A CLOSER LOOK AT SHALE: REPRESENTATIVE ELEMENTARY VOLUME ANALYSIS WITH LABORATORY 3D X-RAY COMPUTED MICROTOMOGRAPHY AND NANOTOMOGRAPHY

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ABSTRACT

Though naturally occurring in many regions of the world, shale rock microstructure continues to be much of a mystery. Pore sizes may be very small, typically low 100s of nanometers and even below 10s of nanometers. It is thus very important to determine the volume size that must be examined to understand the oil reserves in a macroscopic shale rock formation, as the small features require a very high resolution imaging system, which usually come with limited field of view. This makes precise quantification of the microstructure a daunting challenge, especially when the analysis needs to be performed in 3D to capture the tortuous paths taken by the pores.

The introduction of ultra-high resolution imaging systems is now shedding light on the problem, with the commercialization of precise laboratory x-ray imaging tools. Here, a novel suite of x-ray computed tomography systems is shown to provide unique insight into shale microstructure. Large volumes are measured with as high as sub-1 μm resolution using laboratory-based x-ray computed microtomography (VersaXRM) to localize regions-of-interest (ROIs) for further higher resolution analysis. A ROI of cubic volume with ~65 μm on each side is isolated for precise analysis with a novel laboratory-based x-ray computed nanotomography system (UltraXRM) capable of 50 nm resolution for quantification of porosity within the shale sample.

Using the multi-length scale resolution imaging systems described here, a representative elementary volume (REV) quantification has been performed, which identifies ~30 μm as the minimum volume that must be considered in order to quantify pores in shale down to 150 nm linear dimensions. Using a 3D field of view capable of sampling ~4 of these REVs, a precise microstructure analysis is carried out, within which further calculations of pore tortuosity and connectivity are demonstrated. The non-destructive nature of x-ray imaging further opens the door to innovative experimentation, such as time-evolution and studies of microstructure response to varying environmental parameters, such as temperature cycling or surfactant treatment.

INTRODUCTION

How far from the pavement does one need to look in order to calculate the number of cars currently in use world-wide? Zooming out just a few meters, one might see a single mini-van – should we then assume that the world is packed with highways and mini-vans? By zooming out a few kilometers, one might capture long stretches of highway filled with rush-hour traffic, including the vans and large trucks – but do we assume then that there are no motorcycles? If a spot for observation is chosen from space at random, what if chance put the measurement in the middle of the Pacific Ocean?

All studies of microstructure, particularly those of porosity, must deal with the question of representative elementary volume (REV). With larger fields of view come naturally coarser resolutions and a careful balance must be struck to ensure that the majority of features are captured with enough measurement statistics for high confidence in the results. There may be an REV at each length scale and for each volume size, making REV characterization a critical part of experimental design before embarking on extensive microstructure studies.

Digital core analysis (DCA) represents a novel advancement for optimizing drilling site feasibility and oil extraction efficiency measurements for geological core analysis [3]. In this technique, a sample is virtually segmentation into its various material constituents using an imaging system tuned to the appropriate parameters for the material under investigation. The pore phase may be subsequently isolated and then run through an advanced modeling routine to extract fluid flow parameters, such as permeability [3, 8], as well as pure microstructure parameters, such as porosity and pore shape analyses [7].

Shale is an abundant resource across many different world regions. While it is thought to contain significant amounts of natural gas, which could be converted into fossil fuels, there remains a limited understanding of how to most efficiently extract the organic constituents. This is due, in part, to the problems in characterizing shale microstructure using physical methods, as the small pores make traditional direct fluid intrusion measurements very difficult. DCA, on the other hand, has been recently demonstrated to provide a new, efficient characterization approach for many types of geological analysis and is now being extended into shale [10]. Measurements of REV are thus of high importance at this time, as the baseline must be set before routine characterization may be implemented.

Non-destructive imaging approaches are growing in popularity for DCA due, in part, to their ability to analyze one sample multiple times at varying length scales. This enables the precise localization of pores of many different sizes and ensures that all significant contributors to fluid flow and organic composition are accounted for in modeling. The most commonly used technique is x-ray computed tomography (x-ray CT) [3, 7, 8, 10], which allows repeated imaging of one sample [6] and returns results in a relatively short period of time. This approach has gained adoption for many different types of reservoir rocks, such as oil sands and carbonates [3], and has recently gained traction for shale, due to resolution improvements in the instrumentation.

METHOD

Recent advancements in the field of x-ray optics have pushed the resolution of new x-ray CT systems to unprecedented levels. By pairing a high-efficiency x-ray condenser lens with a high-resolution x-ray imaging objective, these x-ray microscopes are now capable of producing resolution in the 10s of nanometers [1-2] while maintaining penetration of up to 100 μm for both carbonates and silicates. This technology has been in development for many years at synchrotron facilities and government laboratories, but has recently been extended to stand-alone laboratory systems [9]. Figure 1 shows a sketch of one such laboratory x-ray microscope, the Xradia UltraXRM-L200, which is the only commercially-available system of its kind known to the authors at the time of this writing and the system used to obtain the results that follow.

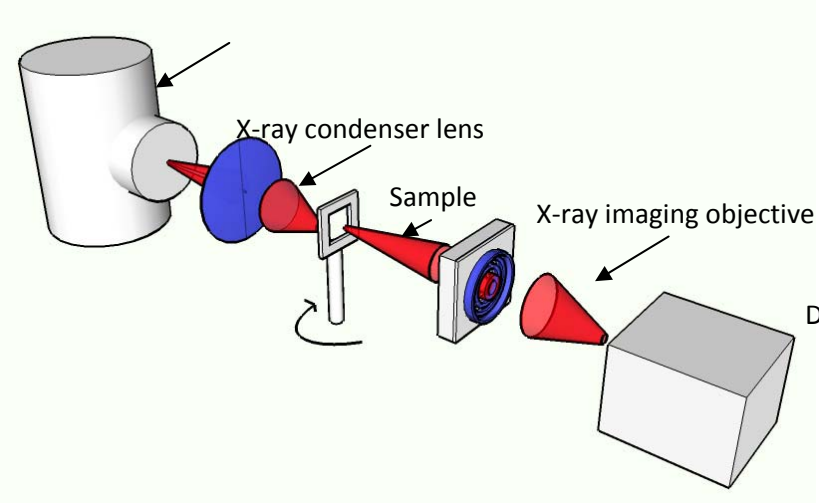

Figure 1. Schematic diagram of a laboratory x-ray microscope.

Imaging Procedure

Imaging with this x-ray nanotomography (nano-CT) system typically represents the culmination of a multi-length scale characterization process. Samples are first imaged with an x-ray microtomography (micro-CT) system in order to characterize the porosity from the tens to single micron size scale. These images are then analyzed for regions of interest (ROIs) where porosity may be inferred, but not measured, and then subsequently investigated with increasingly higher resolution for precise measurements of porosity and fluid flow paths. The Xradia VersaXRM is one such micro-CT system, which provides a novel geometry enabling virtual sub-sampling, a “zoom-in” approach that reduces the need for sample preparation in order to achieve high-resolution results, and is thus suitable for measurements up to entire plugs. This system is demonstrated to pair with the UltraXRM, wherein small samples are physically extracted from the plug and imaged with nano-scale resolution, as shown in Figure 2. Due to the small sample sizes needed for imaging, the nano-CT technique is best suited for small plug sections, as demonstrated here, rather than drilling cuttings; however, future work may expand the scope of this analysis to include a wider variety of core sample types.

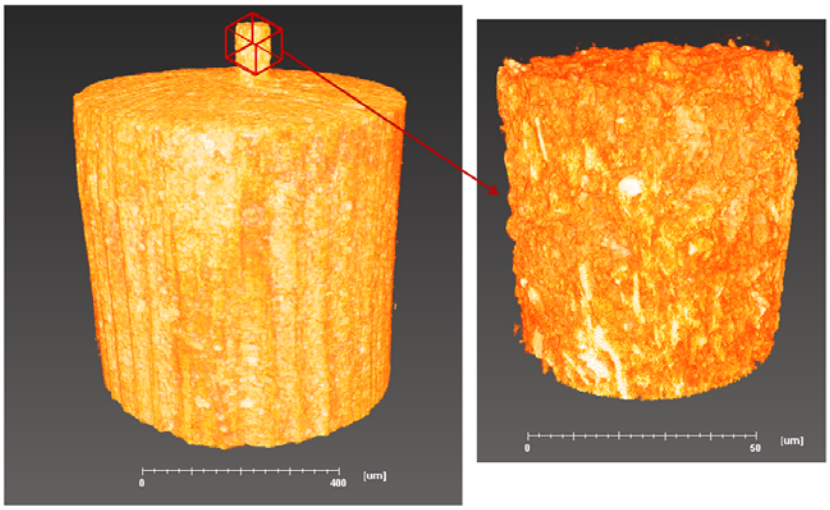

Figure 2. Multi-length scale concept for DCA, using shale as the example material. The high-resolution micro-CT volume (left) is first used to locate micron-scale pores within a plug. A representative region is then selected and physically extracted for nano-CT analysis (right)

Analysis Procedure

After imaging, the virtual slices are digitally processed into volumes that may be used for modeling and advanced analysis. A watershed segmentation algorithm is used to virtually segment the image into its various material constituents [5], such as silicate, pyrite/inclusion, pore, etc. This reduces the volume from tens of thousands of grey scale units to a 3D matrix of 0's, 1's, 2's, and 3's, corresponding to exterior (air), pore, silicate matrix, and high-Z inclusions, respectively. Figure 3 shows an example of the results of segmentation. While the watershed segmentation has been found to have some dependence on initial seeds, which may be affected by imaging noise [4], the images produced by the nano-CT system are very clear, thus it is the preferred segmentation method of the authors at the time of this writing.

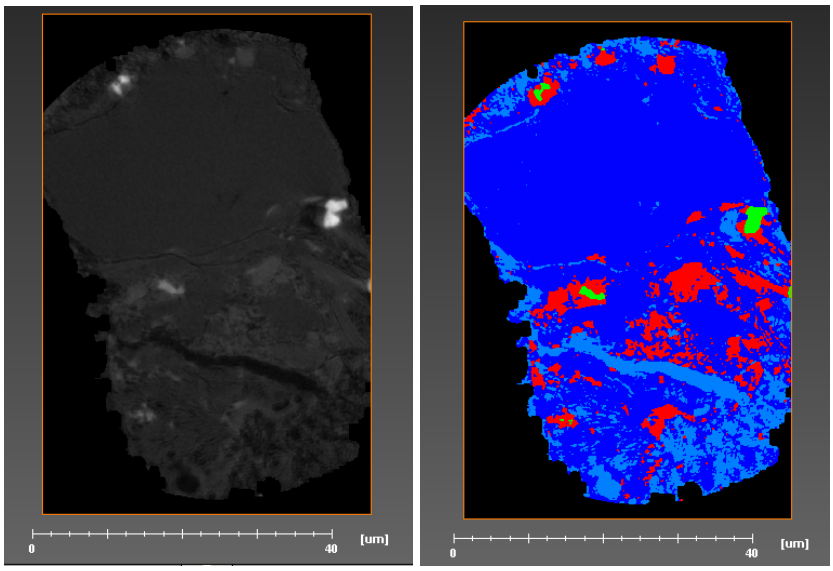

Figure 3. Results of segmentation of the nano-CT results from a sample of shale. The light blue represents the pore phase, dark blue the silicate matrix, red the mid-Z inclusions, and green the high-Z inclusions. The exterior remains black.

RESULTS

The nano-CT segmentation results were subsequently used to characterize the representative elementary volume needed for resolution on the ~100 nm length scale. In order to do this, the sample was examined for porosity, which was calculated by dividing the integrated pore volume by the total sampled volume (excluding the exterior voxels). The volume was then digitally sub-sampled into cubes of smaller and smaller dimensions, which were systematically tiled and used for porosity measurements in 3D. The entire volume was considered, followed by dimensional downsampling factors of $2^{1/2}$, $2^{1/3}$, 4, and 8. Figure 4 shows a pictorial example of this procedure.

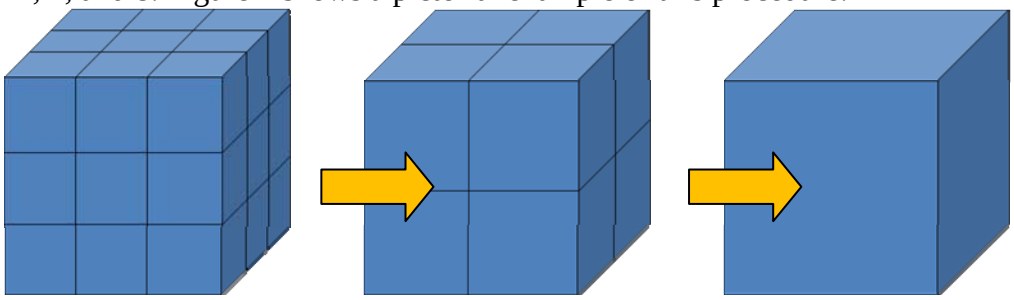

Figure 4. The volume was sub-sampled into cubes of different sizes, which were systematically tiled through the segmented volume for localized porosity analysis.

Within each cube, the total volume of voxels corresponding to the pore phase was calculated, then divided by the total volume of the sub-sampled ROI (i.e., the cube volume excluding the exterior voxels). The result was called “porosity” and is shown as a function of ROI volume in Figure 5.

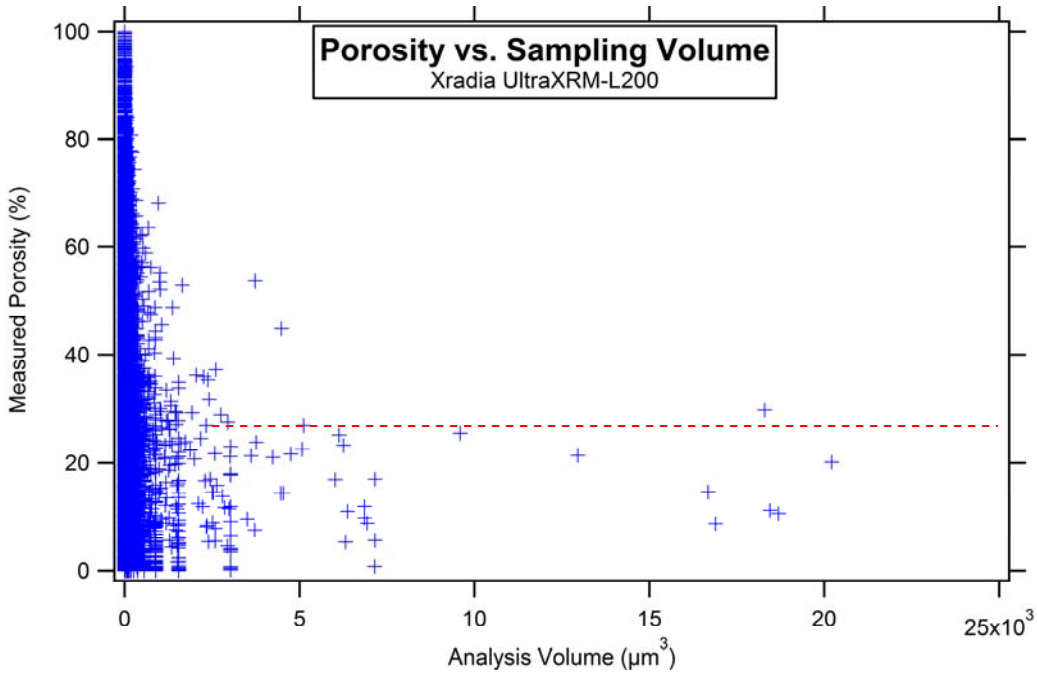

Figure 5. Porosity measurements as a function of analysis volume. REV is considered to be the point at which the porosity converges to the full volume porosity, which is indicated by the dashed red line.

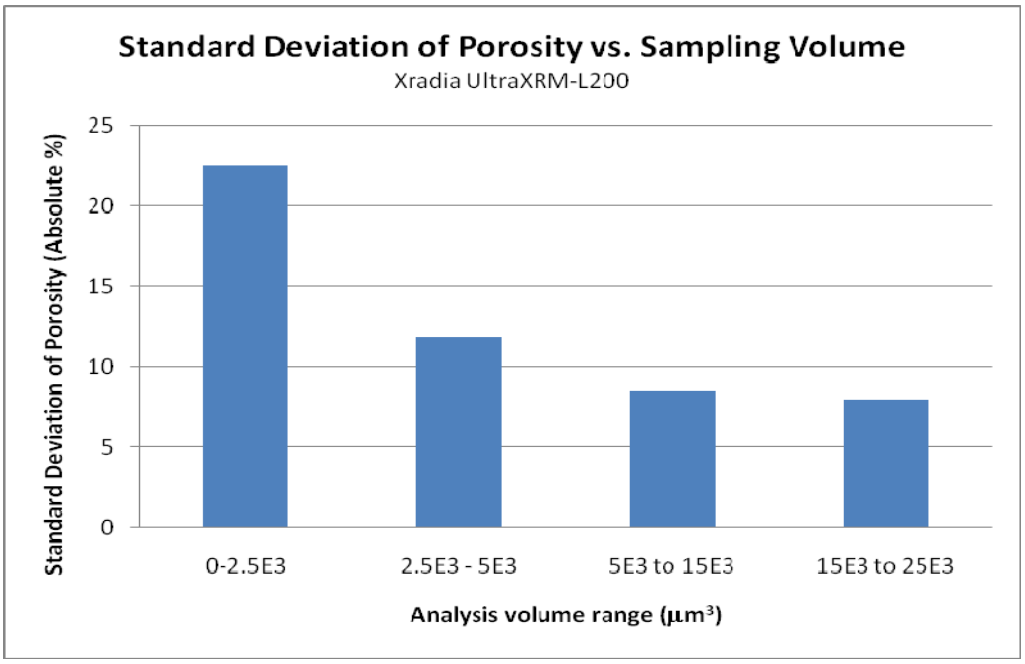

Figure 6: Standard deviation of porosity measurements as a function of analysis volume ranges. Reasonable convergence occurs between 5000-15000 µm^3, but the highest precision occurs between 15000-25000 µm^3.

DISCUSSION

With resolution held fixed, results should be expected to increase in precision as the analysis volume also increases, due to the increase in measurement statistics. Thus, the analysis of these results should be taken as a search for the volume at which the porosity is in close, regular agreement with the porosity of the full volume. The full volume porosity across the nano-CT's maximum volume visualization with 150 nm resolution was measured to be 17.2%, which was segmented using the watershed method previously described. This number was validated by crushing a similar sample from another section of the core and measuring the volume of the crushed material, which also gave a porosity value of ~17%. The results here were meant to demonstrate the need for higher statistics (i.e., measurements throughout a larger volume) in order to match the results from analysis of the full volume, validating imaging results to themselves, and correlating to indirect porosity measurements. Thus, the representative elementary volume was considered as the sub-volume at which the porosity results matched all of the larger volumes and results from the other method, for which the full imaging volume was measured to be the extreme case. Convergence around that point occurred at ~17000 µm^3, corresponding to a cube of 25.7 µm on each side, which is graphically depicted in Figure 6. This measurement thus suggested that the representative elementary volume for this shale sample was 17000 µm^3, indicating the need for both high resolution (to see the pores down to the ~100 nm size scale) and a reasonably large field of view.

The results of this study should not be surprising, but instead validate the need for an imaging system that can provide precise analysis across a larger volume. The application of x-ray nano-CT is thus very well suited for imaging shale at this length scale due to its unique capability to provide sufficient resolution and enough field of view to sample several REVs in a single scan. The non-destructive nature of x-rays further allow the repeated imaging of one ROI, enabling microstructure characterization after changing environmental conditions, such as pressure & hydration. The details of these repeated imaging studies have been approached elsewhere [6] and their importance is worth noting as a significant strength of CT.

CONCLUSION

Here, we have demonstrated the application of a non-destructive 3D x-ray imaging system for precise analysis of shale. The multi-length scale imaging capabilities are of critical importance to the growing field of digital core analysis, as exemplified by the REV study demonstrated on this nano-scale x-ray microscope. The large field of view is found to be sufficient to sample several REVs of this shale sample at once, further supporting the high measurement statistics inherent in this technique.

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