of thin sections (Braun, 2012; Cultrone et al., 2011; Knappett et al., 2011) yield phase characterisation of temper materials and clay matrix, which can be indicative for both geological background of the raw materials and firing conditions of the ceramics. X-ray diffraction (XRD; e.g., Schmidt et al., 2012; Maggetti et al., 2011), and, more recently also used in archaeometric studies, electron backscatter diffraction (EBSD; see Peruzzo et al., 2011) are utilised to determine the crystal structure and therefore the chemistry of minerals.

Besides age and provenance of raw materials, the most distinct characteristics of ancient pottery derive from actions by the manufacturers of the artefacts. The different modes of forming a vessel leave characteristic signatures in the ware (e.g., Lindahl and Pikirayi, 2010; Berg, 2008), and also firing conditions have, in addition to control the mineralogy of the ceramics, an impact on the development of porous structures within the sherds (for a review, see Tite et al., 2001). Aside from thin section microscopy and visual examination from surface markings, further information on forming techniques by inspection of void and temper distribution and orientation can be provided using radiography. Although applied to pottery in earlier studies (Van Beek, 1969), the potential of X-radiography for ceramics was fully appreciated with the work of Rye (1977). Since the field of diagnostic radiology was revolutionised by the introduction of X-ray computed tomography (CT), X-ray CT has had broad applicability to material sciences. Also in archaeological investigations, CT was used to non-destructively infer the interior of valuable, so far intact artefacts (e.g., analysis of Roman cremation vessels by Anderson and Fell, 1995). Promoted by increased capability of computers and imaging software, and particularly by the invention of a new type of micro-focus X-ray source, both feasibility and resolution of the CT technique improved drastically. To date, X-ray sources with small (less than 10 µm) diameter focal spots operate in bench-top systems that achieve object resolutions down to the sub-µm range (X-ray microtomography, or µ-CT). The X-ray microtomography method, like medical tomography, employs X-rays to inspect the three

dimensional distribution of matter inside the object of investigation. The volume reconstruction of the material density arrangement in the samples allows detailed inspection of the fabric of the pottery sherds on micrometre scale in 2D and as well as in 3D.

The appearance of high-resolution X-ray microtomography fuelled research in many disciplines, e.g. earth sciences (quantification of rock textures and reservoir characterisation: Ketcham, 2005; Jerram et al., 2009; Remeysen and Swennen, 2008), palaeontology (dinosaurs: Sereno et al., 2007; dental tissue: Rossi et al., 2004; trace fossils: Kiel et al., 2010), metallurgical engineering (grain boundary fractures: Garcia et al., 2009). High-resolution microtomography (synchrotron radiation) has also been used in archaeometric context, e.g., to investigate the carbonized remains of a Citrus-like fruit in a funerary offering deposit dated back to the beginning of the 6th century BC (Coubray et al., 2010), and, e.g. to study the impregnation of archaeological waterlogged wood with consolidation treatments (Bugani et al., 2009).

As high-resolution µ-CT detects – and localises – attenuation differences within the sampling volume, it is sensitive to chemical composition in terms of mean proton number and electron density of the material. Thus, it does not provide single element analysis. However, if the phase composition is known from other techniques, it is straightforward to recognise the phases in the volume reconstruction. Therefore, the method is distinctive for different temper materials as well as variations in the clay matrix. Moreover, because of the high density contrast between solids and voids, the µ-CT method is suited extraordinarily well to survey occurrence and orientation of the porous structure within the sherds. Thus, the application of µ-CT on ceramic fragments can be applied to study details related to production technology, and, by quantitative analysis of fabric compounds, utilised to perform compositional investigations.

The present study deals with an archaeometric approach to ceramics from the final Mesolithic – early Neolithic site Hamburg-Boberg 15, which is situated on one of several small, occupied sand dunes located in the marshlands of the Elbe Urstromtal in the southeast of Hamburg (Fig. 1). The site is considered an important location for the documentation of the Neolithisation of the Jutland peninsula, which comprises the mainland part of Denmark and

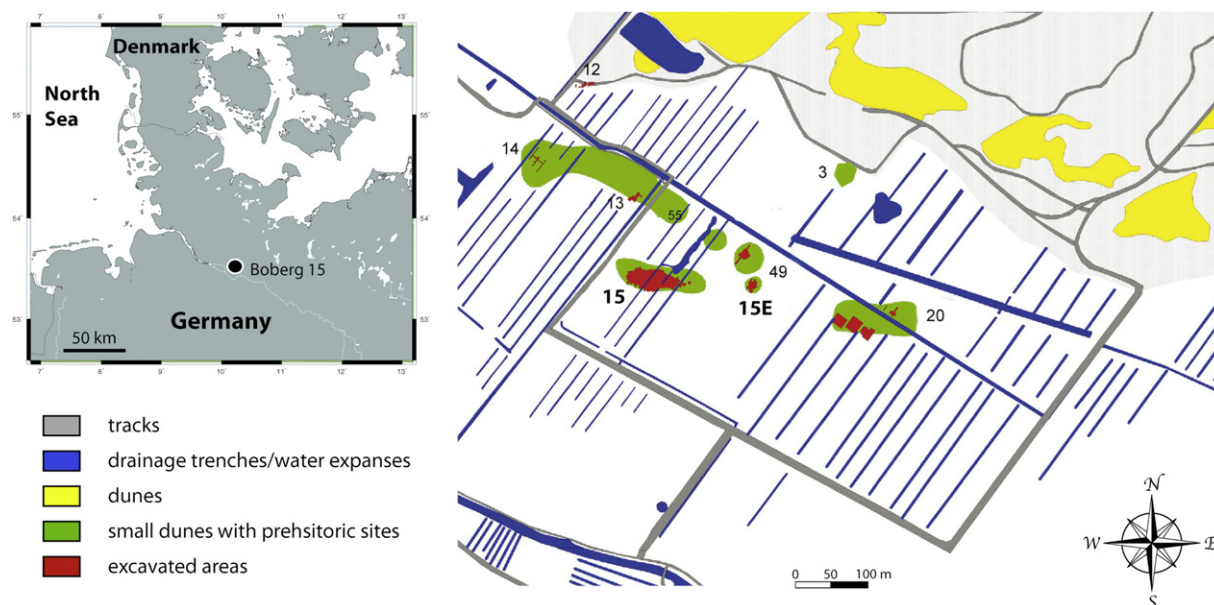


Fig. 1. The Endmesolithic–Neolithic sites of Hamburg-Boberg (53°30′27.7″N 10°08′27.2″E) are situated on small, flat sand dunes in the marshlands of the Elbe-Urstromtal, located on the Jutland peninsula, which comprises the mainland part of Denmark and parts of northern Germany. Regional map produced with GMT (Wessel and Smith, 1991).

parts of northern Germany. The Boberg area harbours pottery artefacts covering the final Mesolithic Ertebølle culture as well as the early to middle Neolithic Funnelbeaker culture. Moreover, there are numerous finds of sherds that exhibit a foreign morphology. Excavated already in the 1950s, the material is known because of its typologically foreign ceramics. During the last decades, the same typologically foreign sherds were related to different archaeological cultures, e.g. Linearbandkeramik, Stichbandkeramik, Rössen, as well as Baalberge, Gatersleben and newly Michelsberg (summarized in Klassen, 2004; Ramminger, 2012). Although Boberg is one of the most important sites for the debate on the Neolithic transition in northern Germany and southern Scandinavia, the material has never been presented completely, and the interpretations of the function of the site have differed strongly (e.g. Schindler, 1953, 1961, 1962; Schwabedissen, 1994; Hartz et al., 2000). Regarding northern Germany, it has been suggested that changes in the subsistence strategies rely on a network of connections established between the Mesolithic people of northern Germany and the Neolithic people of more southern regions (e.g. Schindler, 1961; Laux, 1986; Schwabedissen, 1994; Hartz et al., 2000). These studies have often drawn upon ceramic typology and stylistic comparison with other regional cultures, while issues of pottery technology have rarely been addressed, and the question of import or imitation of the typologically foreign ceramics at Hamburg-Boberg has never been discussed. Therefore, we think that a more sound understanding of the site can be achieved by addressing also key aspects of the manufacturing process and the desired use of the ceramics. Thus, the combination of analytical methods and typological analysis, and not an approach solely based on the latter, may strengthen the discriminative power of hypotheses concerning the complex archaeological record of the site. As this is the first step in an ongoing project to assess the potentials and limitations of a technological approach to ceramic style and exchange for the site Hamburg-Boberg, a discussion concerning the cultural or technical roots of ceramics is outside the scope of this work.

This study was mainly sparked by the prospect of evaluating the potential of an integrative assessment of a composition-related (type) as well as a manufacturing-related (preparation) description of clay matrix, solid temper compounds and the porous system of the pottery fragments. The porous system, consisting of voids created by organic temper that vanished during the firing process and of porosity gained by the forming process and the firing technique, is a key to decipher certain steps in the production chain. Especially the use of organic material bears potential to infer many ancillary conditions of pottery production. Mixing organic matter with clay material is a basic recipe for making building material and sometimes pottery. Many traditional societies use plant temper. In Middle European prehistory, it is especially known from the early phase of the Linearbandkeramik (Cladders, 2001) as well as within Epi-Rössen and Michelsberg groups, e.g. in France and Belgium (Constantin and Kuijper, 2002). Some recent studies have underlined the technological function of plant temper (Skibo et al., 1989; Sestier, 2005; Tsetlin, 2003). Sestier et al. (2005) pointed out, that, despite the fact that organic-tempered pottery is quite common in some cultures, only few studies have been carried out on the use of plant temper in Neolithic sherds (Sestier et al., 2005, p. 252). As a potential reason they mention the lack of an analytical technique appropriate to the characterisation of organic fragment “ghosts” in pottery. Plant temper of prehistoric ceramics is probably more common than previously thought, and therefore deserves more intense research. The inclusion of fibres enhances the tensile strength of the wet and dry paste, and improves water exchange through macroscopic porosity (Sestier, 2005). Earlier on some trials were made to show the structure and form of plant inclusions by filling the voids with a fluorescent

polymer (Sestier et al., 2005), aiming at the botanical identification and a quantitative evaluation. While SEM studies (e.g., Tomber et al., 2011) that focus on analysis of organic inclusions explicitly require organic remains, the μ -CT method is able to investigate the very cavities that have been left by now vanished organic material (see, e.g., Kiel et al., 2010, who investigated the nature of trace fossils in oligocene whale bones by morphometric analyses of borehole cavities made by a bone-eating worm).

In this paper, we want to present high-resolution X-ray microtomography as a supplementary research tool in the study of prehistoric pottery. We want to evaluate the potential and the limitations of the μ -CT method, concerning the characterisation of nature, preparation and abundance of temper agents. Furthermore, we want to assess the applicability of μ -CT observations on cavity morphology and orientation of the pore structure to infer the coiling technique that was applied in the forming process of the vessels.

2. Materials and method

In this pilot study, we report the application of a non-destructive and non-invasive method for textural analysis of ceramic artefacts. Five selected potsherd samples have been investigated using high-resolution X-ray microtomography to find distinctive markers for technological features which supplement the typological characteristics of the sherds.

The morphometric analysis of the ceramic microtexture involved the following steps:

- (1) qualitative analysis of the temper material (grain size and form, density and homogeneity of the non-plastic components)
- (2) quantitative analysis of the temper material (volume percent) identified according to grain size and form (roundness)
- (3) quantitative analyses of the open and closed pores, which can be seen as a product of firing-temperature in combination with manufacturing techniques.
- (4) qualitative analyses of the orientation of the porous system, which is very strongly influenced by the forming technique that was utilized.
- (5) semi-quantitative analysis of micro-components of the clay.

2.1. Archaeological samples

For the pilot study we selected five ceramic samples, one Mesolithic and four Neolithic sherds. The five fragments are not representative for the site, neither is the organic temper in the Mesolithic sample typical for this age – there also exists Mesolithic ceramic with rock temper at the site. We chose the samples because they comprise a variation of all potential temper materials occurring on the site (rock fragment temper: all four Neolithic fragments; grog: 15-93; organics: 137-27). To evaluate the accuracy of the method, the fragments 131-05, 13-137 and 138-67 were selected because of their similarity in containing solely angular rock fragment temper. In addition, sample 138-67 was chosen because of its decoration: we were interested in potential differences that might be associated with a more complex production chain including decoration. Moreover, the Mesolithic sherd 137-27 is presumably fired in an open bonfire at moderate temperatures and therefore might exhibit a less sintered clay matrix. Since sherds of this culture have already been investigated in respect to vessel forming techniques, we were particularly interested to explore the applicability of high-resolution X-ray microtomography on this task.

Sample 131-05 (Fig. 2A) is a light outwards everted rim of an undecorated Funnelbeaker vessel. The wall thickness is 11 mm. The

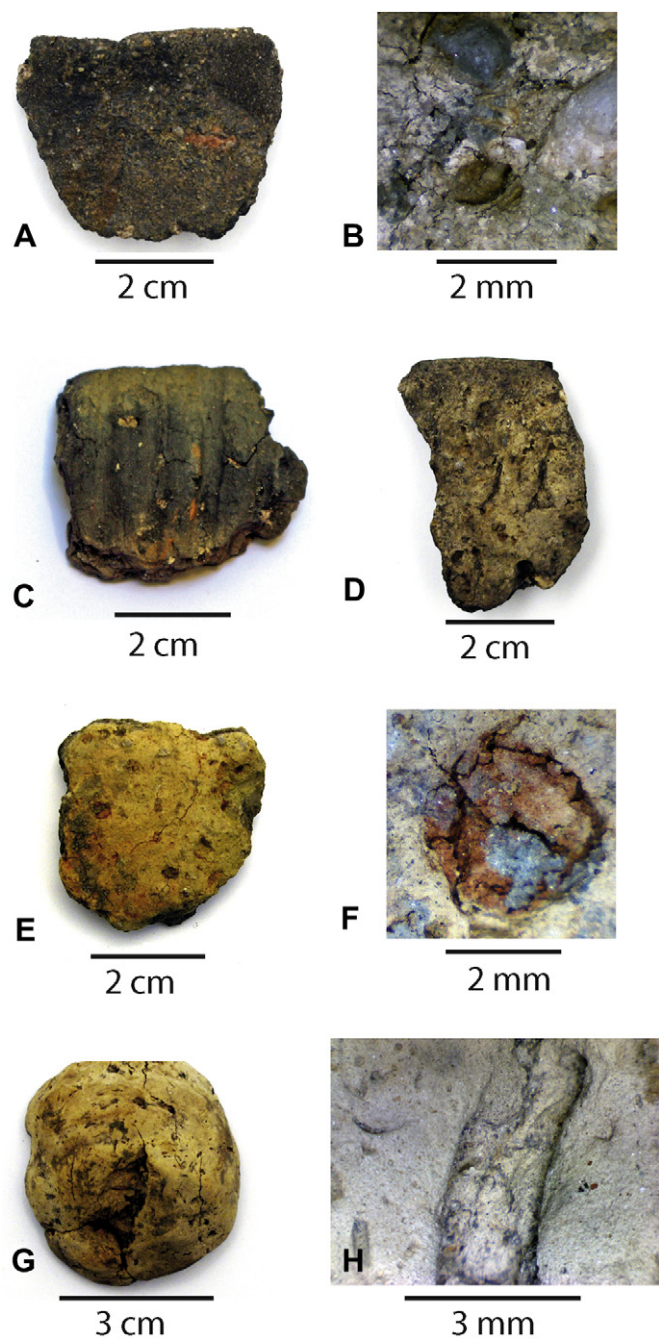


Fig. 2. Photomicrographs of the ceramic artefacts. (A) and (B) Sample 131-05: rim of a Funnelbeaker vessel (A) with inclusions of cracked granite in different sizes (B). (C) Sample 138-67: body sherd of a small Funnelbeaker with vertical incised lines. (D) Sample 13-137: rim with fingernail imprints. (E) and (F) Sample 15-93: undecorated sherd (E) with large grains of grog temper (F). (G) and (H) Sample 137-27: pointed bottom of an Ertebølle vessel (G); cracks and holes are residues of the organic temper (H).

ware colour at the inner and outer surface is dark brown. The core is middle brown. Macroscopically visible in the coarse clay are white and red grains of granite temper up to 10 mm in length. The size of most of the temper grains varies between 1 and 3 mm in diameter. The granite fragments are of angular to sub-angular shape typical for ceramics of the Funnelbeaker culture (Fig. 2B).

Sample 138-67 (Fig. 2C) is a body sherd from a small Funnelbeaker with a decoration of vertical, incised lines typical for Funnelbeaker culture. The fine clay is tempered with small granite

grains of 1–2 mm in size. The dark grey inner surface is well smoothed as is the middle grey outer surface. The wall thickness is 8 mm.

Sample 13-137 (Fig. 2D) is a rim of a vessel decorated with fingernail imprints. The wall thickness is 11 mm. The outer surface is light grey and eroded. The smoothed inner surface is better preserved and of middle grey colour. The coarse clay is tempered with sharp-edged fragments of granite up to 3–4 mm in length. Nail and finger imprints on the rim occur occasionally on Ertebølle beakers in Ringkloster, Jutland and other sites (Stilborg and Bergenstråhle, 2000) as well as on vessels of the Funnelbeaker culture, but traditionally ornamentation of Ertebølle vessels has been described as sparse.

Sample 15-93 (Fig. 2E) is one of few sherds in the Boberg inventory which deviates from the majority because of different temper materials. On the light red outer surface several rounded inclusions of orange/red colour are visible (Fig. 2F). Under the binocular microscope, the structure of the grains indicates that they may be grog temper. The inner surface of the sherd is dark grey while the core is bicoloured red/dark grey. The wall thickness is 10 mm.

Sample 137-27 is a pointed bottom, which is characteristic of the S-shaped beakers of the final Mesolithic Ertebølle culture (Fig. 2G). The wall thickness is 7 mm, which is rather thin for Ertebølle beakers. But regional variations in the shape and the building techniques are already mentioned (e.g. Stilborg, 1999; Stilborg and Bergenstråhle, 2000). The ware colour is grey beige at the inner and outer surface as well as in the core. Numerous fine holes and cracks which are traces of organic temper materials are macroscopically visible. Under the binocular microscope, the clay matrix shows a dense texture without any mineral inclusions (Fig. 2H). The clay is quite soft, thus the firing-temperature seems to have been in the lower range. That is in line with observations on Ertebølle vessels from Scandinavia, whereby they seem to have been fired in an open bonfire to a maximum temperature of 500–600 °C for not more than half an hour (Hulthén, 1977, p. 37).

2.2. X-ray microtomography method (μ -CT)

The X-ray microtomography technique, like medical tomography, employs X-rays to inspect the three dimensional distribution of matter inside the object of investigation. The term “micro” indicates that the resolution achieved is in the μ m-range. The method is based on the detection and subsequent localisation of the degree of attenuation of the incident X-rays in the sample. The attenuation of X-rays by matter depends on both the chemical elements it consists of as well as their density.

The X-ray microtomography scans of the prehistoric potsherds were done using the SkyScan1172 system (SkyScan, Belgium) at the Institut für Geowissenschaften, Christian-Albrechts-Universität Kiel, Germany. The SkyScan1172 is a tabletop unit for high-resolution scans suitable for sample sized up to 35 mm in diameter and ca. 55 mm in height. The pottery fragments were scanned with a beam energy of 100 kV, a flux of 100 μ A and a copper-aluminium filter at a detector resolution of 17.3 μ m per pixel using a 360° rotation with a step size of 0.4° in the oversized scan mode (3 connected scans). Reconstruction of the spatial information on the linear attenuation coefficient in the archaeological samples was done using the SkyScan software Nrecon running on a cluster of 3 networked PCs. The program uses a modified Feldkamp algorithm (Feldkamp et al., 1984). For each sample, a 2000 \times 1000 matrix (or larger) of up to 2250 slices per stack is available. As a result of the reconstruction procedure, each image consists of voxels (volumetric pixels) and has a certain thickness depending on the detector resolution. Thus, a stack of images contains true

volumetric information. The information about the varied X-ray absorption is encoded as grey values in the black-and-white images. Segmentation (i.e. the recognition of different fabric compounds), image analysis and volume rendering was done using the SkyScan software CTAn, CTvol and CTvox. To investigate the range of connectivity within the porous network, a two-step procedure for segmentation of the pores was applied: first, a dataset containing the total porosity was produced. Subsequently, only the closed porosity was segmented into a second dataset. Within the free image processing package Fiji, the two datasets were merged via the colour channel submenu. As a consequence of the colours chosen, after the merge the open porosity that is connected to the surface of the sherd is displayed in green (PDF-version of this article)/dark grey (printed version), whereas the closed porosity, that is not connected to the sherd surface, is coloured yellow/bright. To visualise the models in 3D, the plug-in 3D Viewer (see Schmid et al., 2010 for details) has been employed.

3. Results

The volume reconstruction of the material density arrangement in the samples allows detailed inspection of the fabric of the pottery sherds on micrometre scale in 2D and as well as in 3D. Each voxel represents the mean linear attenuation coefficient for X-rays of the corresponding volume of the sample and depends on density and atomic number of the scanned material. In the reconstructed images, areas of high attenuation (e.g. dense minerals in the clay matrix and rock fragment temper) are represented by light grey values, whereas areas of low X-ray absorption are encoded in dark grey (clay matrix) or black (voids). Segmentation of voxels into the different fabric components is obtained by grey level thresholding. The accuracy of the morphometric investigations is therefore dependent on both voxel resolution and choice of segmentation thresholds. By a comparative study of sandstone cores well characterised by standard laboratory techniques it was found that the

porosity of both sandstone types could be reproduced within one percent point (Kahl and Holzheid, 2009).

The ceramic microstructure (see Fig. 3) is dominated by two prominent features: cavities in the clay body and a high abundance of temper, consisting of rock fragments or grog of various sizes. By means of image processing, sherd porosity and orientation of the porous system, as well as cavity morphology, differences in nature, preparation and abundance of temper agents and in the character of the clay body, as well as abundances of the fabric components can be quantified by computer aided morphometric analysis.

3.1. Solid temper: rock fragments and grog

Abundance and nature of temper agents differ among the five samples (see Fig. 4A–E) and reveal technological variations in the methods applied by the potters. Rock temper is absent in the Mesolithic vessel. The two types of solid temper present in the Neolithic pottery demand different treatment in the segmentation step.

3.1.1. Rock fragment temper

All mineral compounds contained in the fragments of crushed rock exhibit significantly higher attenuation coefficients for X-rays than the clay matrix. Therefore, this type of temper can easily be segmented by the grey level for further morphometric analysis.

3.1.2. Grog

Grog, which is fired and then ground up clay, reveals attenuation coefficients that are slightly lower than those of the clay matrix. However, both materials exhibit a certain scatter in the attenuation properties. Thus, both materials are represented by voxel of overlapping grey levels in the reconstruction images. Therefore, a straightforward segmentation by the grey level is hindered. A successful segmentation of the grog component can be achieved by

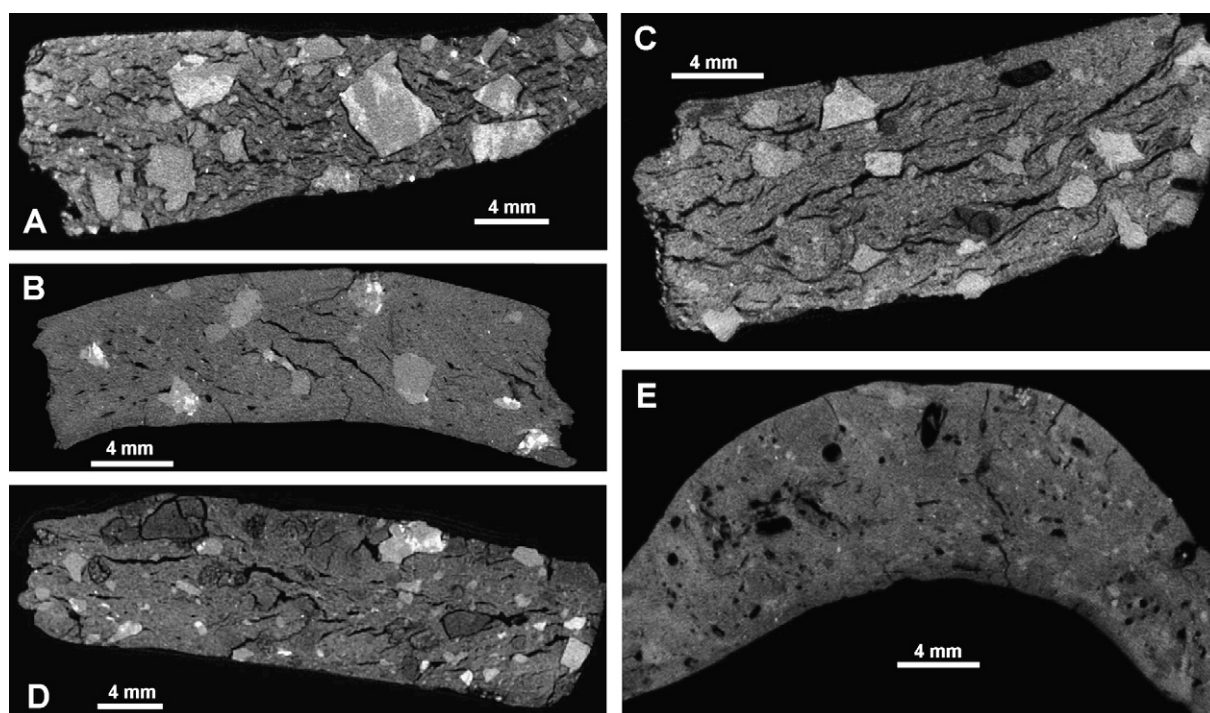


Fig. 3. Reconstructed images of Neolithic pottery fragments excavated from the northern German site Hamburg-Boberg. (A) to (E): samples 131-05, 138-67, 13-137, 15-93, 137-27. The samples show distinct differences in cavity morphology, in nature, preparation and abundance of temper agents and in the character of the clay material.

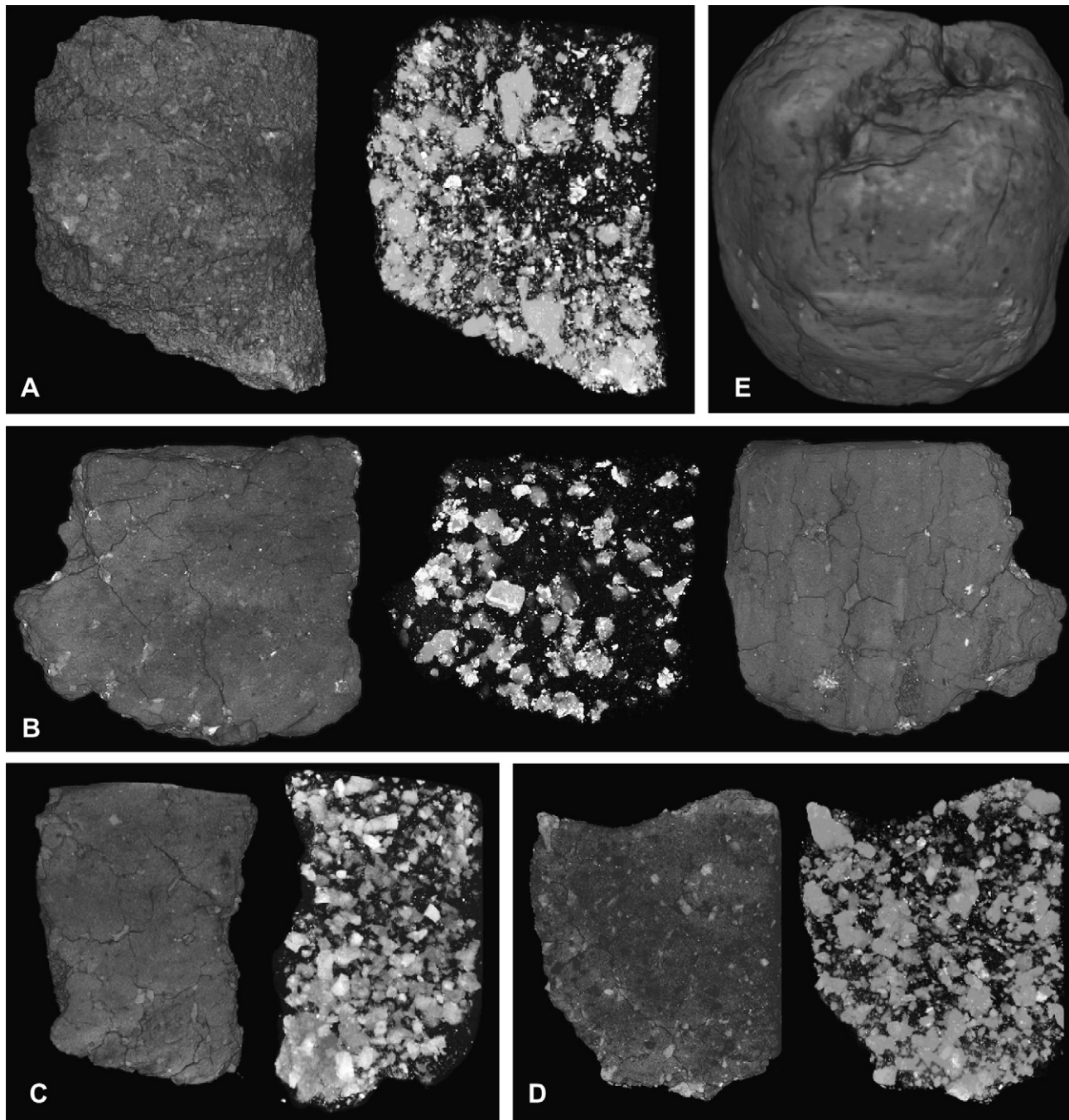


Fig. 4. Micro-CT-renderings of Neolithic pottery fragments. (A) to (E): visualisation of potsherd morphology and the spatial distribution of temper in the samples 131-05, 138-67, 13-137, 15-93 and 137-27.

iterative steps of noise reduction by nonlinear anisotropic diffusion methods (Frangakis and Hegerl, 2001). Since this procedure involves a lot of user interaction, it was applied on a small number of grog fragments.

Angular rock fragments make up the sole added temper in sherd 131-05 (content 17.4 vol. %, see Table 1) or the most abundant in 138-67 (9.8 vol. %) and 13-137 (13.0 vol. %). Moreover, the latter two contain minor amounts of organic temper (see below). Sample 15-

Table 1
Results of 3D analysis of fabric compounds in the pottery fragments.

	131-05	138-67	13-137	15-93	137-27
Total porosity (vol. %)	5.3	1.4	3.3	5.2	4.4
Closed porosity (vol. %)	1.7	0.9	1.7	1.2	1.8
Rock fragment temper (vol. %)	17.4	9.8	13.0	10.8	—
Temper agents	Rock fragments	Rock fragments, organic	Rock fragments, organic	Rock fragments, grog	Organic

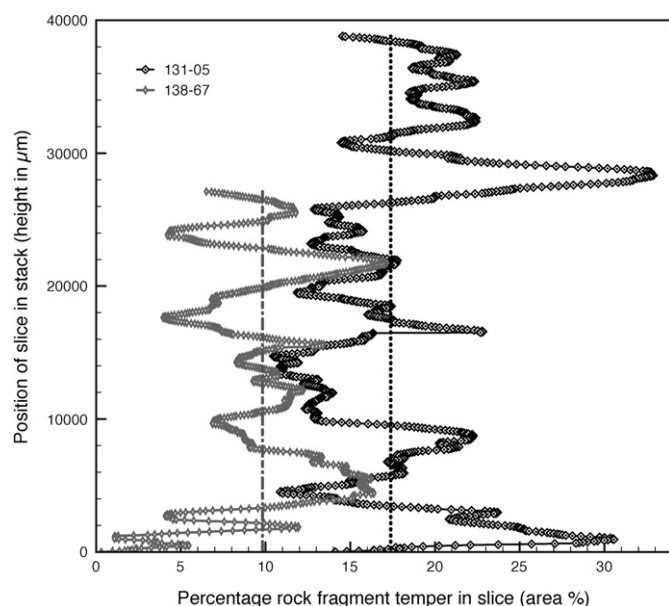


Fig. 5. Comparison of the percentage of rock fragment temper derived from 3D and 2D analysis. The results of the 3D analysis of the image volume (whole stack; values are denoted by the stippled lines) are compared with the results of the 2D analysis of each single slice (exemplified for two fragments: 131-05 (see Fig. 4A), data shown in black; and 138-67 (see Fig. 4B), data shown in grey). As the thickness of the slices (17.3 μm) is within the range of the thickness of a thin section (25 μm), the graph shows the impact of the sampling position for thin section preparation on the results of 2D temper analysis.

93 contains 10.8 vol. % rock fragment temper and sparse grog temper. The fragment of the Mesolithic Ertebølle vessel (137-27) is devoid of any solid temper fragments. Tempering was performed using only organic material (see Section 3.3 below).

A comparison of the percentage of rock fragment temper derived from 3D and 2D analysis underscores the advantage of morphometric image analysis using true volumetric datasets. In Fig. 5, this is exemplified for the two fragments 131-05 and 138-67. 3D analysis (for resulting volume percentages see previous paragraph) utilises the image volume of the whole stack, whereas 2D

analysis focuses on each single slice (enabling the determination of area percentages like in thin section microscopy). In Fig. 5, the results of the 3D analysis are compared with the results of the 2D analysis of each single slice, the location of which is given as height in μm within the sherds (see Fig. 4A (sample 131-05, and 4B, sample 138-67), for illustration: the orientation of the stack axis within the sherd is upward). The results of 2D analysis on the content of rock fragment temper in sherd 131-05 range between 10.7 area % and 32.8 area % (3D analysis: 17.4 vol. %), and in sample 138-67 between 0.3 area % and 17.2 area % (3D: 9.8 vol. %). As the thickness of the slices (17.3 μm) is within the range of the thickness of a thin section (25 μm), the graph shows the impact of the sampling position for thin section preparation on the results of 2D temper analysis.

Morphometric analysis of individual 3D objects and size class frequencies identify technological differences in the preparation of the rock temper. Frequency analysis of the rock fragment temper in the Neolithic sherds singles out the very high number of rock fragments in sherd 131-05 (Fig. 6A). The relative size class distributions of rock fragment temper in the Neolithic pottery fragments samples 131-05, 138-67, 13-137 and 15-93 reveal a narrow sorting grade in sherd 138-67 (Fig. 6B).

To elucidate potential differences in the preparation of the raw materials, shape analysis has been applied to the different temper agents. The roundness, a particle shape parameter in 2D, characterizes the smoothness of the particle shape contour and is defined as:

$$R = 4A / (\pi D_{F,max}^2)$$

where A is the projected area of the particle onto a plane, $D_{F,max}$ is the maximum Feret's diameter.

A comparison of the 2D-roundness parameter of the rock fragments exhibits no distinctive differences among the four Neolithic sherds (Fig. 7), since the rock fragments are of granitic/felsic gneiss origin and thus would break into similar geometries when crushed during the temper preparation. The grog temper of sample 15-93 shows a size-dependant pattern: large grog fragments deviate strongly from the ideal circle (which is signified by a roundness value of 1), indicating that these particles are split into several pieces. In places, cavities also exist inside grog temper grains.

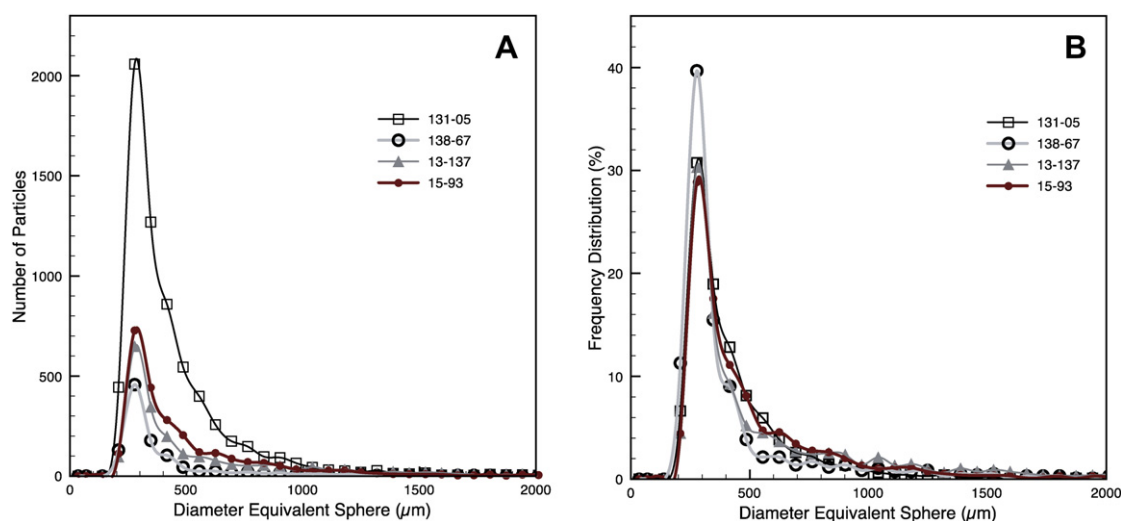


Fig. 6. Analysis of the rock fragment temper. (A) Abundance of rock fragment temper in different size classes in the Neolithic pottery fragments. Note the high number of rock fragment temper in sherd 131-05. Rock fragment temper is not present in the Mesolithic sherd. (B) Frequency distribution of rock fragment temper in the Neolithic pottery fragments samples 131-05, 138-67, 13-137 and 15-93. The narrow sorting grade in size of the fragments in sherd 138-67 is distinctive.

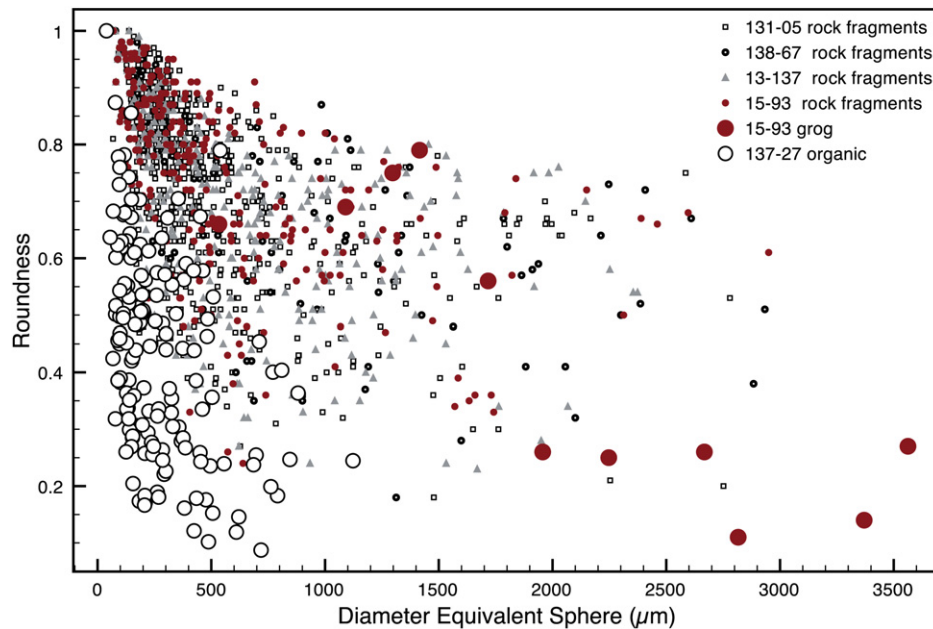


Fig. 7. Comparison of the 2D-roundness parameter of different temper agents. The rock fragment temper of the samples 131-05, 138-67, 13-137 and 15-93 exhibit no distinctive differences. The grog temper of sample 15-93 shows a size-dependant pattern: large grog fragments deviate strongly from the ideal circle (which is signified by a roundness value of 1), showing that these particles are cracked. The organic temper of sample 137-27 is characterized by low roundness values, which indicates that the majority of the material used was of rod-shaped geometry.

3.2. Porosity

By means of image processing, different aspects of the porous system within the sherds can be characterised. Porosity (the volume portion of voids in respect to the total volume of the sherd), as well as the frequency distribution of the various pore sizes can be extracted from the image volume. Moreover, the spatial arrangement of the pore structure, and the range of connectivity of the pores can be visualised (see below).

Although sherd 138-67 has voids that are caused by organic temper (see below), the porosity is remarkably low (1.4 vol. %, see Table 1). While similar in amount and nature of the temper, this sample contains less cavities around the rock fragments in comparison to the potsherds 131-05 (5.3 vol. %) and 13-137 (3.3 vol. %). The 5.2 vol. % porosity found in the grog-tempered sherd 15-93 exceeds the 4.3 vol. % pore space in the Ertebølle culture vessel (sample 137-27), where the voids mainly derive from burnt out organic temper. Fig. 8 shows the frequency distribution of pore sizes in the Neolithic pottery fragments.

Porosity in the sherd can develop from different sources. The amount and nature of porosity in pottery are strongly related to technological variations in the production chain (e.g., type of the clay and firing technique, Morariu et al., 1977; forming process: Lindahl and Pikirayi, 2010; firing: Tite et al., 2001). In hand-made pottery, different modes of forming a vessel leave distinct signatures in the ware. If organic temper is used, it will vanish during the firing process and create voids. The firing process will also cause shrinkage of the clay matrix and, if present, of grog components that, in previous use, might have been exposed to lower temperatures than those that prevailed in the production of their current host sherd. Additional cracks in the ware might develop during cooling because of varied thermal expansion coefficients of different fabric compounds. Finally, cracks can be caused by influences of the depositional background. In sherds 131-05 and 13-137 (both Funnelbeaker culture, see Figs. 3A, 4A and 3C, 4C), cavities related to the forming technique (see Section 3.4 below) and the

firing process occur. During the firing process, shrinkage of the clay matrix caused the development of cracks surrounding the rock fragment temper. In Sherd 138-67 (see Figs. 3B and 4B; presumably Funnelbeaker culture) voids surrounding the rock temper fragments are rare (and when present much thinner). In Sample 15-93 (see Figs. 3D and 4D), abundant cracks around rock fragment temper and grog temper occur. Fragment 137-27 (Figs. 3E and 4E) shows porosity that documents the use of organic temper.

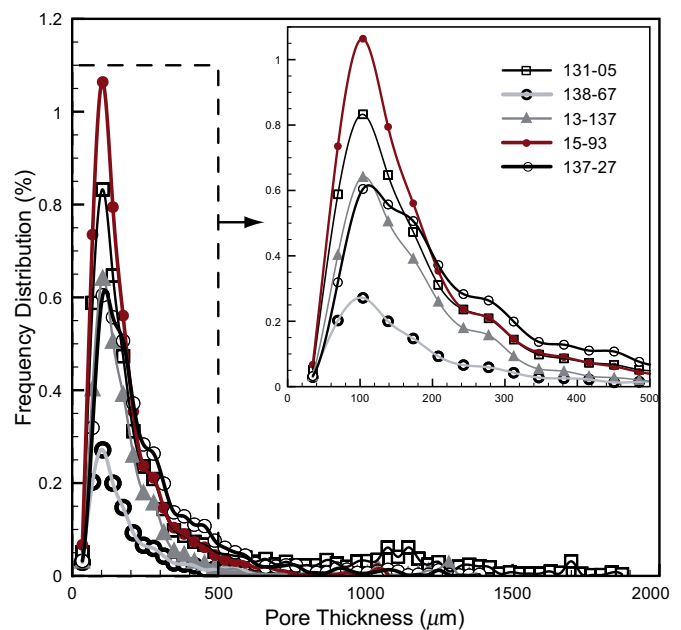


Fig. 8. Frequency distribution of different pore size classes contributing to the cavities in the Neolithic pottery fragments. The inset is a zoom-in of the part surrounded by the dashed line.

Investigation of the interconnectivity of the sherd porosity takes full advantage of the volumetric datasets provided by microtomography. The range of the connectivity within the porous network is visualised in the coloured renderings of the pore structure in Fig. 9: open pores that are connected to the sherd surface, are visualised in green; closed porosity is displayed in

yellow. Fragments 131-05 (Fig. 9A), 13-137 (Fig. 9C) and 15-93 (Fig. 9D) display abundant voids that penetrate the sherd. In sample 15-93, porosity around grog temper causes a large connectivity of the pore system. The porous structure of sample 138-67 (Fig. 9B) is different: characterised by abundant voids, no connectivity of the pores into the sherd can be observed. The development of large,

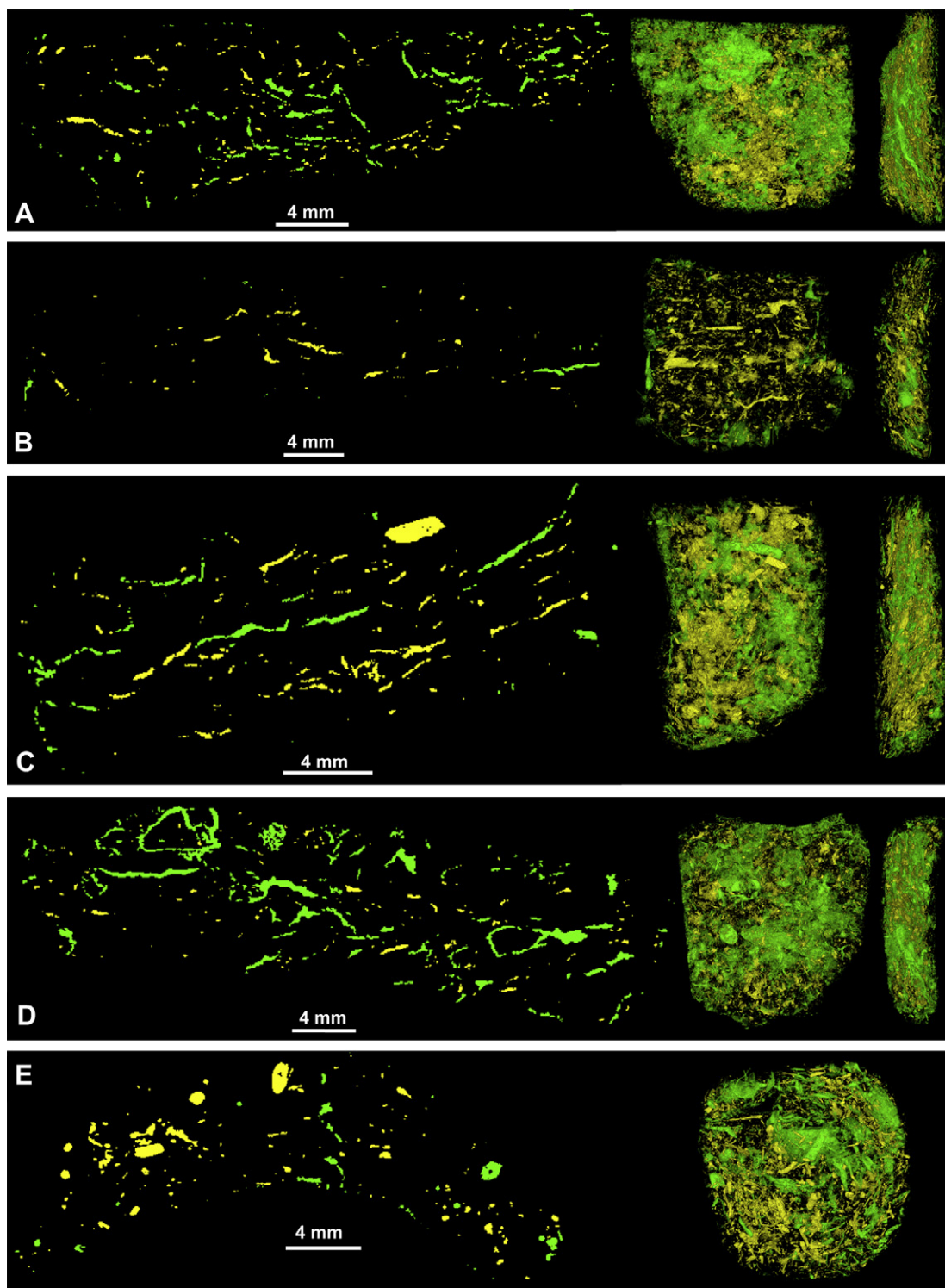


Fig. 9. Range of connectivity within the porous network of the sherds. Distinction of open (voids connected to the sherd surface, coloured green/dark grey) and closed porosity (coloured yellow/bright) in the Neolithic pottery fragments. (A) to (E) samples 131-05, 138-67, 13-137, 15-93 and 137-27. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

connected pores might have been hampered by the dense clay matrix. In sherd 137-27 (Fig. 9E), the frequent use of organic temper does not generate interconnected pore systems that invade the sherd. Instead, the pores that penetrate or crosscut fragment 137-27 can be related to cracks inherited from the depositional situation.

3.3. Organic temper

Inspections of the cavity morphologies reveal the use of organic temper in the Neolithic samples 138-67 (Fig. 10A), 13-137 (Fig. 10B) and especially in the Mesolithic fragment 137-27 (Fig. 10C). Most of the voids are not connected to the open porosity of the sherd and

thus would not be accessible for invasive methods. The voids caused by plant temper in sample 137-27 could already be seen by naked eye, but the organic inclusions in sample 138-67 and 13-137 were only detected by X-ray microtomography. The organic components in sample 137-27 show a marked heterogeneity, which implicates the use of different organic materials, may be coming from dung or moss. Most of the macroscopically observable impressions seem to come from broken straws, while others come from small leaves. Whereas the plant fragments in sample 137-27 were clearly added as a temper material, sample 138-67 and 13-137 show only few organic inclusions, which could be small rootlets and therefore natural components of the clay. The organic temper of sample 137-27 is characterized by low roundness values (see

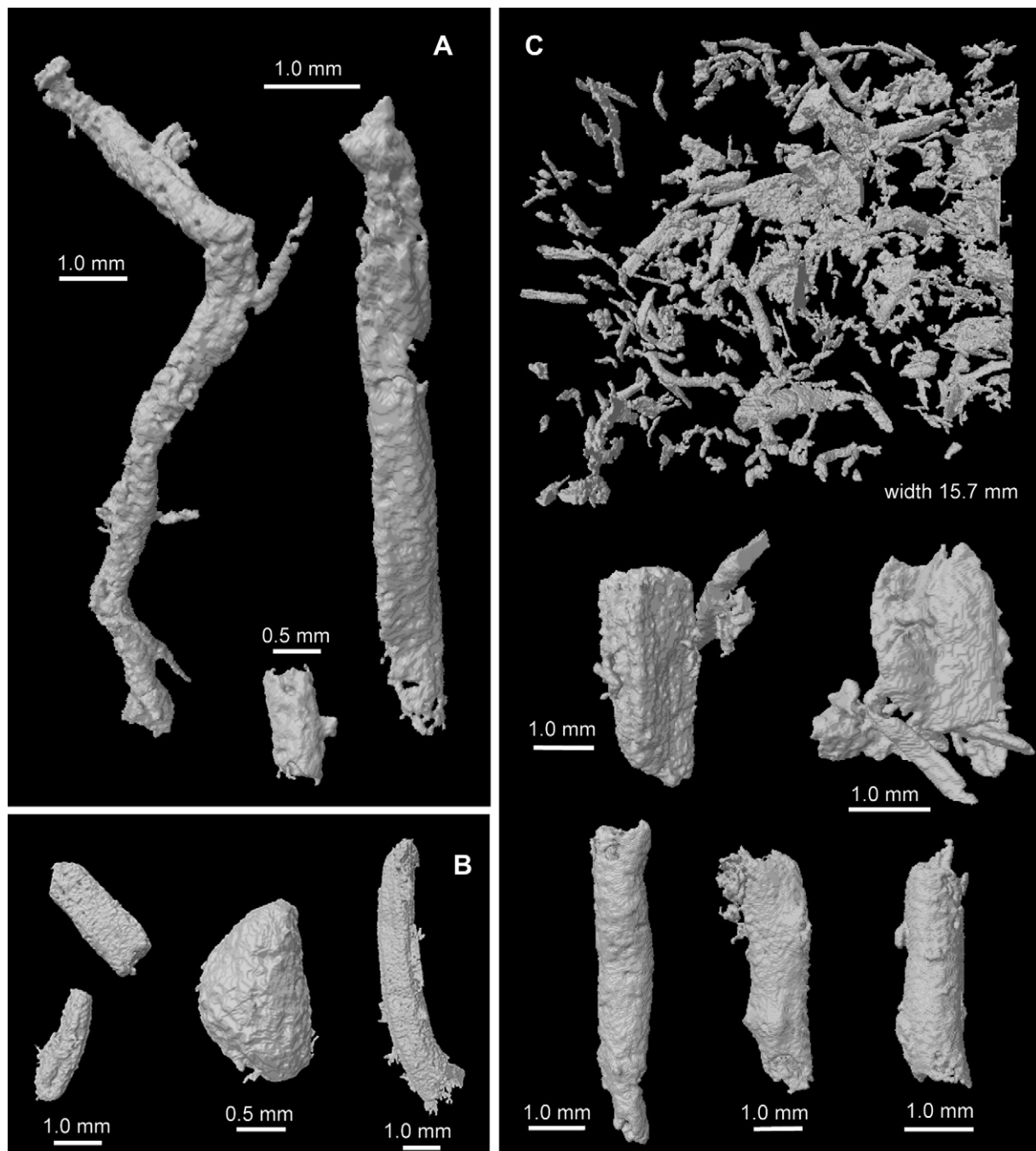


Fig. 10. Cavity morphologies reveal the use of organic temper agents in the samples 138-67 (A), 13-137 (B) and 137-27 (C).

Fig. 7), which indicates that the majority of the material used was of rod-shaped geometry.

The above-mentioned investigation of organic temper illustrates two advantages of the μ -CT method: (1) Cavities can be visualised and morphometrically analysed, although the organic temper was completely burnt out during the firing process, and thus no plant remains are available for direct analysis; (2) All cavities in the sample are included in the analyses. In the case of invasive methods, only the open porosity would be accessible for analysis.

3.4. Pore structures indicating vessel forming techniques

The orientation of the sherd porosity is very strongly influenced by the forming technique that was utilized by the potter. By stereo microscopic inspection of polished breakages impregnated with araldite plastic and a fluorescence agent under UV-light, Lindahl and Pikirayi (2010) presented the clearly distinct signatures that different modes of vessel forming impose on the pore structure. The simulated manufacture of four different vessels with either two types of coiling technique (U and N, see Lindahl and Pikirayi, 2010, Fig. 6) as well as two types of modelling technique (pressing or pulling of a lump of clay) revealed a clearly distinct curving and orientation of the pores from one wall surface to the other (specific for U- and N-technique) in contrast to the wall parallel pore structures of the two different modelling techniques. Among the two coiling techniques, a distinction is clearly evident by the orientation of the pores: the U-technique can be recognised by the typical u-shaped porous structures oriented from one wall surface to the other, whereas the samples formed with the N-technique exhibit the distinct diagonal orientation of the pores, pointing to the walls.

The volume renderings of the prehistoric sherds, cut at different levels perpendicular to the vessel walls, display characteristic features of the above mentioned forming techniques. The pore structure of the samples 131-05 (Fig. 11A), 13-137 (Fig. 11C) and 15-93 (Fig. 11D), pointing diagonal from one wall surface to the other, is indicative for the N-technique. In case of sample 13-137, the porous structure bends near the wall surface as a consequence of moulding the coils during the production process. In fragment 15-93, also the temper is aligned concordant with the pores. The volume cuts of sherd 15-93 permit an additional observation related to the firing process: the connectivity of the porous network, imposed on the clay matrix by the forming process, is disconnected by the rock temper. Cracks, developed due to the shrinkage of the clay body and expansion of the initial pore structure, cannot pass the rock temper. In contrast, the grog temper is surrounded by voids, indicating a differing behaviour during the firing process. The porous structure displayed by fragment 138-67 (Fig. 11B) can also be assigned to coiling by N-technique. However, the clay matrix is remarkably dense.

3.5. Clay matrix

The fine-grained nature of the clay matrix imposes certain constraints on the applicability of the μ -CT method. The resolution of the reconstructed images (17.3 μ m per cubic voxel) allows segmentation of the coarser compounds distributed throughout the clay matrix (i.e. silts and fine sands). Since the size of these “coarser” compounds is just above the object resolution, segmentation of clay matrix compounds is limited to mineral phases that are highly absorptive for X-rays (like biotite and oxides) that exhibit steep gradients to the surrounding clay body. Like in the case of temper, the frequency distribution of these phases can offer distinctive features to discriminate various clay sources.

Fig. 12 demonstrates the segmentation of heavy mineral particles in the clay groundmass in sherds 15-93 and 137-27. As a result of the segmentation procedure, the heavy minerals that belong to the rock fragment temper are not included in the selection. The

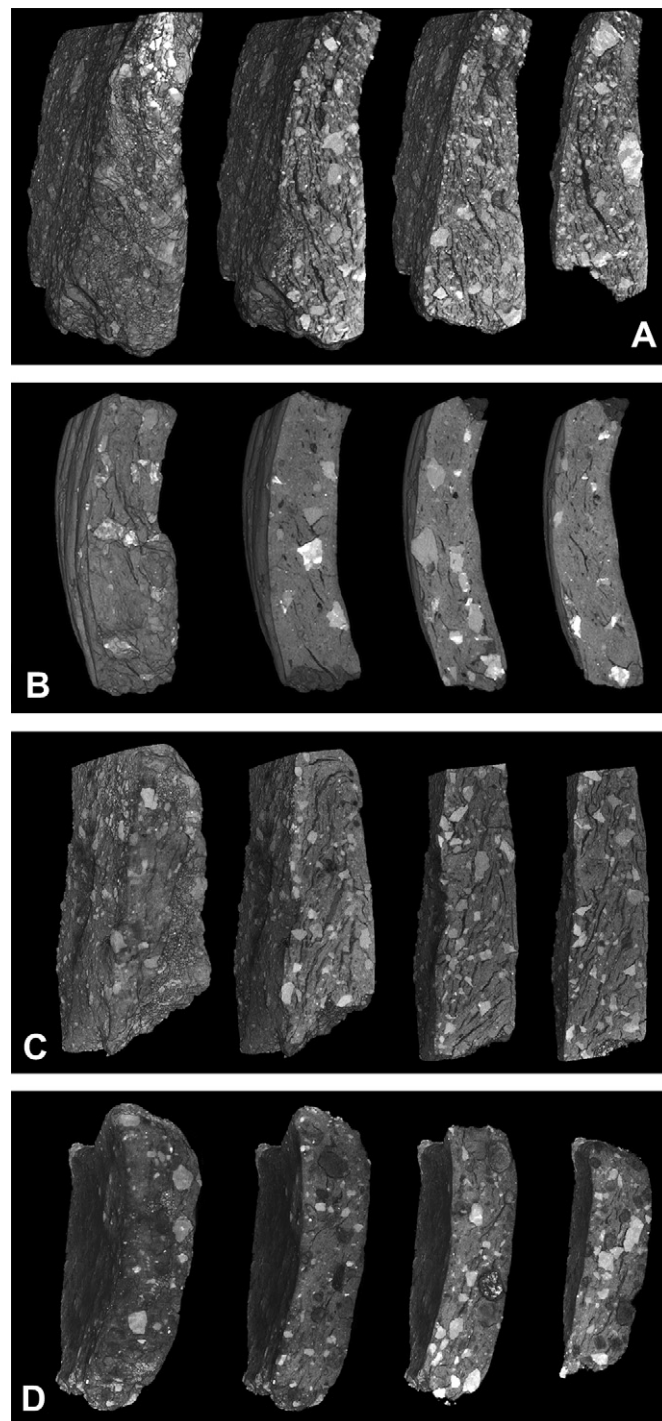


Fig. 11. Volume renderings of the ceramic fragments, cut at different levels perpendicular to the vessel walls. (A) sample 131-05 exhibits the distinct diagonal orientation of the pores from one wall surface to the other, that originate by forming with the N-technique. (B) fragment 138-67 displays a porous structure, that can be assigned to coiling with N-technique. However, the clay matrix is remarkably dense. These features could originate from a modelling technique. (C) and (D) samples 13-137 and 15-93, formed by N-technique. In fragment 15-93, also the temper is aligned concordant with the pores.

frequency distribution of highly absorptive minerals in the clay groundmass within different size classes is shown in Fig. 13. In samples 138-67, 13-137 and 15-93 particle numbers peak in the second smallest size class, i.e. within the resolution of the μ -CT scanner. Note the high number of small heavy mineral particles in the clay groundmass in sherd 131-05 and the lower abundance in sherd 137-27. The maximum of the grain frequency distribution in samples 131-05 and 137-27 tends to occur below detector resolution.

However, since X-ray μ -CT distinguishes phases on the basis of their linear attenuation coefficient (which depends directly on electron density and effective atomic numbers of the mineral, and the energy of the incoming X-ray beam), a qualitative evaluation of the homogeneity of the clay matrix is within the scope of microtomography. Figs. 3E and 12C exhibit the heterogeneous nature of the clay matrix in the Mesolithic sherd 137-27.

4. Discussion

In order to find distinctive markers for technological features that can supplement findings on typological characteristics as well as results of petrographic and chemical analyses of the ceramic material, we employed high-resolution X-ray microtomography to distinguish the ceramics on the basis of their plastic and non-plastic components.

By means of true 3D morphometric analyses of the reconstructed μ -CT images it has been possible to characterise nature, abundance and differences in the preparation of temper materials

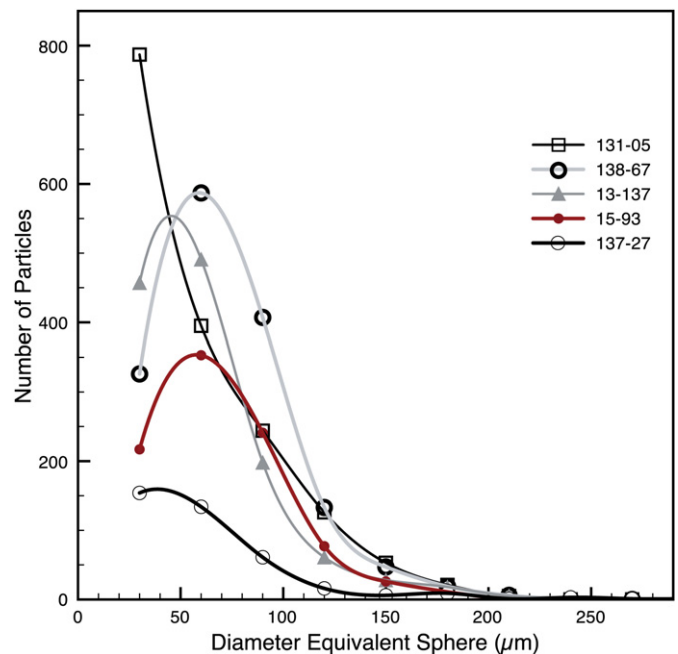


Fig. 13. Abundance of heavy minerals in the clay groundmass in different size classes in the Neolithic pottery fragments. Note the high number of small heavy mineral particles in the clay groundmass in sherd 131-05 and the lower abundance in sherd 137-27.

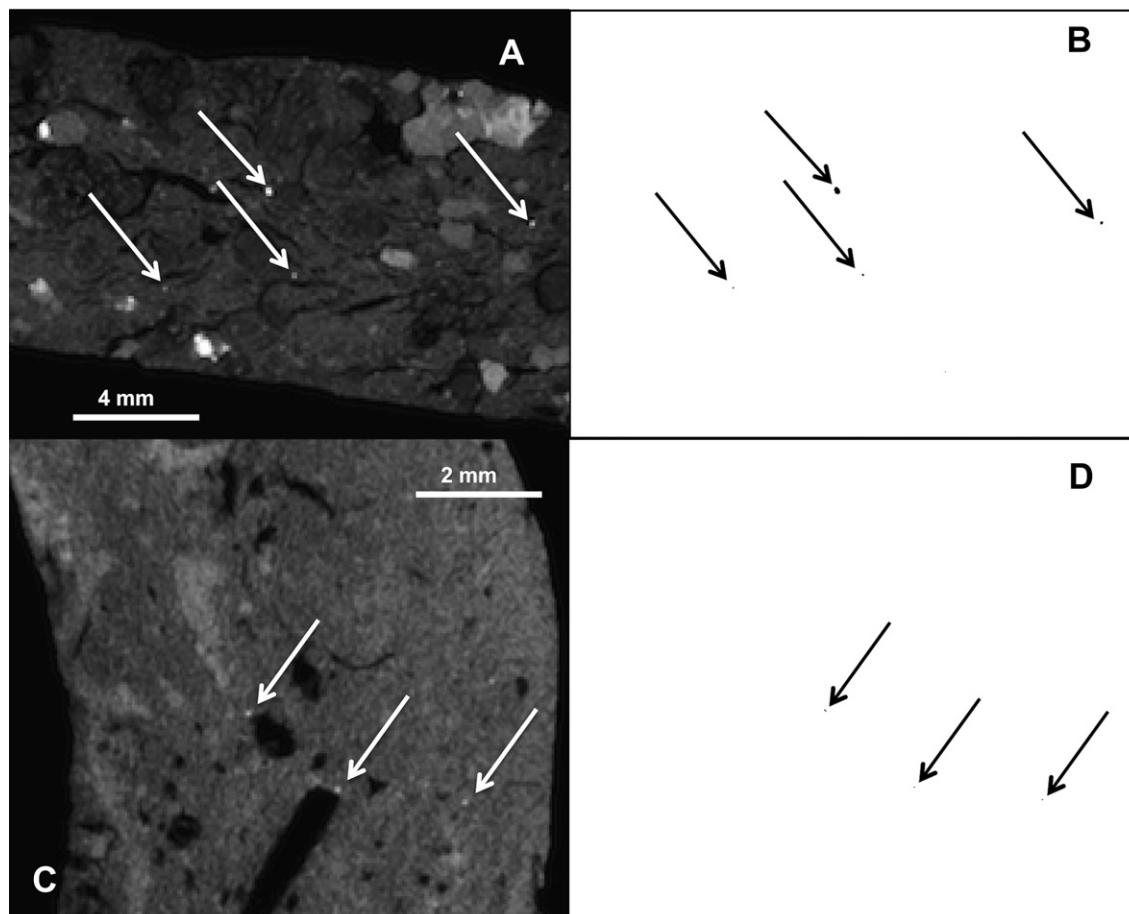


Fig. 12. Segmentation of heavy mineral particles in the clay groundmass of the sherds. (A) and (B) sample 15-93, (C) and (D) sample 137-27. As a result of the segmentation procedure, the heavy minerals that belong to the rock fragment temper are not included in the selection (B), (D).

(Section 3.1). As a consequence of the highly contrasting behaviour of the weakly attenuating matrix and the strongly attenuating rock fragment temper concerning the absorption of X-rays, the latter could be easily segmented by differing grey values in the reconstructed images. Therefore, the 3D analysis of the volume fraction of rock fragment temper in the sherds was straightforward. Moreover, a comparison with the results from 2D analyses of single image slices (comparably to thin sections in terms of thickness) revealed a fundamental impact of the sampling position for thin section preparation on the results of 2D temper analysis. Also the determination of abundance (particle numbers) and frequency distribution (particle size) of the rock fragments yielded distinctive differences between the sherds concerning the sorting grade, which could be indicative for a deliberate use. In contrast, the morphometric characterisation of rock fragment temper in terms of particle shape could not identify significant differences between rock fragments contained in different sherds. However, this is not to be expected, as the shape of crushed rock fragments results from the fracture behaviour which is characteristic of the material itself. On the other hand, shape analysis is distinctive when applied on different materials, as indicated by the differences in shape between rock fragments, grog and organic temper. Although not present in the range of temper materials within our samples, we presume that certain modes of temper (such as detrital quartz grains in sandstones derived from beach sediments, crushed shell) could be distinguished by shape, as indicated by the work of Lin and Miller (2005) on shape analysis of quartz sand and rock particles. The segmentability of grog, which exhibits an attenuation coefficient only slightly lower than that of the clay matrix, is hampered by a certain scatter in the attenuation properties of both fabric compounds. Therefore, the segmentation of this solid temper phase involves a lot of user interaction and sophisticated filtering processes, which limits its practicability. However, for the human eye (in contrast to the automated segmentation procedure) the differences in attenuation are clearly visible, even more pronounced than the differences in birefringence of fired clay matrix and grog in thin section. Hence, the very inspection of the image material by volume reconstruction and appropriate clipping methods (i.e. cutting of the volume reconstructions from different angles) provides valuable insights in abundance and distribution of grog temper within the ceramic fragments.

Morphometric analysis of volume fraction and thickness distribution of the porous system (Section 3.2) within the sherds yields distinctive quantification of the porosity. Investigation of cavity morphology, range of connectivity and orientation of voids reveal information concerning the origin of the pores (caused by organic temper, forming technique or shrinkage during firing or cooling). The application of high-resolution microtomography is of particular importance to derive information about the nature of organic temper, when all plant remains were completely lost during the firing process. Despite the lack of organic material available for direct analysis, visualisation of the true 3D volume data and its detailed morphometric characterisation (Section 3.3) facilitates cooperation with archaeobotanists for possible specification of the origin of the organic material. Furthermore, μ -CT has proved to be extraordinary powerful in the investigation of the manufacturing technique that was applied in the forming process of the vessels (Section 3.4). By clipping of volume reconstructions and by visualisation of the range of connectivity within the sherds, distinct differences in morphology and orientation of the pore structure can be used to infer the actual coiling technique that was applied. Although stereo microscopic inspection of polished breakages impregnated with araldite plastic and a fluorescence agent under UV-light reveals the clearly distinct signatures that different modes of vessel forming imposed on the pore structure, the investigation is accompanied by a destructive

preparation of the samples. In contrast, high-resolution μ -CT yields multiple views by non-destructive and non-invasive morphometric characterisation.

Although limited by the resolution of the reconstructed images, distribution analysis of the coarser compounds of heavy minerals in the clay matrix can offer distinctive features to quantitatively discriminate various clay sources. As demonstrated by the investigation of the Mesolithic sherd, a qualitative evaluation of the homogeneity of the clay matrix is well within the scope of microtomography. As in the case of grog temper, valuable information concerning the heterogeneous nature of the clay material is provided by the image material.

5. Conclusion and archaeometrical perspectives

In this study, we used high-resolution X-ray microtomography as a tool to investigate the microstructure of selected prehistoric potsherd samples from the Endmesolithic–Neolithic site Hamburg-Boberg 15 (northern Germany). The potential and the limitations of the μ -CT method, concerning the characterisation of nature, preparation and abundance of temper agents, have been addressed. Furthermore, we reported the excellent applicability of μ -CT observations on cavity morphology and orientation of the pore structure to infer the coiling technique that was applied in the forming process of the vessels.

As the pronounced heterogeneity of prehistoric earthenware requires a careful inspection of large parts of the ceramic microstructure, high-resolution microtomography is a valuable, supplementary research tool for the non-destructive and non-invasive investigation of ceramics. The μ -CT method and its ability for computer-aided morphometric analyses offers a variety of advantages for the inspection and quantification of textural distinctions, such as non-destructive analysis of nature and spatial arrangement of the fabric compounds. Moreover, by morphometric quantification of volume and surface properties as well as size distributions of fabric compounds, differences in amount or preparation of the temper can be characterised. X-ray microtomography is extraordinarily suited to infer the use of organic components, even with all plant remains completely burnt out during the firing process. As microtomography is sensitive to both open and closed porosity, all cavities in the sample are included in the μ -CT analyses. Furthermore, the range of the connectivity within the porous network can be visualised in coloured renderings of the pore structure. In contrast, for invasive methods only the volume fraction of the porous structure that is connected to the sherd surface is accessible for analysis. Quantitative analysis of clay matrix compounds is limited to highly attenuating mineral phases. However, valuable information concerning the heterogeneity of the clay material is provided by image analysis of the volume reconstructions.

Supplementary to petrographic and geochemical analyses of ceramic materials, high-resolution X-ray microtomography is a valuable research tool in the investigation of pottery. The method has considerable potential to contribute to the reconstruction of the production technology, e.g. processing of raw materials or forming techniques. The results of both qualitative and quantitative morphometric analyses of the true three-dimensional image material can then, in turn, be combined with chemical analyses. Moreover, in case of a relatively uniform geological background, when thin section petrography or geochemical analysis of major and trace elements might not be distinctive, analysis of the frequency distribution of temper among different size classes may yield distinguishable results. Furthermore the μ -CT method could be utilised to ensure the homogeneity of samples envisaged for geochemical analyses, especially with respect to the distribution of chemically diverse fabric compounds. As high-resolution

microtomography is an adequate method to infer the use of organic components, an important development for the future will be a systematic cataloguing of macroscopic botanical remains to characterise production sites in conjunction with vegetation history. As underscored by our investigations, the application of high-resolution X-ray microtomography presented here may open new avenues for the study of ceramic technology.

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