#### PS Multi-Scale Shale Imaging Challenges: Examples from US and European Plays\*

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Search and Discovery Article #41928 (2016)\*\*
Posted October 31, 2016

\*Adapted from poster presentation given at AAPG 2016 Annual Convention and Exhibition, Calgary, Alberta, Canada, June 19-22, 2016

#### **Abstract**

Quantitative 2D and 3D imaging datasets are of great value to the petroleum industry as they can provide the basis for geological reservoir models, which aid calculation of in-place volumes and modelling of permeability flow. Because of the high resolution required to image small scale features of interest (nm), the areas and volumes of data sets are inevitably small when compared to the typical scale of geological variability. Here we document, using examples from US and European shale plays, pertinent issues to consider when imaging mudstones, and in particular the limits of resolution and the representative nature of 2D and 3D imaging data sets. We present data taken from multi-scale X-ray CT and SEM observations (through four orders of magnitude in resolution; from 10s microns to 10s nanometres). Imaging nanoscale porosity in mudstones requires the highest resolution techniques available in either 2D (standard SEM) or 3D (FIB-SEM). Connected pore networks within and between mudstone components are overwhelmingly small (nm-um length scale). The small size of pores measured attests to the low permeability of mudstones, but also highlights the undefined pore volumes beyond the limits of resolution (~5-10nm), which are confirmed by petrophysical measurements. At lower resolutions (um) and larger areas and volumes (mm<sup>3</sup>), x-ray nanotomography can be used to provide 3D volumes. Mesoscale features such as sedimentary textures can be observed allowing nanoscale datasets to be contextualised. However, discrimination of mudstone components is a challenge, with differentiation between organic matter, pores and clay minerals difficult or impossible due to similar attenuation characteristics of these phases. Another challenge comes from relating nanoscale and microscale datasets; ideally, numerous nanoscale datasets should be generated to statistically confirm that measurements are representative across scales. At the lowest resolution (10s um) and sample volume (cm<sup>3</sup>), x-ray microtomography can produce 3D datasets that approach the core scale. This creates an opportunity to bridge from imaging dataset to petrographic or core studies. However, the low resolution of these data means that only large features such as fractures and fossils are observable. We use our data to highlight the scales and methods of observation that best provide reservoir quality-specific information in shale plays, and to propose an initial upscaling approach for such data.

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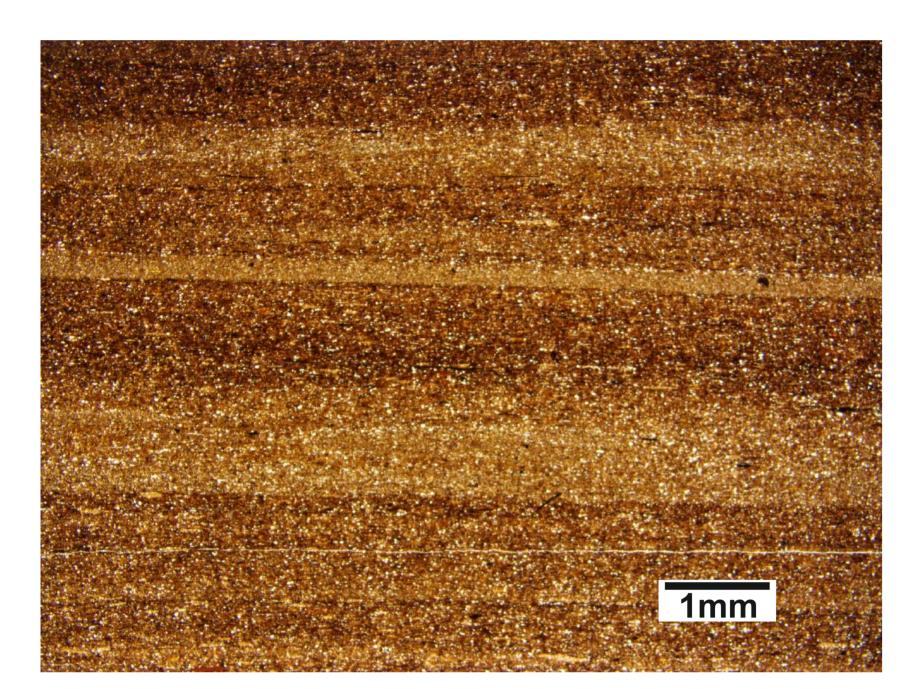
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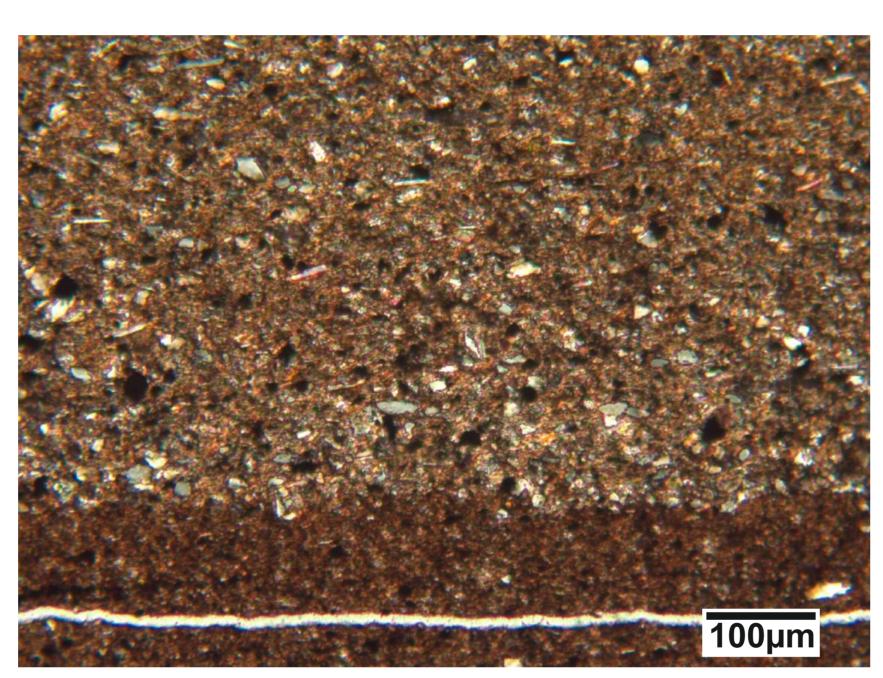
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#### Introduction

Quantitative 2D and 3D imaging datasets are of great value as they can provide the basis for geological reservoir models. Reservoir models can be used for calculations of in-place volumes and in modelling permeability flow. In shales, features of interest include pores, organic matter, minerals and fractures.

Quantitative data collection of this range of components within the same sample set often proves problematic because of the varying scale (10-100,000 nm) of the components imaged. Mudstones are also highly heterogeneous. Therefore, combined multi-scale imaging is essential to capture data on a full range of mudstone components.





Examples of lamina-scale variability in shales

Pores within mudstones are particularly small (nm), and quantitative data collection requires the use of high resolution imaging techniques. As a result, the areas and volumes of these data sets are on a similar scale (10-1,000 nm) to the heterogeneity inherent within shales. Care needs to be taken to ensure that imaging data is representative of the studied samples.

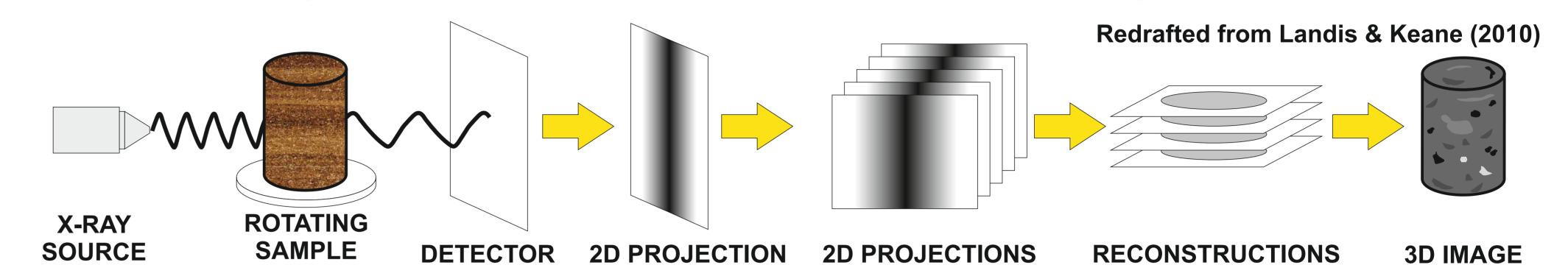
This work focusses on examples from the Mudstone and Shale Gas Reservoir (MSGR) research group at the University of Manchester to explore the following key research questions:

- 1) What are the limits of resolution at varying scales?
- 2) How representative are 2D and 3D datasets?
- 3) How can imaging datasets be applied?

## Imaging overview

### X-ray computed tomography (XCT)

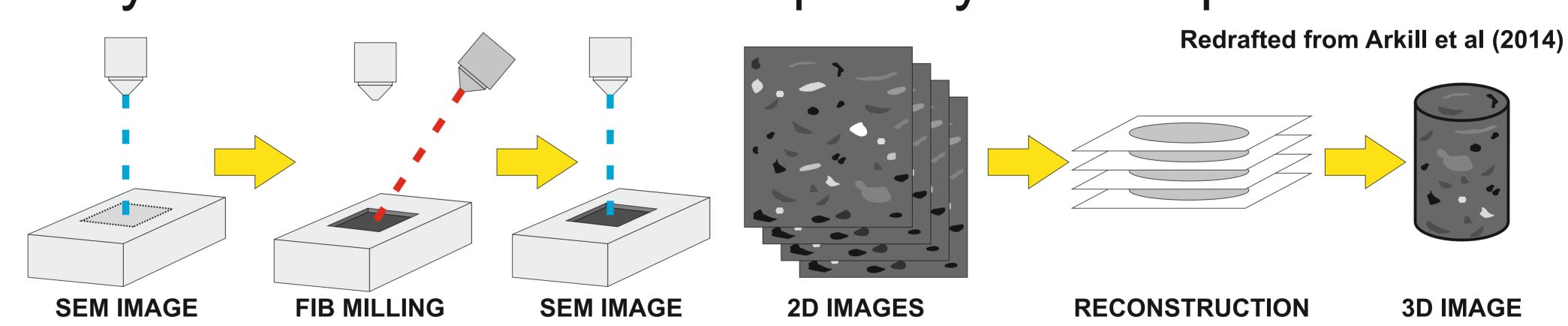
X-ray attenuation is controlled by the density of the mineral through which the x-ray is passing. A series of 2D attenuation images are collected by an x-ray detector as sample is rotated 360°. Using the series of images a 3D volume is reconstructed. Image analysis of the 3D volume allows components to be differentiated and quantified.



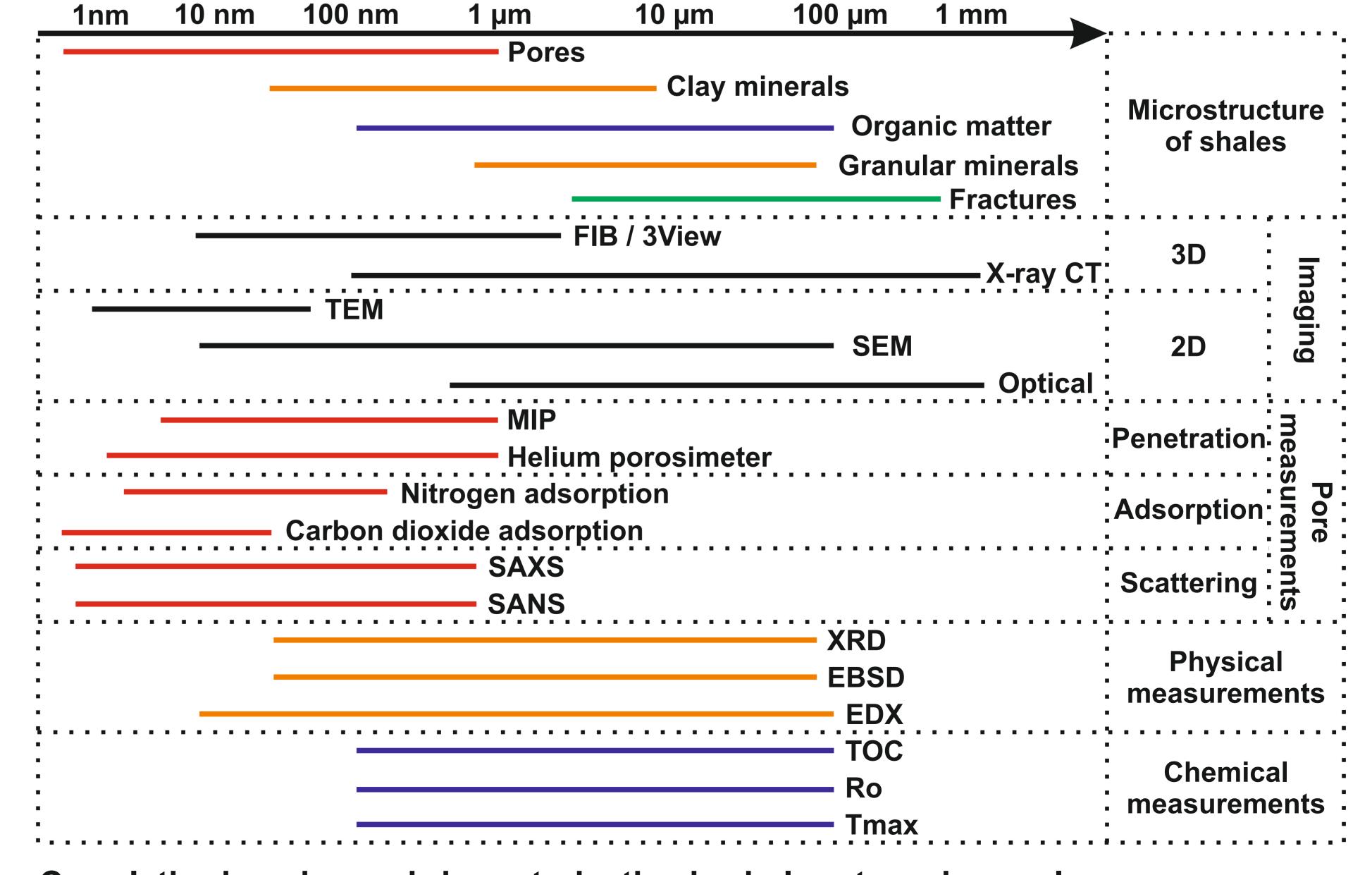
Schematic illustration of XCT technique

# Focused ion beam-scanning electron microscopy (FIB-SEM)

lon milling sputters away a thin layer of sample (5-20 nm), producing an extremely flat surface which can be imaged with an electron microscope. Repeated sputtering and imaging over the same area produces a series of 2D electron images. These 2D images can then be reconstructed to produce a 3D volume. From which image analysis can differentiate and quantify the components.



Schematic illustration of XCT technique & FIB-SEM techniques



Correlative imaging and characterisation in shales at varying scales.



# Multi-scale shale imaging challenges: Examples from US and European plays

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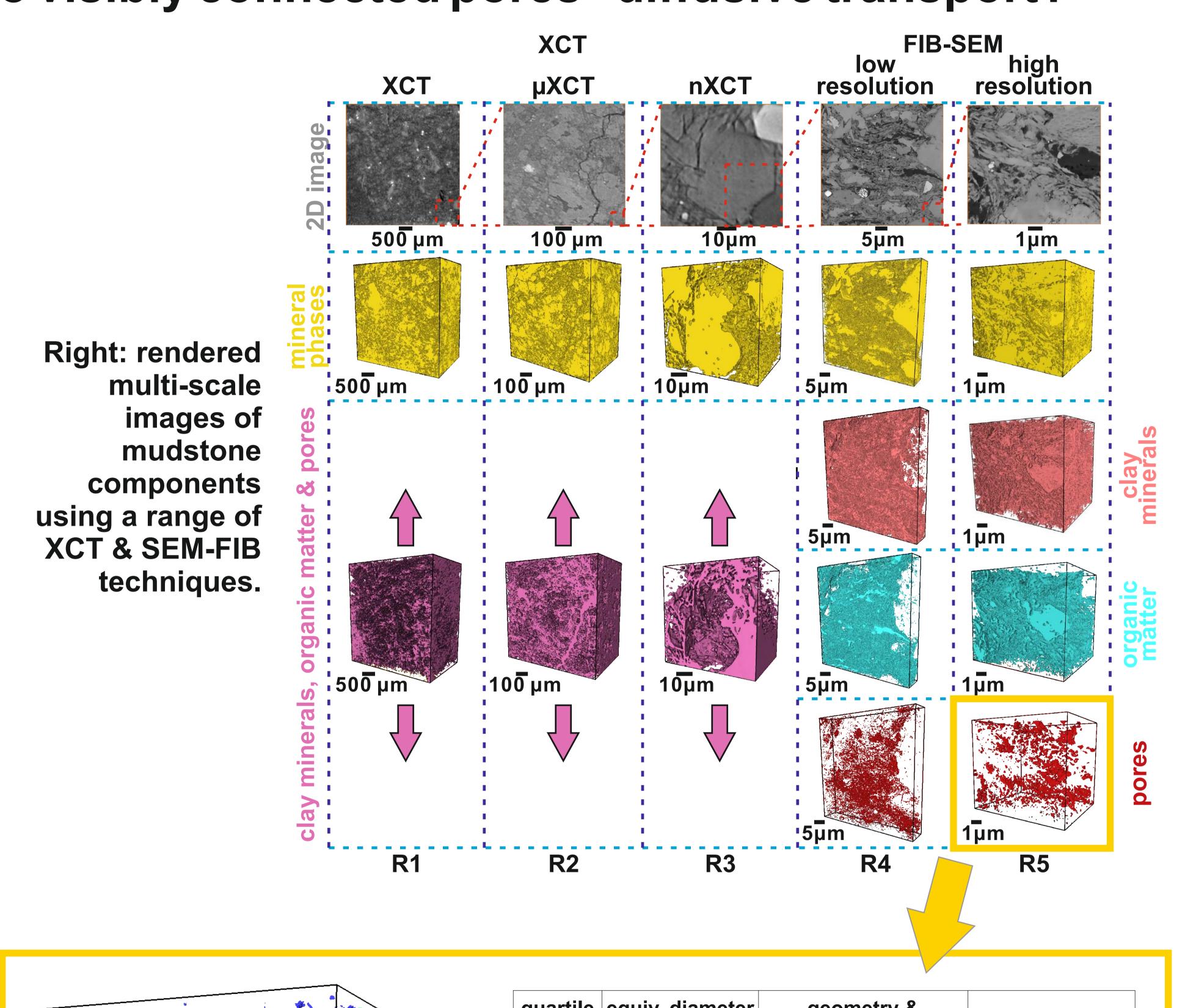
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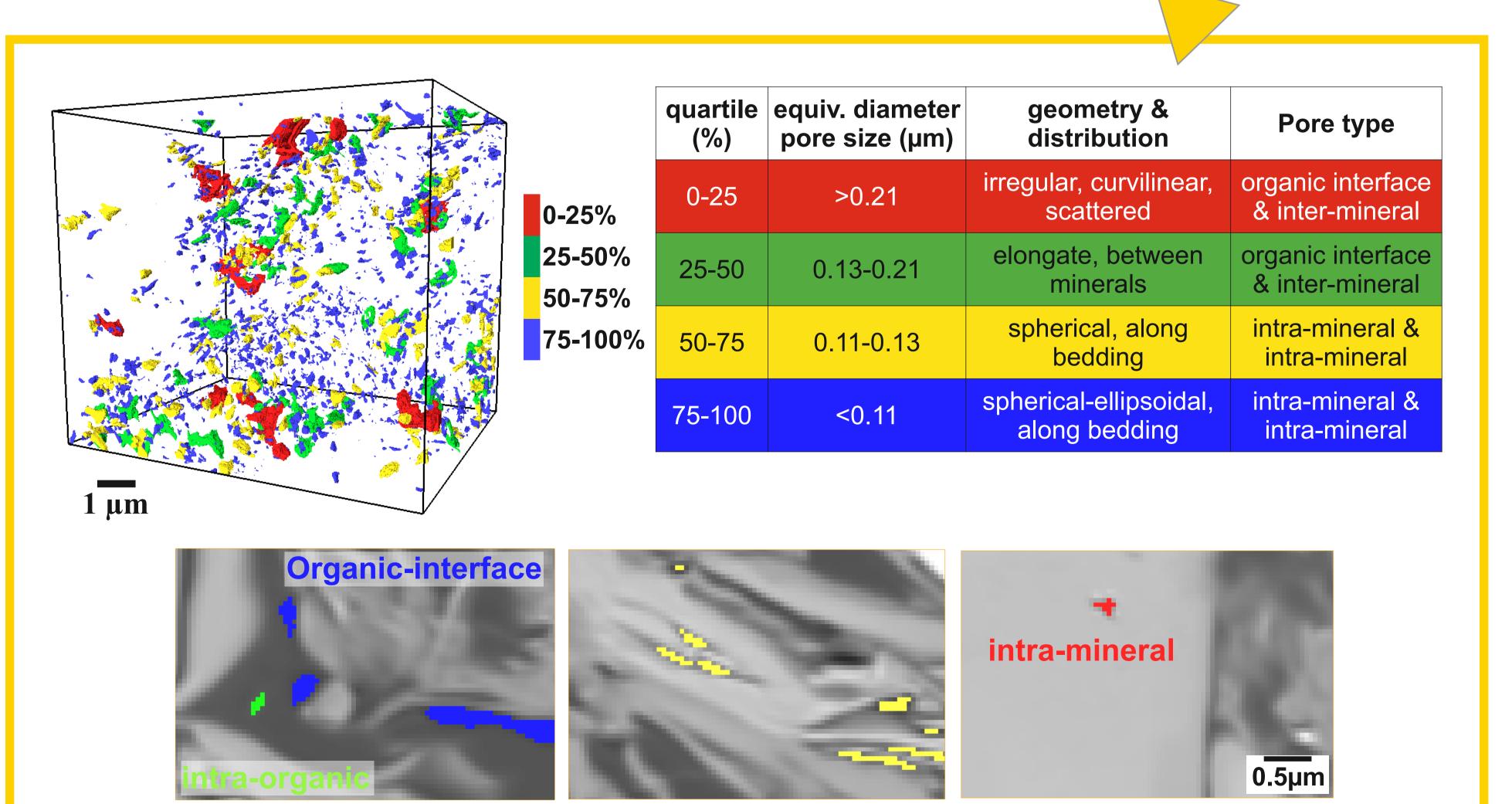




## 1) What are the limits of resolution at varying scales?

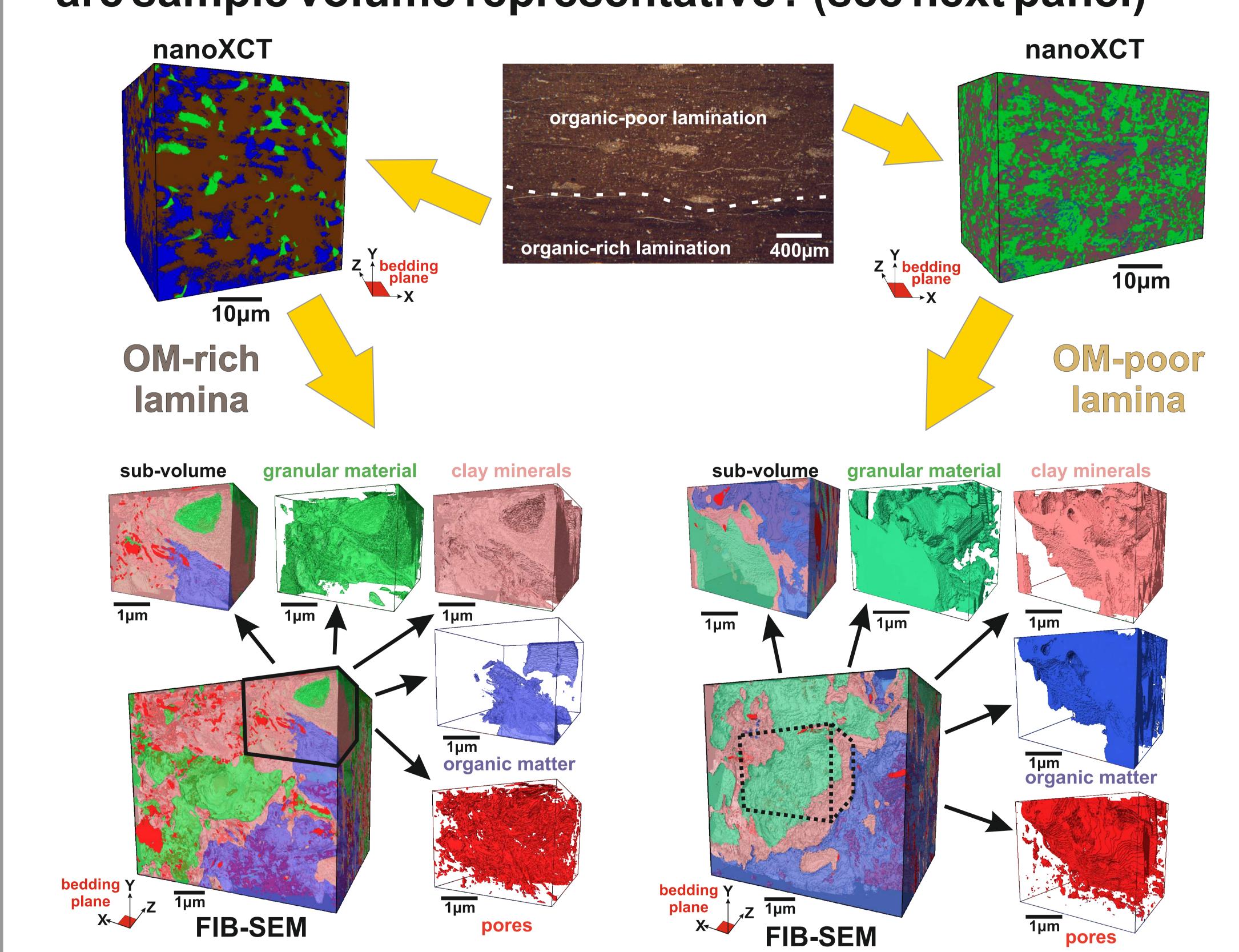
- Bowland Shale, UK
- Imaging techniques at a range of scales demonstrate which components which can be quantified
- Four pore types largest are inter-mineral and organic interface pores
- No visibly connected pores diffusive transport?



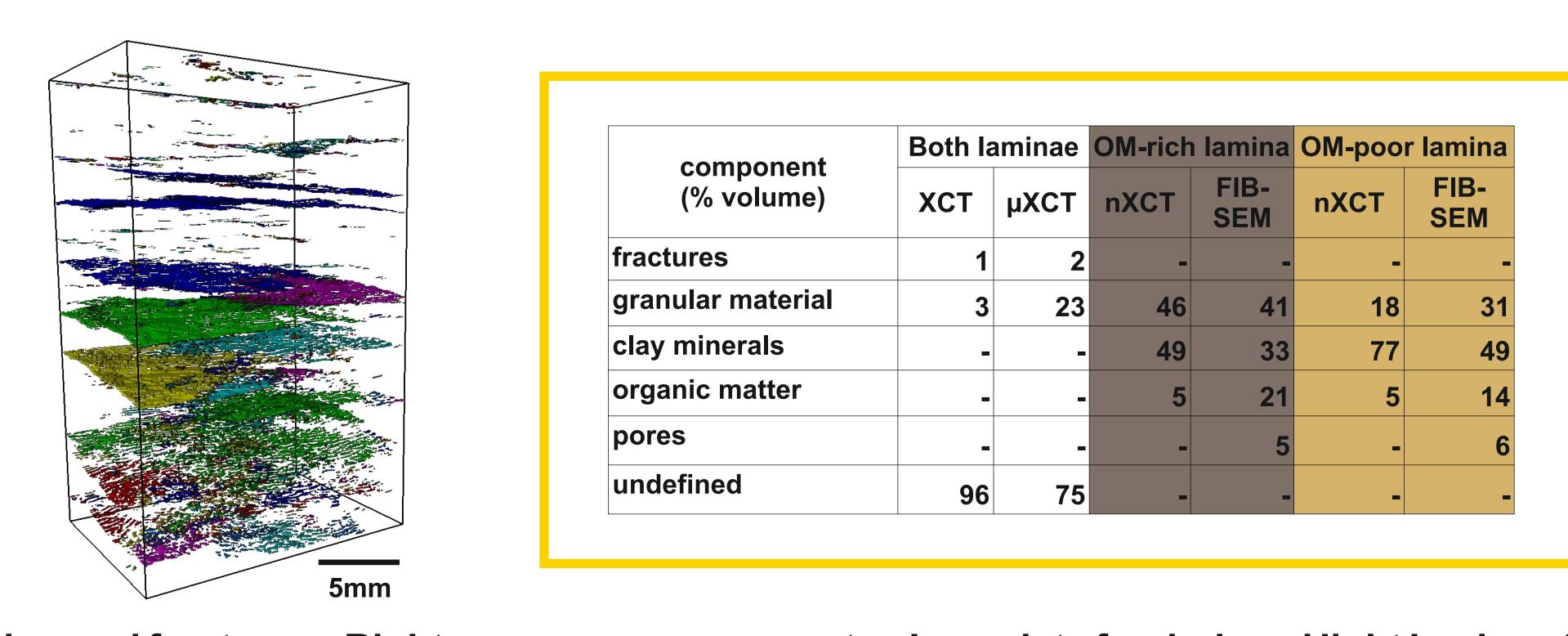


Top left: rendered image of FIB-SEM pores classified by quartile volume. Top right: summary pore data at high resolution. Bottom: different pore types

- Haynesville-Bossier Shale, USA
- Fractures best imaged at microXCT
- nanoXCT enables granular material, clay minerals and organic matter to be differentiated
- FIB-SEM best for all components except fractures. But are sample volume representative? (see next panel)



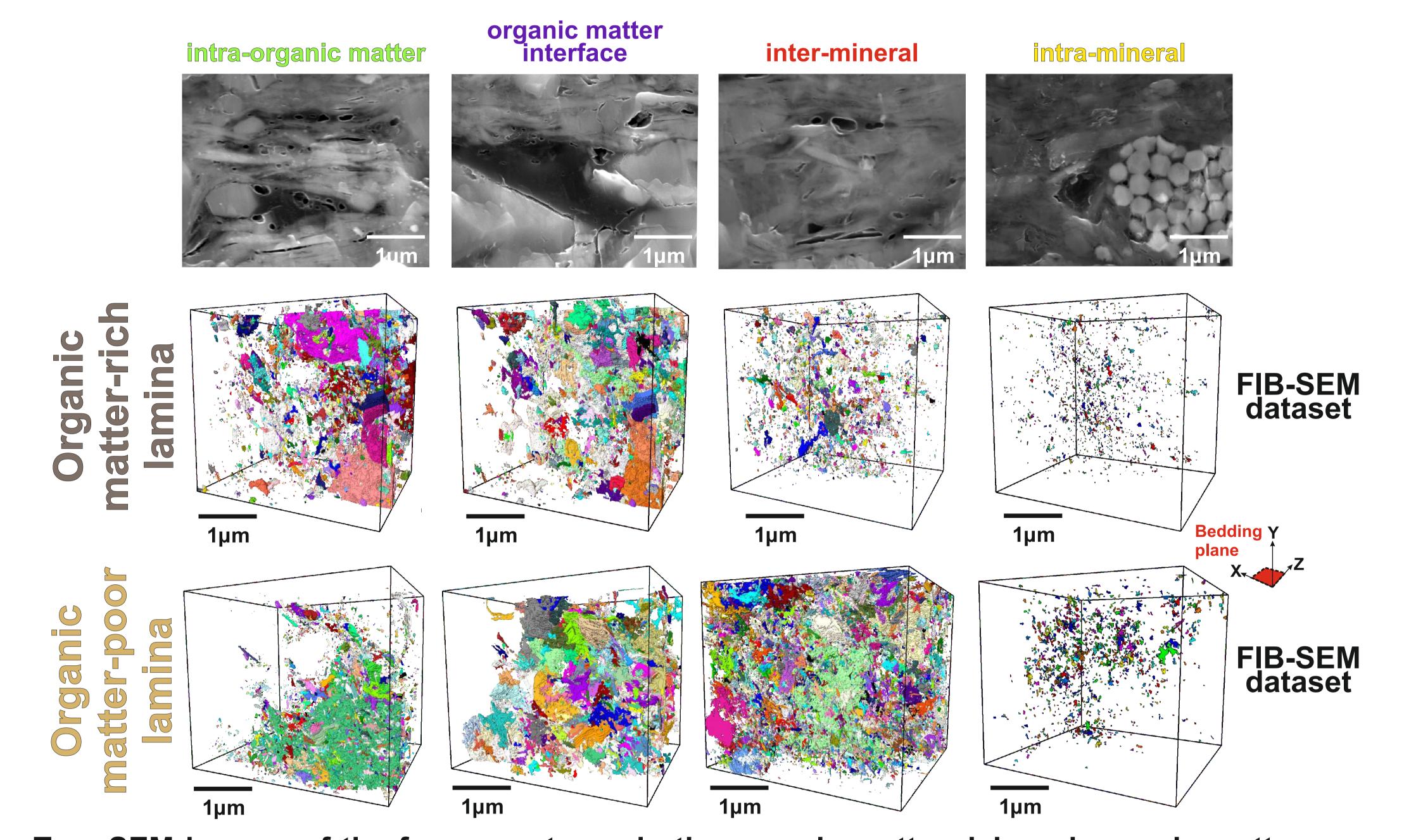
Top centre: thin section image of organic-matter poor and organic-rich laminae. Top left: nano XCT rendering of organic matter-rich lamina. Top right: nano XCT rendering of organic matter-poor lamina. Bottom left: rendering of FIB-SEM volume from organic-rich lamina. Bottom right: rendering of FIB-SEM volume from organic-poor lamina.



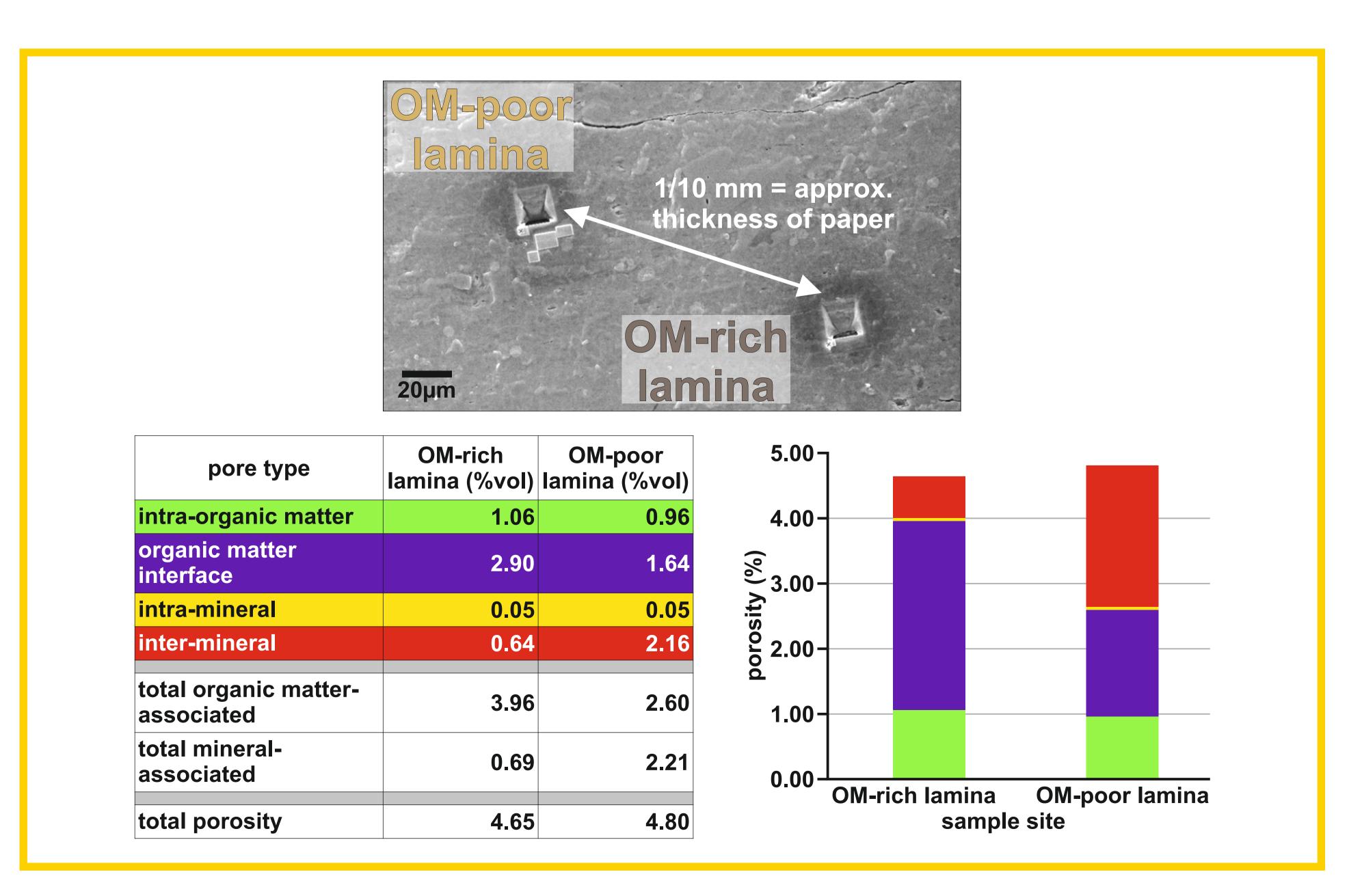
Left: imaged fractures. Right: summary component volume data for dark and light laminae from various image techniques.

## 2) How representative are 2D and 3D datasets?

- Haynesville-Bossier Shale, USA
- Two imaging sites ~100 um apart
- Distinct difference in the volumes of inter-mineral and organic matter interface porosities

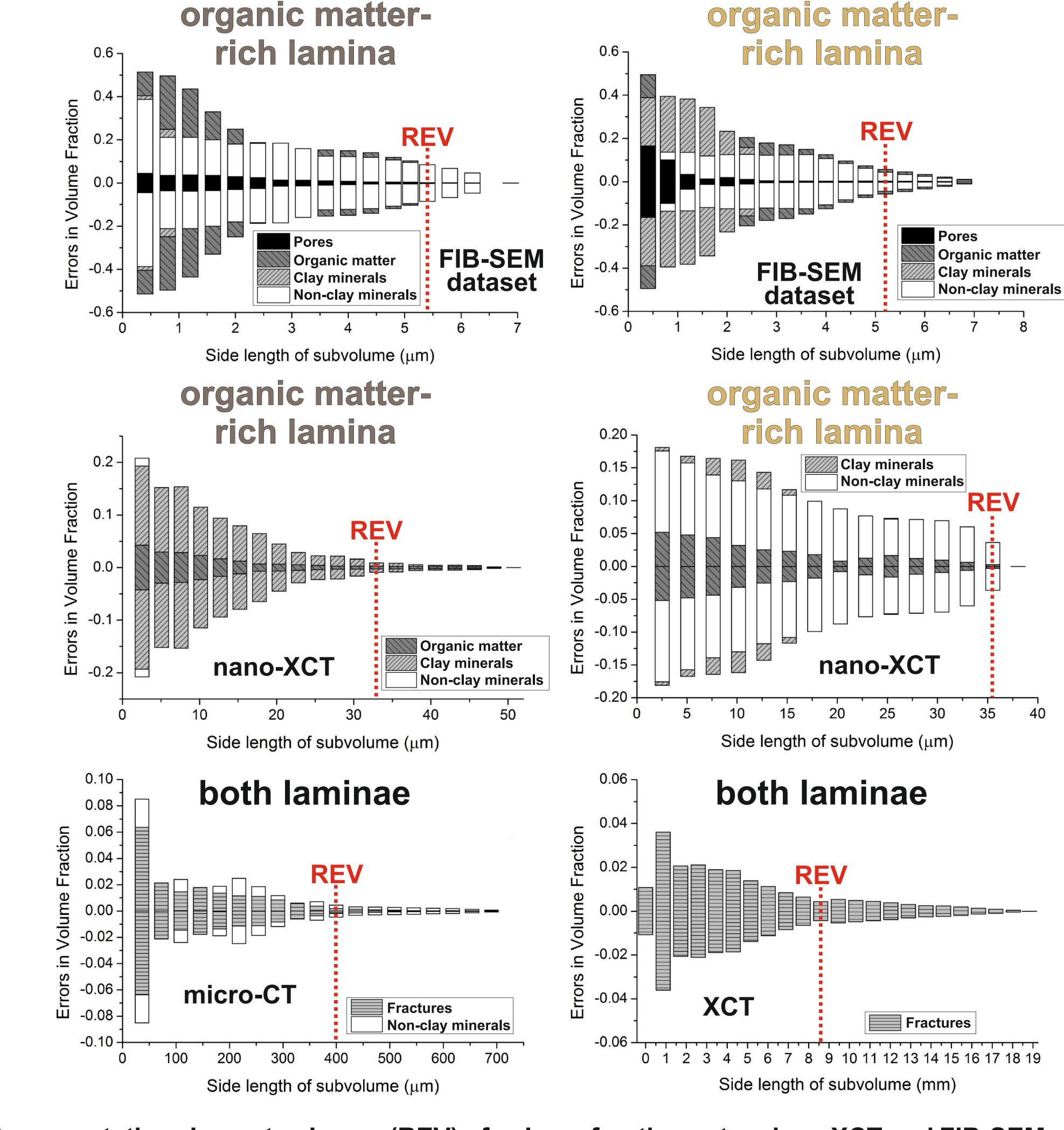


Top: SEM images of the four pore types in the organic matter-rich and organic matter-poor laminae. Bottom: volume renderings of pore volumes (%) for each pore type.



Top: SE image of two FIB-SEM image sites. Left: summary table of percentage porosity for each lamina and pore type. Right: bar chart of percent porosity for each in each lamina and pore type.

- Haynesville-Bossier Shale, USA
- Representative Elementary Volume (REV)- minimum volume large enough to capture representative heterogeneity
- REV calculated by measuring the volume fraction of each component at incrementally decreasing volume intervals
- Volume of each component at each interval is compared with the volume fraction of the same component in the original total sample volume
- Errors are calculated at each step
- Only where errors are <0.1 are volumes considered representative</li>

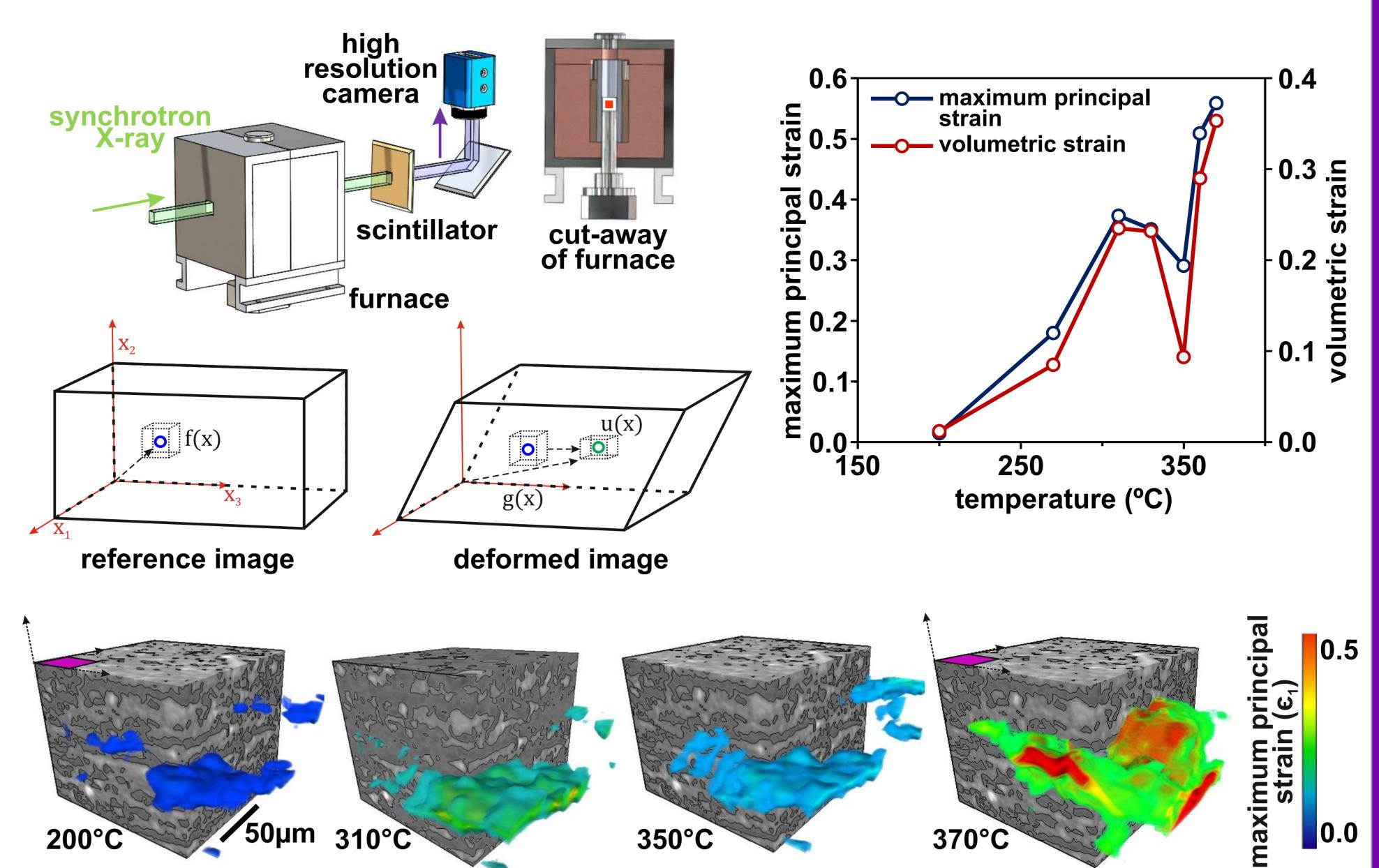


Representative element volumes (REV) of volume fractions at various XCT and FIB-SEM scales for differing shale components

## 3) How can imaging datasets be applied?

#### Fracture imaging

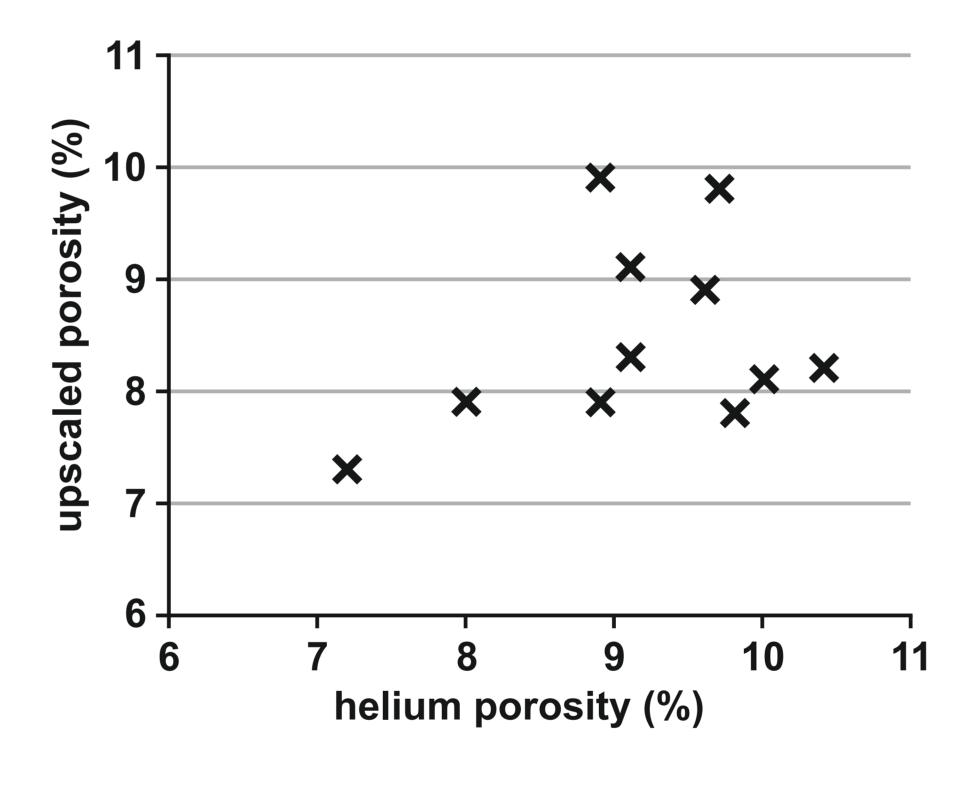
- As well as composition and texture, fracture development can be imaged in 3D using XCT
- Immature Kimmeridge Shale incrementally heated producing fractures
- Digital volume correlation (DVC) measures displacements in 3D
- Opening mode displacements perpendicular to bedding drive fracture nucleation
- Fracture propagation results from the connection of smaller fractures
- Fractures preferentially form in laminated organic matter between mineral clusters



Top left: heating experiment set-up & schematic of the DVC method Top right: maximum principal strain and volumetric strain vs. temperature. Bottom: progressive maximum principal strain with increasing temperature within rendered XCT volume (warmer colours equal greater maximum principal strain)

## <u>Upscaling</u>

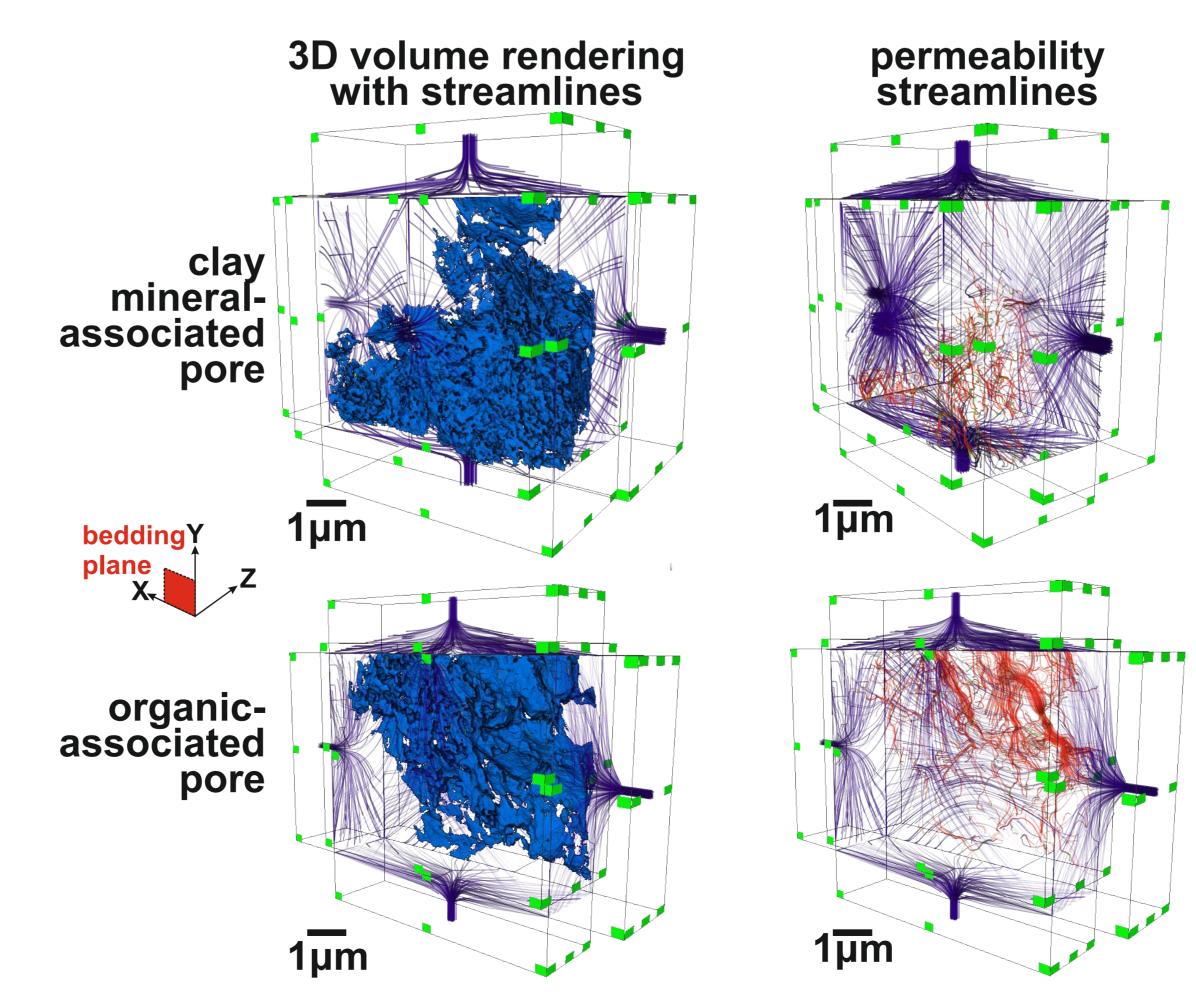
- Successive upscaling with each technique
- In step 1 (high to low resolution FIB-SEM), volume coefficients are calculated for each pore type (pore volume x associated component volume)
- In the successive upscaling steps, components are imaged and quantified at accompanying scales
- For example, in step 2 (low resolution FIB-SEM to nXCT), the pore volume coefficients in step 1 are multiplied by the volume of components imaged at the nXCT scale
- This produces a total porosity value for the larger sample size, but accounts for the variability imaged at each scale



Scatter plot of measured core plug porosity vs. upscaled porosity

## Permeability simulation

- Imaged pores (>20nm) unconnected in Haynesville-Bossier
- N<sub>2</sub> data indicates the presence of pores <20nm which must locally provide connectedness and permeability
- Permeability simulations performed on large imaged pores within organic matter and clay minerals
- Permeability in imaged organic matter pore found to be x36 that in the imaged clay mineral pore



Permeability simulations utilising 3D imaging data

## Summary

- 1) Quantitative imaging of mudstone components requires a multi-scale approach due to the small size and variability of components
- 2) The volumes of different pore types varies significantly on a small scale (100µm). REV analysis ensures that the data are representative
- 3) Imaging applications include fracture development, upscaling and permeability modelling

## Acknowledgements

MSGR project sponsors:



Thanks also to Loic Courtois & Anne-Laure Fauchille (RCaH, University of Manchester), Ernest Rutter, Julian Mecklenburgh, Mike Chandler, & Rochelle Taylor (SEAES, University of Manchester).

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