Characterization of multiscale porosity in activated carbon by X-ray tomography and FIB-SEM

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1 Introduction

Many natural and man-made porous media exhibit pore sizes covering a wide range of length scales. This so-called multi-scale porosity greatly impacts key-properties such as the material's reactivity and permeability and are of interest in areas including energy storage, adsorption, and catalysis.

Activated carbon features an exceptional porosity, a high specific surface area, and a remarkable adsorption capacity. Furthermore, the porous structure of activated carbon enables selective interactions with diverse substances, facilitating efficient removal of pollutants and enabling specific chemical reactions, which render the material ideal for applications in fields such as catalysis, water purification, air decontamination, and energy production [1][2].

The characterization of the multiscale porosity in activated carbon is challenging due to its wide range of pore sizes. Conventional characterization techniques such as mercury porosimetry, X-ray tomography or Scanning Electron Microscopy (SEM), when used independently, offer limited insights into the complete range of pore sizes present in these materials [3]. Consequently, there is a need to develop combined approaches that enable a more comprehensive characterization of multiscale porosity.

2 Methodology

In this study, we propose the combined use of X-ray tomography and FIB-SEM as a promising approach to visualize and quantify the pore system at different scales, aiming to achieve a better understanding of the pore structure and properties of multiscale porous activated carbon samples (Figure 1). By integrating these techniques, we can obtain a more comprehensive characterization of multiscale porosity, incorporating fine resolution information and an extended field of view.

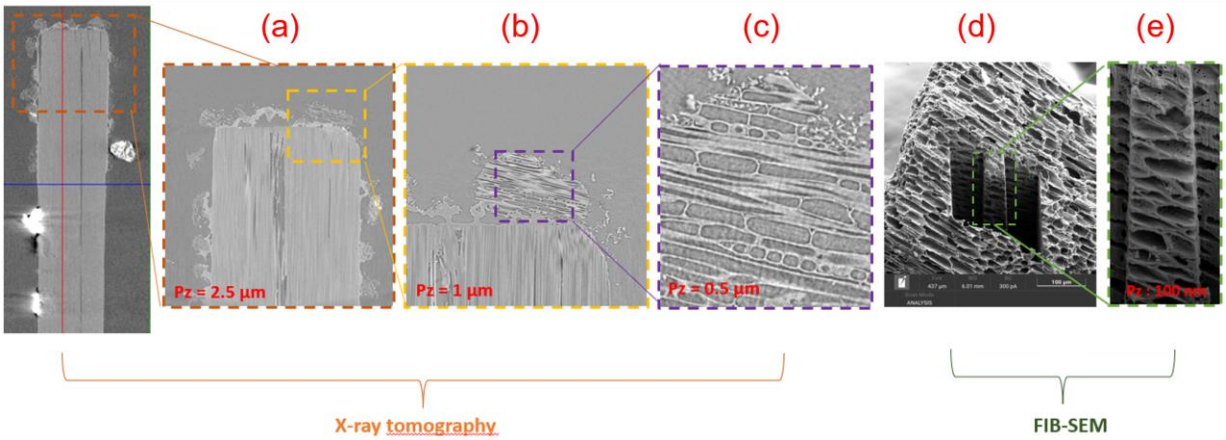

Figure 1: Coupling X-ray tomography and FIB-SEM to characterize activated carbon.

A large number of activated carbon grains were fixed on a carbon rod by means of x-ray transparent varnish. A first X-ray tomography was performed with a Zeiss Xradia Versa 510 at a resolution of 2.5 µm/voxel and allowed for the localization of the different grains as well as an initial impression of their

shape (Figure 1a). A grain was selected and imaged in more detail, first at 1 µm/voxel to capture the full grain (1b), and then at 0.5 µm/voxel (1c) providing more precise information about the macro-scale porosity and its associated morphological and topological parameters. Next the carbon rod was positioned in a FIB-SEM (Tescan Amber X) and a 10 nm conductive carbon coating was applied. The FIB enabled cutting a trench around the region of interest (1d). By intermittently removing material with the FIB and taking images at 100nm/pixel with the SEM, a 3D stack of the volume of interest was obtained (1e). The latter enabled quantifying the part of the pore system invisible by X-ray tomography. This combination of techniques provided a comprehensive view of the pore structure of the seeds, offering valuable insights into pore sizes and shapes (Figure 2) as well as their connectivity (not shown).

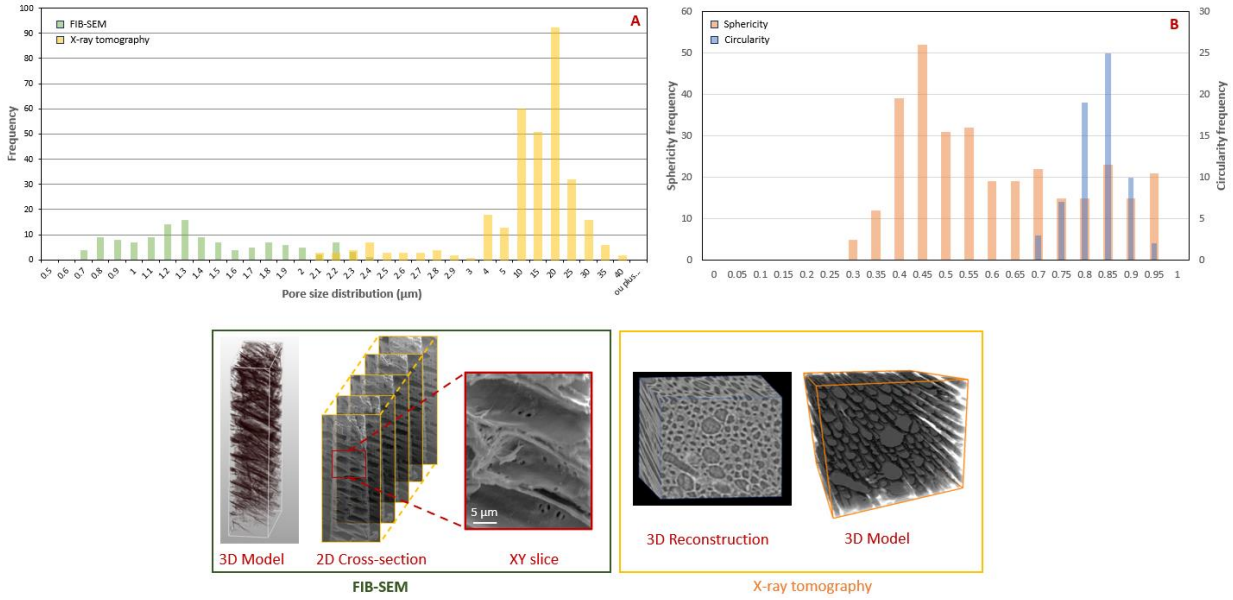

Figure 2: (A) Pore size distribution and (B) sphericity and circularity for the multi-scale porosity present on activated carbon and obtained by combining FIB-SEM and X-ray tomography.

3 Conclusion

In this work, we showed a successful application of true correlative imaging involving both X-ray tomography and FIB-SEM. An activated carbon bead acted as a vehicle to demonstrate how the proposed approach enables to characterise the multi-scale pore system from nanometre up to the millimetre scale. We showed the successful determination of the pore size and shape distribution, yet the methodology is applicable to other morphological parameters as well. The proposed innovative methodology provides a deeper understanding of pore structure and properties, opening up new opportunities for optimizing and utilizing multi-scale porous materials in various industrial applications, including in catalysis, adsorption, energy storage, water remediation and many others.

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