# 3D insight into the hierarchical structure of mesopores via a quantitative TEM analysis of FAU-Y zeolites


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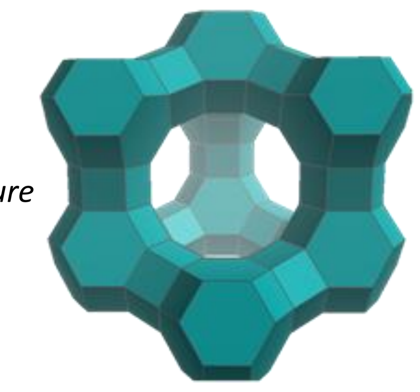
## Introduction

Zeolites are crystalline microporous aluminosilicates. They represent one of the key compounds in the evolution of the heterogeneous catalysis.

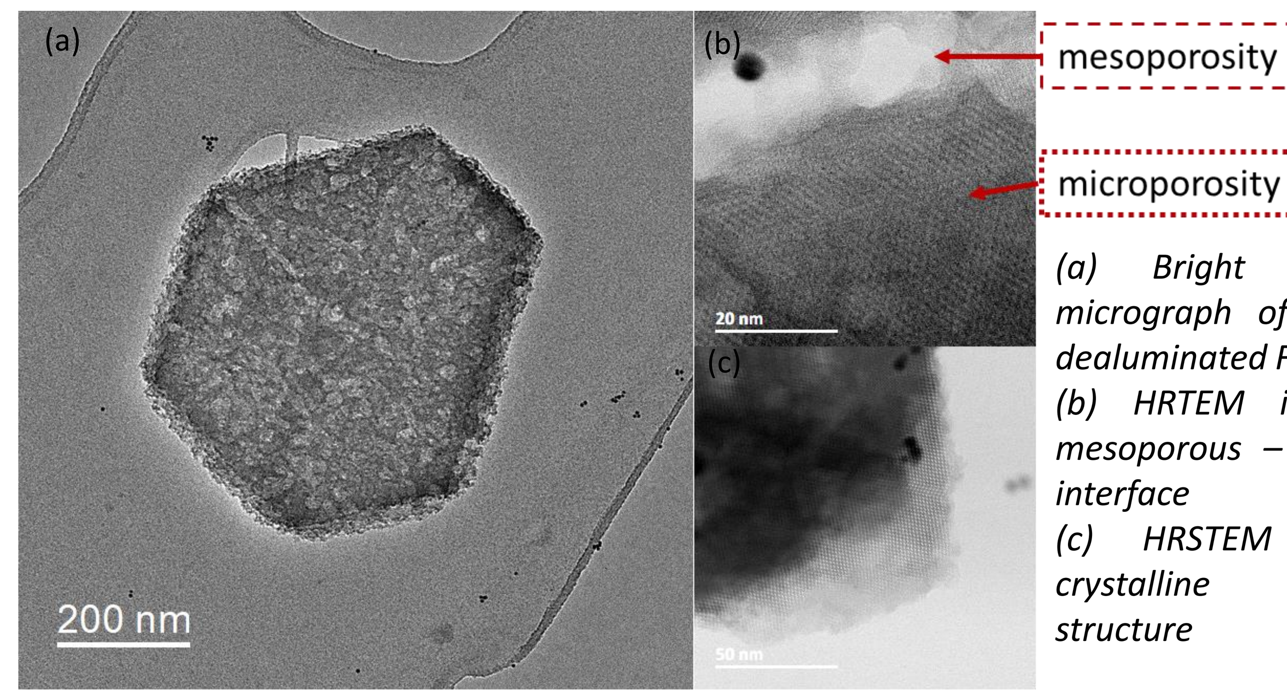
They combine indeed:

- Mass transport
- Solid acidity
- Ionic exchange

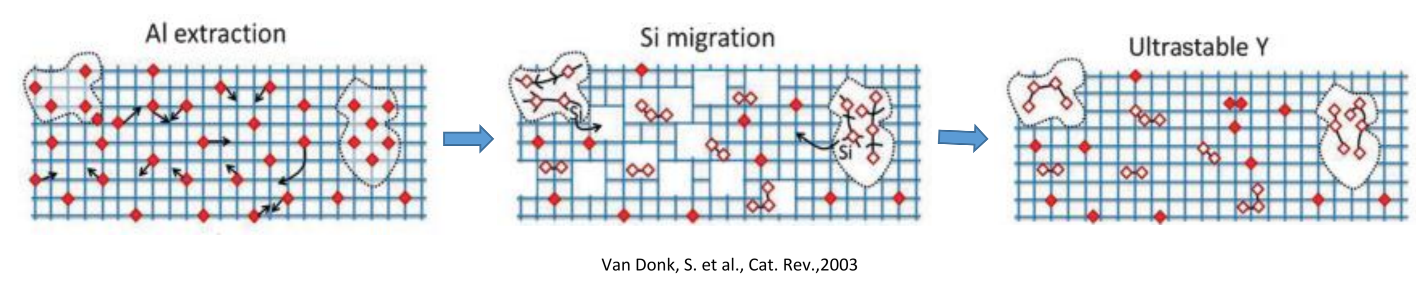


*Faujasite structure*

A hydrothermal treatment is generally performed for enhancing these properties by introducing a hierarchical porosity. Through the removal of framework Al, the structure rearranges for accommodating mesopores and hydrothermally stabilizes.




*(a) Bright Field TEM micrograph of a grain of dealuminated Faujasite Y*
*(b) HRTEM image of a mesoporous – microporous interface*
*(c) HRSTEM image of crystalline microporous structure*




*The steps of the dealumination mechanism during a steaming : the extraction of Al from crystallographic sites is triggered by the nucleation of point defects, followed by the migration of the species until reaching a mesoporous ultrastable Y form (USY) in Faujasite Y zeolites*

## Objectives

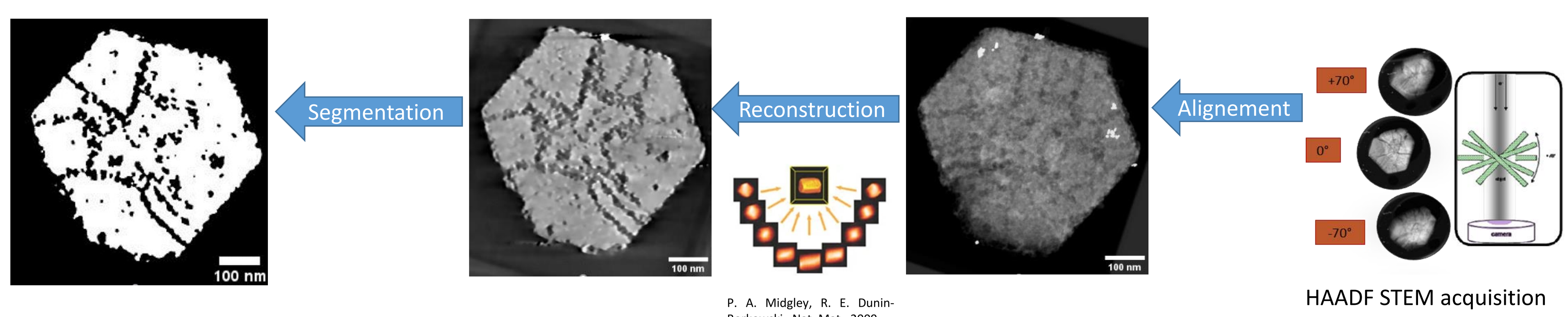
In this frame, a 3D TEM-based study is used for investigating

- the evolution of the porous network during dealumination within a Faujasite Y structure
- quantify the variation in the morphology

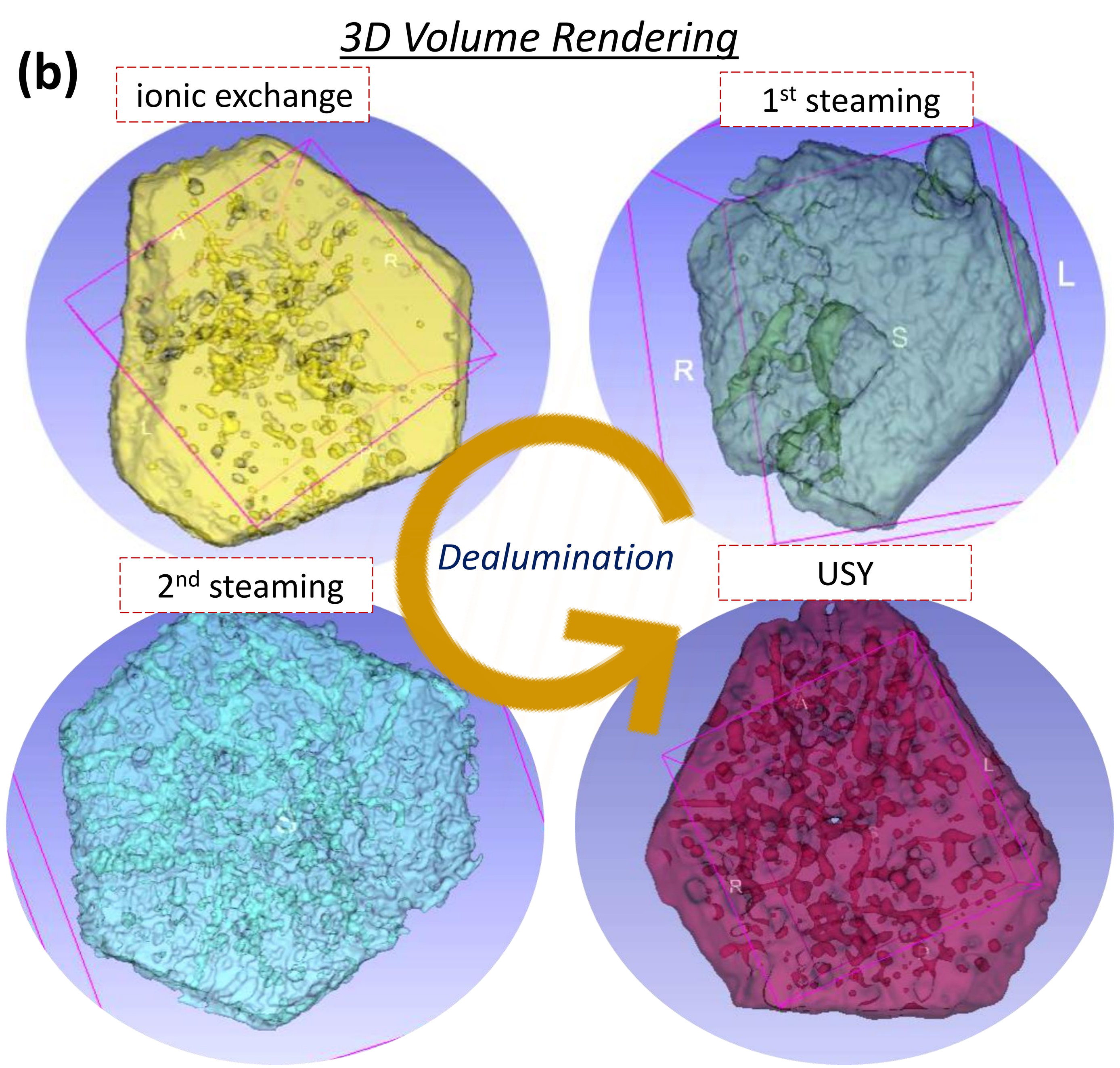
for a series of zeolites on a on-going dealumination process after performing: a low temperature steaming (1st steaming), a $NH_4$ exchange (ionic exchange), a high temperature steaming (2nd steaming) and acid leaching obtaining the Ultrastable Y form (USY)

## Methodology

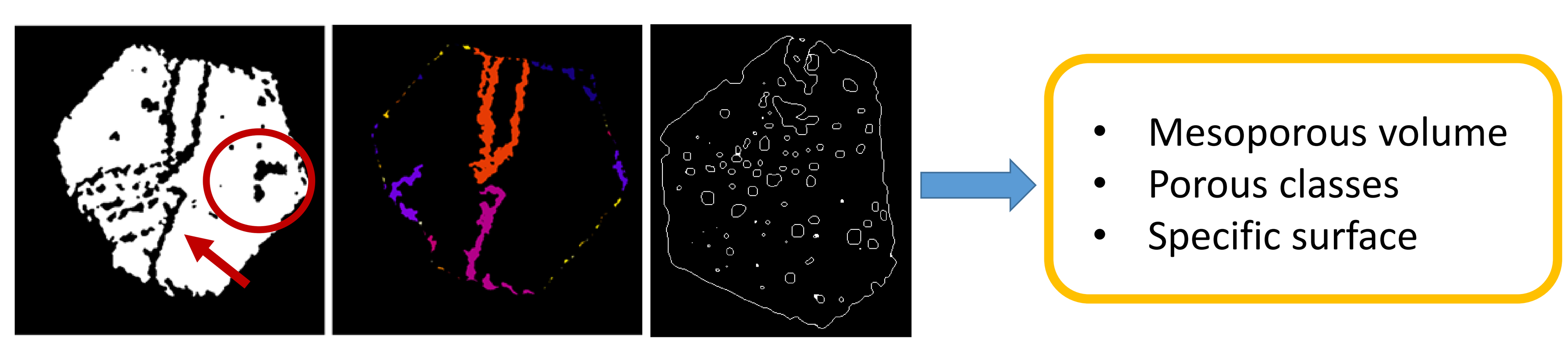
**(a)** *Electron Tomography*




**(b)** *3D Volume Rendering*




**(c)** *Image Processing*

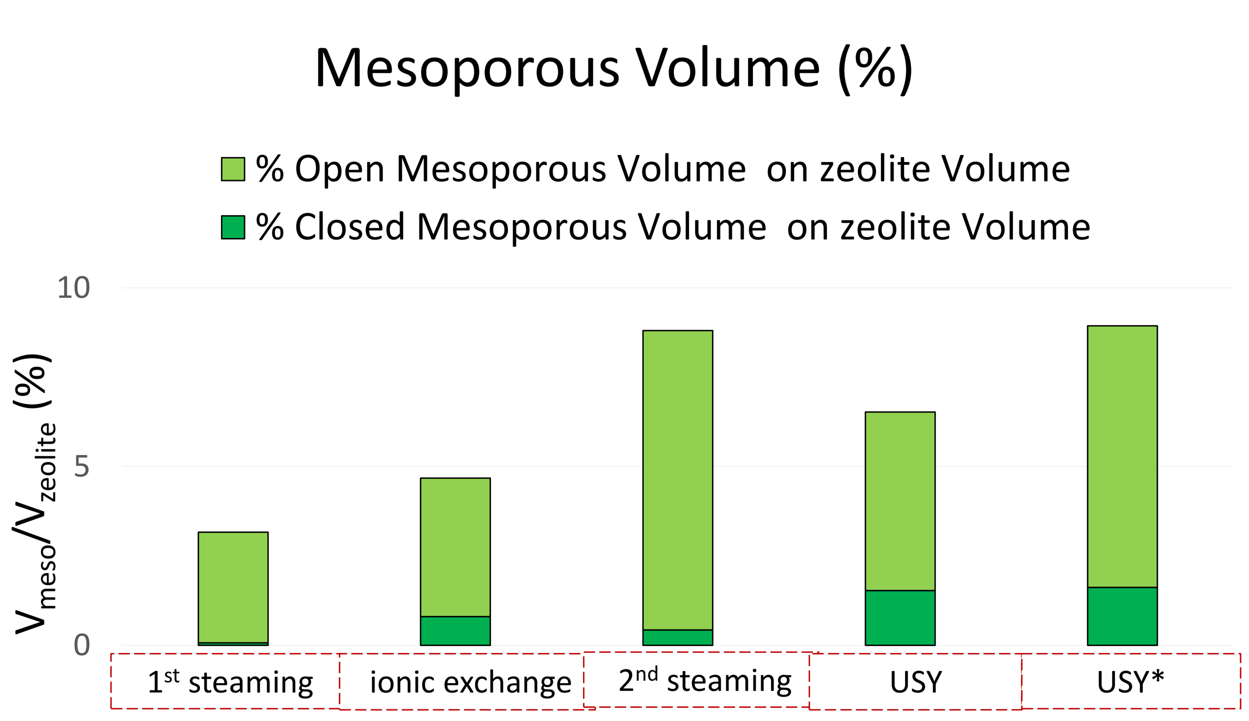


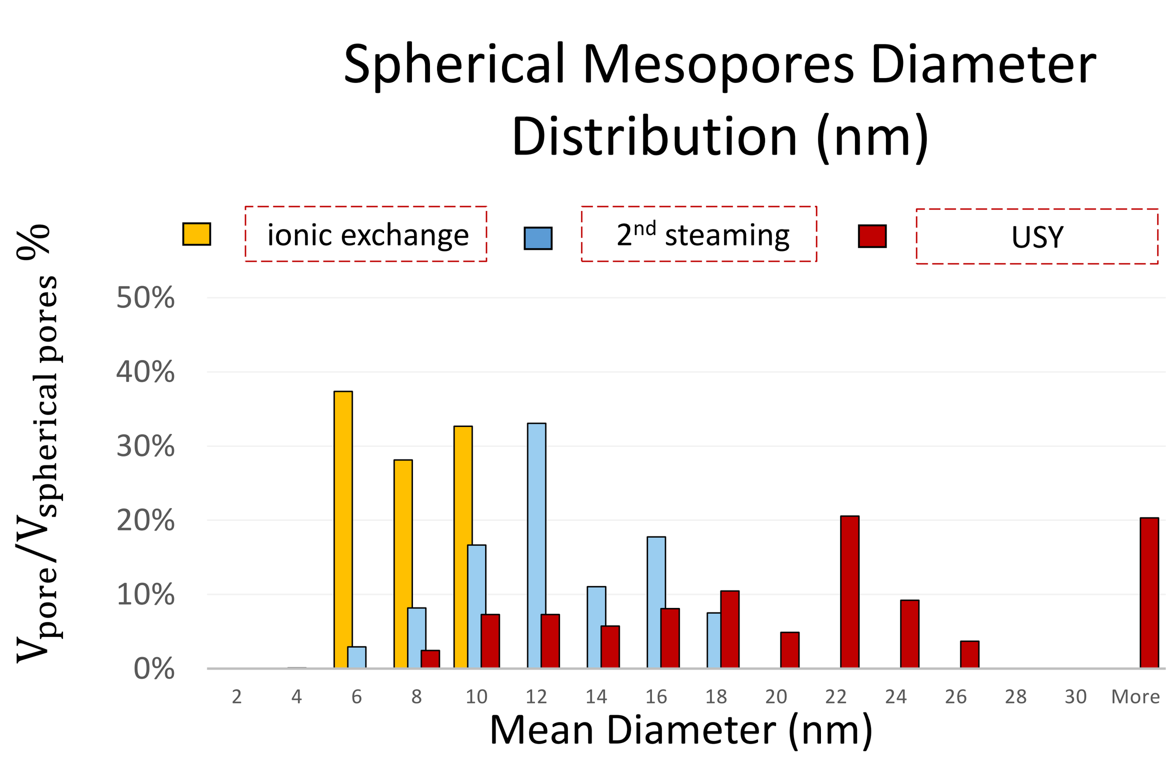

(a) From right to left, the protocol of the electron tomography: the acquisition in High Angle Annular Dark Field STEM mode of 2D projections of the zeolite at different tilt angles, the alignement of the stack of projections, the reconstructed volume through an iterative algorithm and the digital binarization of the same volume.

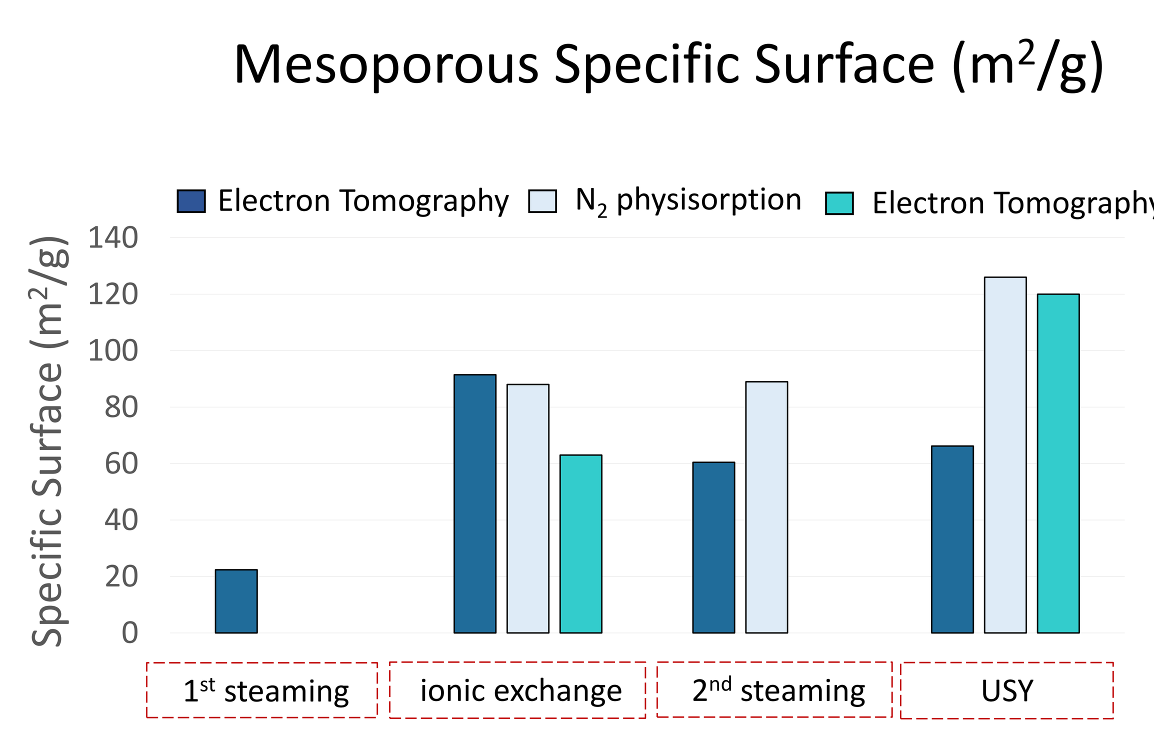
(b) 3D Volume rendering of isosurfaces on 3D Slicer for the 4 specimens at different Si/Al ratios and morphologies due to the level of dealumination.

(c) From left to right, image processing of binarized volumes by ImageJ (Fiji) and PlugIm! (IFPEN) in order to enumerate the voxels of the zeolite, the voxels of the mesopores and the surface voxels.

## Results and Discussion




During the dealumination an increasing trend in the mesoporous volume, specific surface and mesoporous diameter, in agreement with the literature and with the correlated $N_2$ – physisorption analysis

## Conclusions and Perspectives

Morphological descriptors of mesoporous zeolites were obtained through 3D TEM i.e. the Electron Tomography technique. A deep learning based approach is being developed for obtaining further information on the features of the zeolite. The simulations of physisorption graphs for the reconstructed tomographic volumes are under study in order to link the bulk and the local characterizations. An operando TEM approach is also performed to elucidate the phenomenology of the dealumination mechanism by reproducing the steaming reactive conditions inside the microscope.