EUROPEAN UNION
European Structural and Investment Funds
Operational Programme Research, Development and Education

MINISTRY OF EDUCATION, YOUTH AND SPORTS

ICTMS 2022
5th International Conference on Tomography of Materials & Structures
27th June- 1st July 2022, Grenoble, France

Thermal-induced changes of organic temper in archaeological ceramics with helical trajectory X-ray CT

Correlation of volume in time and with Optical and Scanning electron microscope

Jaques V.A.J. [1,*], Blažek P. [1], Petřik J. [2], Slavíček K. [2], Břínek A. [1], Zikmund T. [1], Kaiser J. [1]
[1] CEITEC—Central European Institute of Technology, Brno University of Technology, Purkyňova 123, Brno 612 00, Czech Republic
[2] Department of Geological Sciences, Masaryk University, Kotlářská 2, 611 37 Brno, Czech Republic

Archeological ceramic is full of information from the socio-economic relations within a society to the food storage practices. Various **admixtures** are added to enhance **specific properties** of the ceramic. **Hay** is an under evaluated admixture and when fired, the organic part partially or fully "disappears", thus making it harder to identify. The organic residual pores have the specific morphology of the organic matter and by the analysis of their **volume, shape** and potential organic **residues**, the firing conditions could be determined as well as the admixture/temper used.

We compared **raw and fired experimental ceramic** at **500°C** in **oxidative** and **reductive** atmosphere.

AIM Non-destructively correlate hay residual pores within a ceramic object to its firing conditions

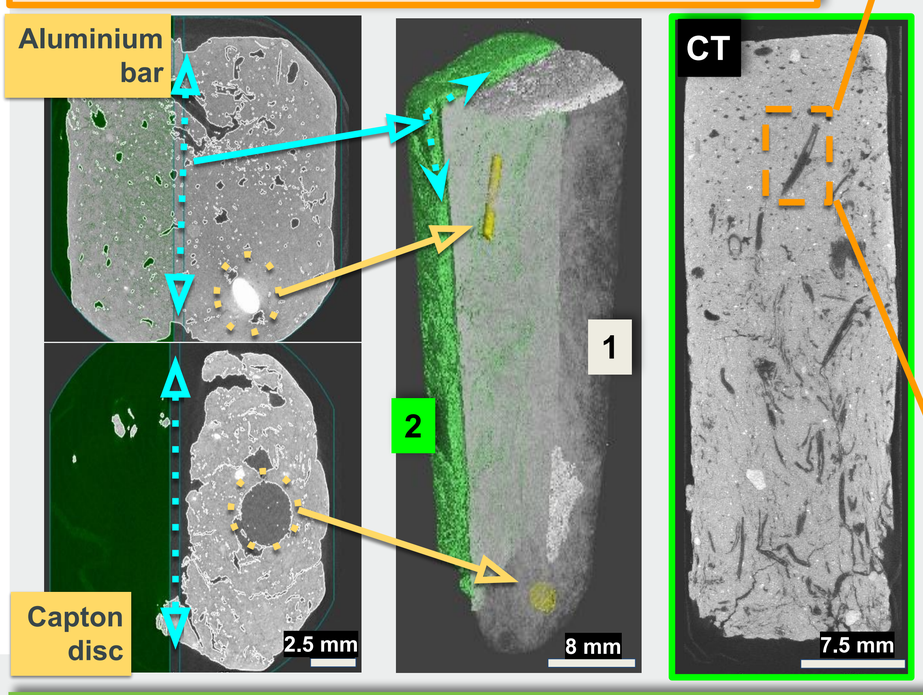
Segment and quantify residual organic matter from original voids and ceramic paste **CHALLENGE**

IMAGE and VOLUME CORRELATION

CT data volume correlation Before/After Firing → Aluminium bar Capton disc

Computed Tomography (**CT**), Optical Microscope (**OM**), Scanning Electron Microscope (**SEM**) correlation → Ceramic shaping

The ceramic was cut in two parts after the shaping guide:
1. To be fired and re-scanned
2. To transform in cross-sections for methods correlation (Green)



SCAN PARAMETERS

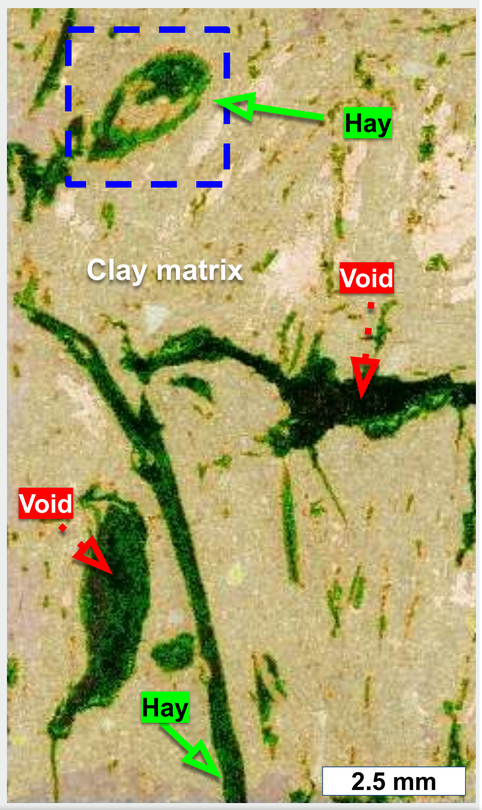
Heliscan ThermoFisher Scientific

Trajectory	Space-filling
Energy	120 kV
Current	130 µA
Exposure	2.099 s
Voxel size	7.1033 µm
Software	VG Studio 3.5

HAY IDENTIFICATION

★ In 3D (Check slice from all XYZ axis)
★ By correlation with other visual methods

Only referring to a single CT slice → it is likely that the interpretation will be wrong on what is a hay twig or not.
The mixing of the hay and the ceramic paste as well as the drying can change the natural shape of the twig.
Broken, cut, flatten and twisted hay twigs occured.

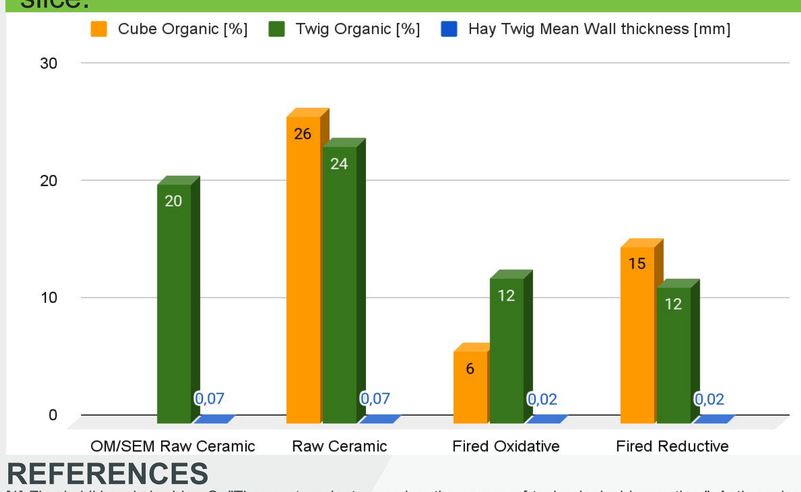


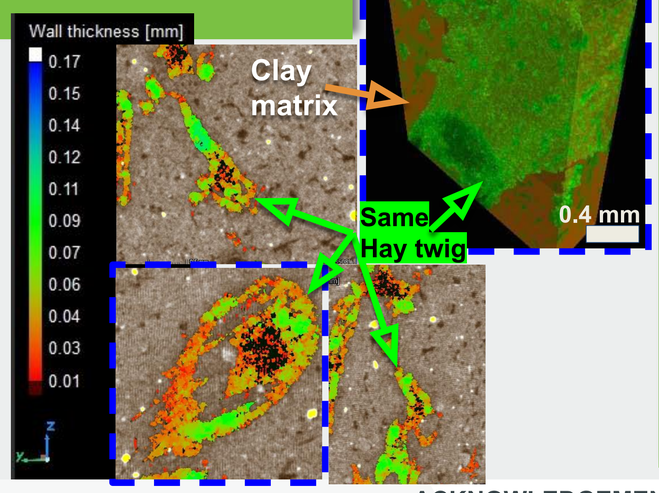
HAY DEGRADATION

The **hay degrades** during the firing process of the ceramic and leaves residues of organic matter and specific shaped-voids. The residues encountered depends directly from the temperature and the oxygenation while the hay voids are relatively independent from the firing process.

HAY QUANTIFICATION

Quantification of organic matter was done once from a specific volume (**Cube**) with segmented components Clay/Organic/Air and then from an extracted a **Single twig**. The mean wall thickness was similar (+/- 0.01 mm) between the volume and the twig analyses. The organic content OM/SEM is a calculation based on the 2D slice.





PRIMARY CONCLUSIONS

1. The **low contrast organic/air** keeps challenging the segmentation algorithms.
2. There is a **lower amount of organic matter** in Fired ceramic than in the Raw paste.
3. **No clear difference** was quantifiable between the two firing conditions, reductive and oxidative.
4. The **voids and organic residues** after firing can help to assess the temper material used.

Further correlative measurements will refine these results.

REFERENCES

[1] Zhushchikhovskaia, Irina S. "The most ancient ceramics: the course of technological innovation." *Anthropology & archeology of Eurasia* 51.1 (2012): 62-78. DOI: 10.2753/AAE1061-1959510103
[2] Riegert, D., and K. Konopka. "Analysis of the structure of archeological objects-ceramic pottery." *Archives of Metallurgy and Materials* 56.1 (2011): 163-170. DOI: 10.2478/v10172-011-0019-7
[3] Hein, Anno, and Vassilis Kilikoglou. "Modeling of the microstructure of ancient functional ceramics and assessment of their performance." *Procedia Structural Integrity* 10 (2018): 219-226. DOI: 10.1016/j.prostr.2018.09.031
[4] Müller, Noémi S., et al. "The influence of microstructure and texture on the mechanical properties of rock tempered archaeological ceramics." *Journal of the European Ceramic Society* 35.2 (2015): 831-843. DOI: 10.1016/j.jeurceramsoc.2014.09.025

CEITEC – Central European Institute of Technology
Brno University of Technology, Purkyňova 123
612 00 Brno, Czech Republic
www.ceitec.eu

*** Contact person**
M.Sc. Victory A. J. JAQUES
e-mail victory.jaques@ceitec.vutbr.cz
phone +420 541 149 867

ACKNOWLEDGEMENTS

We acknowledge the CzechNanoLab Research Infrastructure supported by **MEYS CR (LM2018110)** and Jozef Kaiser thanks for the support of the grant **FSI-S-20-6353**. Victory A. J. Jaques thanks the support from **Grant 2234/62900/21** realised within the project Quality Internal Grants of BUT (KInG BUT), Reg. No. CZ.02.2.69 / 0.0 / 0.0 / 19_073 / 0016948, which is financed from the OP RDE.

MUNI

CEITEC BRNO UNIVERSITY OF TECHNOLOGY