Micro-CT/micro-XRF study of reservoir rocks: examination of technique applicability for core analysis

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Aims
One of the challenges of geological sample study is obtaining volumetric mineralogical properties of the rocks. More often, elemental (mineralogical) content is measured on the surface of a rock specimen and 2D properties are being propagated through the whole volume. In present work we investigate applicability of novel micro-CT/micro-XRF SkyScan 2140 system for obtaining information on spatial distribution of chemical elements in a rock.

Method
The micro-CT/micro-XRF system developed by SkyScan allows adding 3D chemical analysis to non-destructive micro-computed tomography (micro-CT) imaging [1]. It combines in one instrument a micro-CT scanner, which provides high-resolution morphological information and correction maps for chemical analysis and a full-field 3D micro-XRF (micro-X-Ray Fluorescence) scanner for reconstruction of 3D chemical composition inside the sample. Micro-XRF is based on pin-hole imaging coupled with a direct detection CCD (Charge-Coupled Device) operating in an energy-sensitive photon counting mode. Automated registration of the micro-CT image with the chemical composition map inside of the sample brings X-ray tomography to a new level of material property testing.

Results
A sandstone core plug was used to examine advantages of micro-CT/micro-XRF technique for rock core study. Some reference properties of the sample were collected in advance (Table 1, Figure 1). Mineral composition of the rock was obtained by classical XRD technique (Table 1); multi-scale X-ray micro-CT data was collected using SkyScan 1172 machine (Fig.1).

![Figure 1. X-ray micro-CT data for studied sandstone core plug: XY slices of sample scanned with different resolution are shown: A) 30 mm diameter plug scanned with resolution 9 µm/pix; B) 8 mm diameter plug; C) about 2 mm size chip of the rock](image)

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It should be noted, that in order to retrieve mineralogical information, greyscale micro-CT images can be subjected to special image processing procedure called multi-segmentation [2]. Unfortunately, the procedure is hardly applicable to large datasets, constrained with a number of mineral “phases” to be recovered, requires additional reference information and advance mathematical algorithm to be implemented. Figure 2 shows results of multi-segmentation applied to a subset of high resolution greyscale micro-CT data of the studied sandstone. Three main minerals (albite, quartz and microcline) were taken into consideration for image processing.

![Figure 2. Multi-segmentation applied to a subset of high resolution micro-CT data of the sandstone: black is pores, blue is albite; pink is quartz and yellow is microcline](image)

### Table 1. Studied sandstone mineral composition (XRD data)

<table>
<thead>
<tr>
<th>Type of mineral</th>
<th>%</th>
</tr>
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<tbody>
<tr>
<td>albite</td>
<td>39.3</td>
</tr>
<tr>
<td>quartz</td>
<td>31.8</td>
</tr>
<tr>
<td>microcline</td>
<td>24.6</td>
</tr>
<tr>
<td>kaolinite</td>
<td>2.1</td>
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<tr>
<td>smectite</td>
<td>0.9</td>
</tr>
<tr>
<td>illinit</td>
<td>0.8</td>
</tr>
<tr>
<td>calcite</td>
<td>0.5</td>
</tr>
</tbody>
</table>

Results of the micro-CT/micro-XRF study obtained with a 100 µm pin-hole detector are shown on the Figures 3–5. It should be noted, that scanning is performed at the atmosphere conditions (not vacuum) where very low-energy fluorescence photons do not have enough energy to reach the detector. Thus, Si and some other elements are naturally excluded from the observed micro-XRF signal. For the core study it is seen to be an advantage of the technique as more detailed analysis of low-content elements becomes possible.

![Figure 3. XRF signal collected for one angular projection for the whole range of elements (top) and corresponding spectrum of elements (bottom).](image)

Figure 3 represents the XRF signal collected for one angular projection for the whole range of elements (top) and corresponding spectrum of elements (bottom). Note that detector sensitivity as a function of energy is corrected for three processes: absorption in air, transmission through the Be camera window and detection in the CCD itself. As one can see from the spectrum, such elements as Fe, Sr, Ca, K, Zr, Ti, Mn and Zn are robustly detected in the core.

Unfortunately, it was found that X-ray adsorption inside of the core plug is too strong and fluorescence photons cannot escape from inside of the sample and thus cannot be detected. So, we were able to collect micro-XRF data only from the surface of the sample. Thus all data presented further correspond to signals collected from one of sample projection and depth of investigation remains an open question for the technique's applicability for physically dense objects.

The functionality of the SkyScan 2140 system allows analyzing single element distribution within the sample. It is a very powerful tool when combining collected micro-CT data with micro-XRF for selected energy channels. As an example, Figure 4 shows a registered micro-CT shadow image of the sandstone and a micro-XRF data for selected elements: Fe, Ca and Zr.
Figure 3. Top: collected micro-XRF signal for one of scanning projections; bottom: corresponding spectrum of elements. Color coding for elements: K — purple; Ca — pink, Ti — light blue, Mn — orange, Fe — green, Zn — blue, Sr — yellow, Zr — red.

Figure 4. Combined micro-CT (shadow projection) and micro-XRF for selected element distribution: Fe (A), Ca (B), Zr (C).

An interesting and perspective direction of data interpretation is to analyze the spectrum within any region of interest (ROI) selected by the physical element distribution in the sample. In Figure 5 this idea is implemented on the example of Ca. The distribution of Ca is registered over a shadow micro-CT projection image of the sample and shown in Figure 5A together with selected ROI. In Figure 5B a comparison of the detected spectrum of elements from the whole sample (top) with the spectrum of elements collected in the selected ROI (bottom) is presented. As one can find from the analysis of both spectrum, element co-existence can be analyzed and some algorithm for automated procedure of mineral recognition can be devised for data analysis.
Conclusion
Micro-CT/micro-XRF technique applicability to core analysis has been studied on the example of a sandstone core plug of 5 mm diameter. Although 3D visualization of element distribution inside of the rock seems to be problematic as rock matrix is very dense, potential value of the technique cannot be neglected. Thus, the obvious advantage is the fact that no sample preparation is needed for getting 2D elemental/mineral distribution. Moreover, it can be obtained for different angular projections of the sample. Registration with micro-CT data collected for exactly the same location makes it possible to track correspondence between morphological properties and chemical compounds of the sample in a more robust and straightforward manner. Absence of Si in the detected micro-XRF signal can be taken as an advantage that makes analysis of low-content elements in the rock reliable. It should also be noted that micro-XRF data can be obtained with different sizes of pin-holes (different resolution) and can therefore be used for rock heterogeneity analysis.

References: