



Introduction

The knowledge of the elemental composition of carbon rich samples of biogenic, geogenic or technological origin (Fig. 1.) is important for further applications as well as for the identification of the origin via an elemental fingerprint.

The complete digestion of these samples is still an analytical challenge [1]. In previous studies we observed that the digestion efficiency depends on the crystallinity and the grain size (in the case of flake graphite).

Hypothesis:

- **Digestion rate depends on the structure and morphology of the carbon.**
- **Microwave induced combustion leads to a more complete digestion.**

Aim of the work:

Development of a rapid and complete digestion procedure.

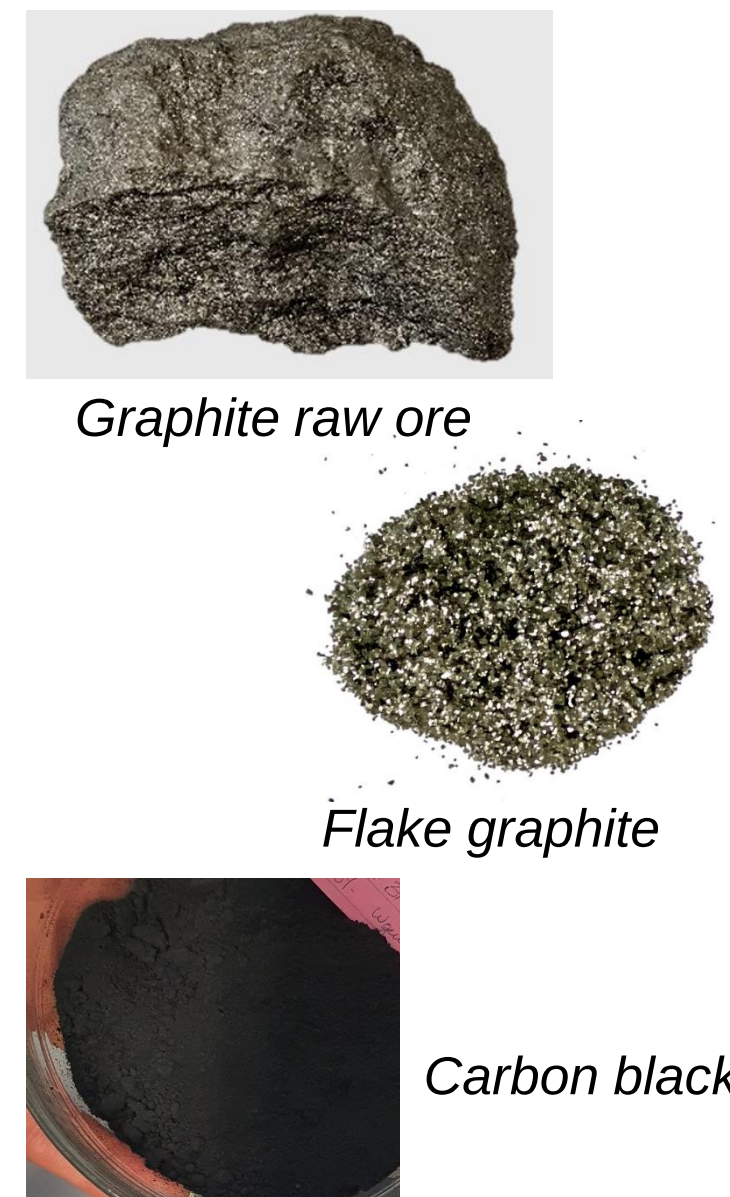


Fig. 1: Types of carbon

Structural characterisation

SEM (Scanning electron microscope):

ZEISS® EVO MA 10 scanning electron microscope (SEM)

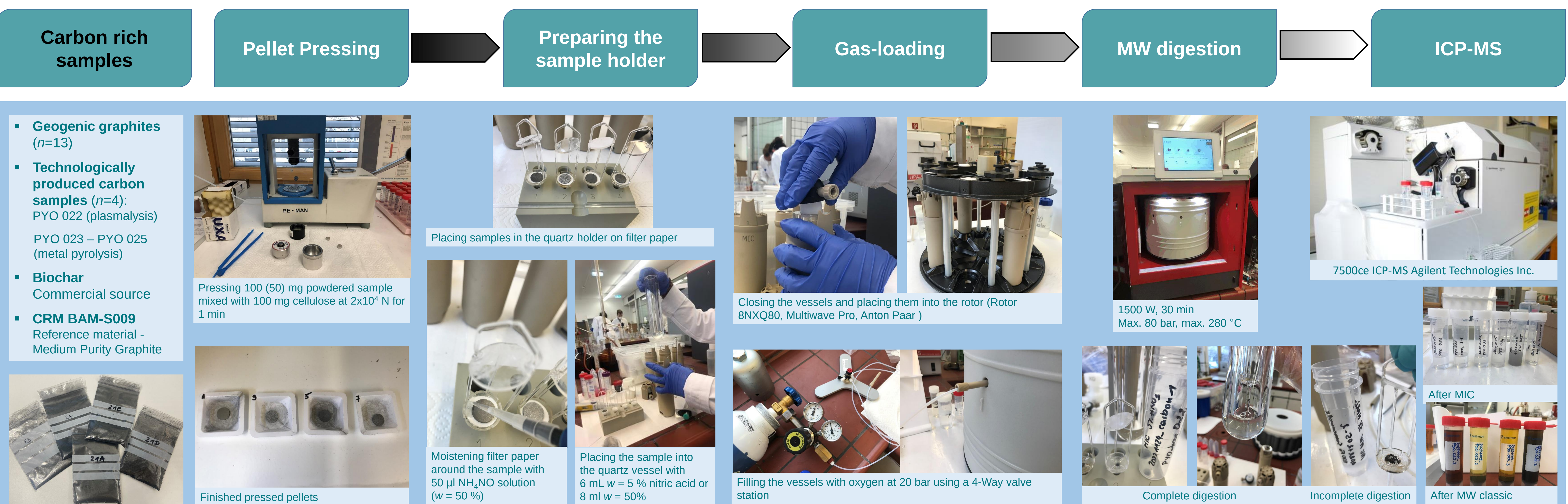
- Morphology characterization via secondary electron images

RAMAN spectroscopy:

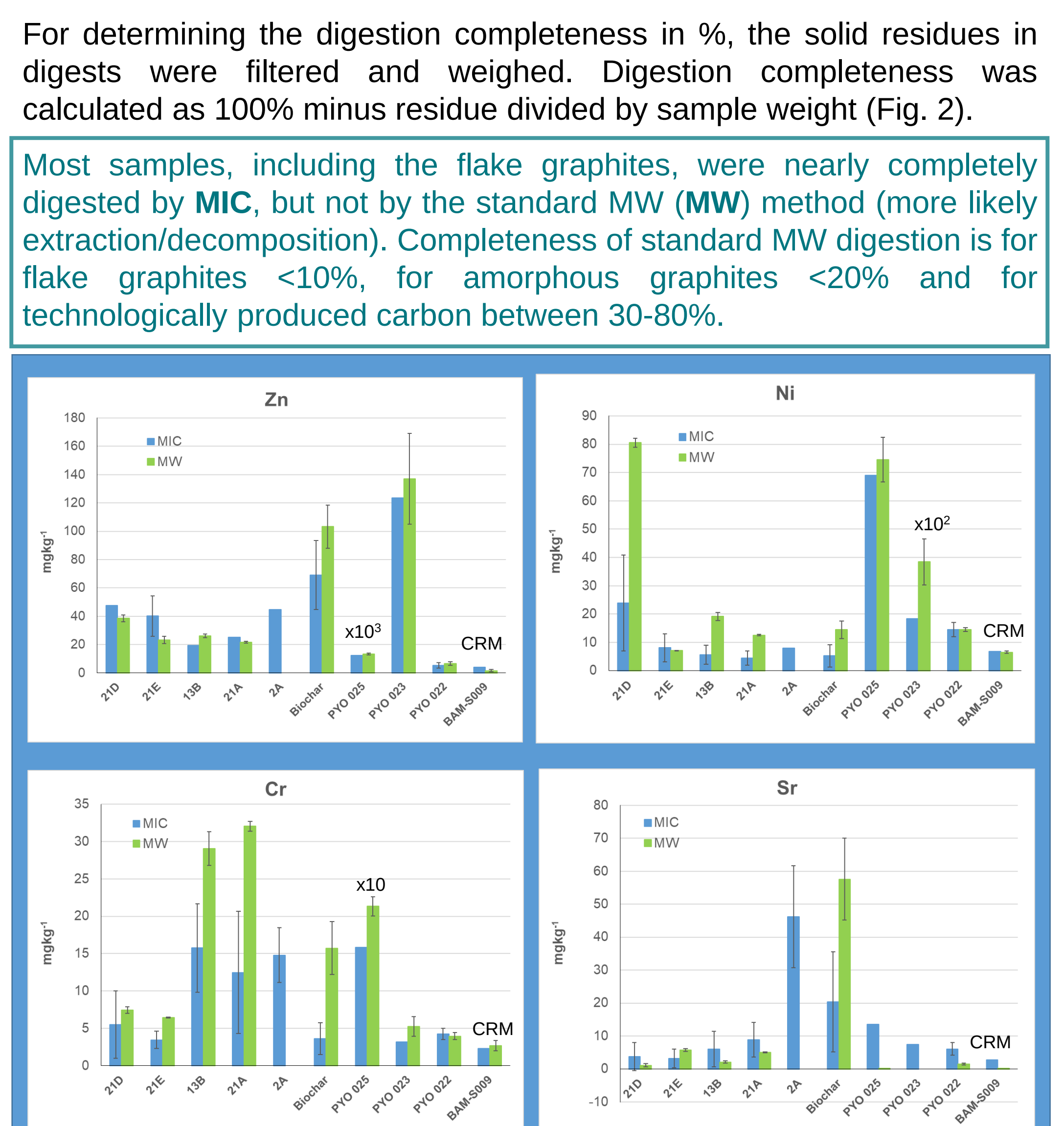
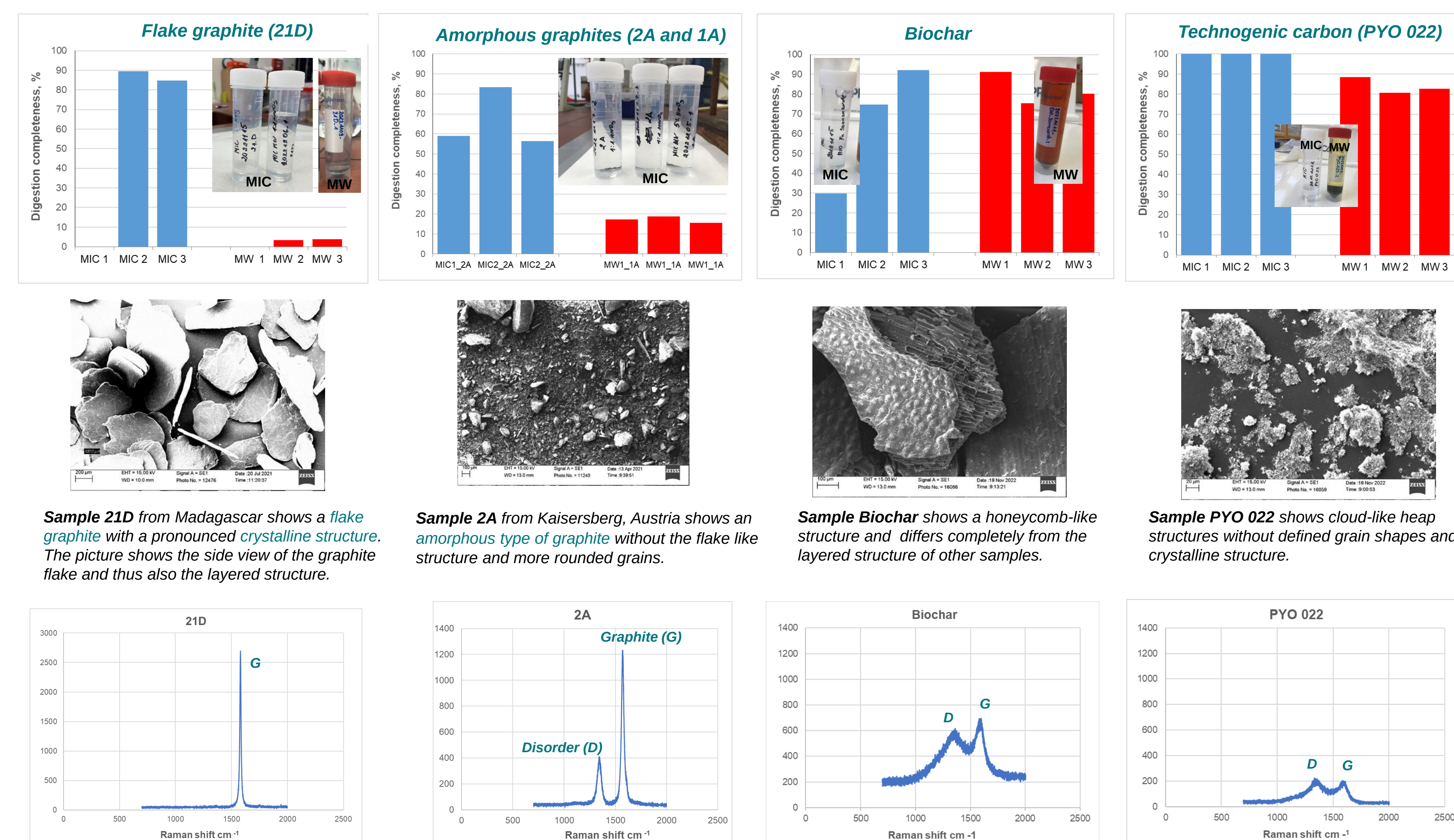
Horiba Labram HR Evolution instrument equipped with a 100mW Nd:Yag (532 nm) laser

- Characterisation of phase mineralogy at the micrometric-scale.
- Evaluation of the lattice ordering.

Microwave-induced Oxygen combustion (MIC) digestion for ICP-MS analysis



Results



References

[1] Cruz, S. M. et al. (2015). Microwave-induced combustion method for the determination of trace and ultratrace element impurities in graphite samples by ICP-OES and ICP-MS. *Microchemical Journal*, 123, 28-32

[2] Rantitsch, G. et al. (2016). On the discrimination of semi-graphite and graphite by Raman spectroscopy. *International Journal of Coal Geology*, 159, 48-56

Conclusions

- **MIC appears to lead to more complete digestions.**
- **Both digestion methods give comparable results for some selected elements.**
- **The reproducibility of the MIC results are considerably lower (incomplete combustion, lower sample mass, contamination by cellulose (Sr), pellet pressing).**
- **The completeness of digestion correlates with SEM and RAMAN results for the structural properties and the associated grain morphology.**
- **Results of SEM, RAMAN are important parameters to discriminate the various graphite types.**

