Doctoral School of Geosciences

Quantifying variability of physical /petrophysical properties of Boda Claystone Formation (BCF) using X-ray computed-tomography scan images (CT)

Ph.D. Thesis

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INTRODUCTION AND AIMS

The Boda Claystone Formation (BCF) represents the fill of a relatively small, continuously subsiding, rift-related basins in the northern part of the internal Variscan orogenic domain (Vozárová et al., 2009). Its location is distributed over two areas in the Mecsek Hills in SW Hungary; Boda block and the so-called Gorica block. Continental sedimentation in the Mecsek Mts. was initiated in the Early Permian and lasted until the early Triassic, yielding a 2000-4000m-thick continental clastic sequence (Haas & Péró, 2004). The transitional and playa sediments of the BCF occur within the Permian fluvial sequence.

The lithology of the BCF starts with fine-grained sandstone beds at the base, overlain by albitic claystone/siltstone, and then continues with claystone, albitic clayey siltstone, and silty claystone with dolomite at the top (Konrád, 1999). Our core sample, 5m long , corresponds to the Gorica block, the upper part of the Ibafa-4 borehole.

This formation has been selected as a potential disposal repository rock formation for high-level nuclear waste (HLW) due to its low porosity, appropriate hydraulic conductivity, and absence of organic residues (Boisson, 2005).

3D X-ray computed tomography scans (CT) could efficiently reveal the spatial distribution of the inner structure of objects examined and provide unrivaled information about materials from scale lengths of meters, down to tens of nanometers non-destructively. The general purpose of this Ph.D. research, therefore, is to accurately quantify the variability of petrophysical properties and density of the rock-forming components of a core sample of BCF using CT scan images. Specific objectives could be addressed as follows:

- Evaluating of 3D small-scale lithological heterogeneities and pore distribution of the Boda Claystone Formation using X-ray CT.
- Assessing the representative elementary volume of rock types of BCF by X-ray computed tomography (CT)
- Calculation the representative elementary volume of porosity of BCF using X-ray computed tomography.

APPLIED CT SCAN IMAGES

A high-resolution X-ray CT facility at the Institute of Diagnostic Imaging and Radiation Oncology, University of Kaposvar, Hungary. The CT measurements were performed on a Siemens Emotion 6 medical scanner. The instrument operates at 120 kVp (peak kilovoltage), with 250 mAs (milliampere-seconds) current, and 1.0 s (sampling intervals). The lateral resolution was (0.1953 × 0.1953) mm^2 with 1.25 mm of scan-slice thickness. The image reconstruction matrix was 512 × 512 pixels. The field of view (FOV) was approximately 9.99 cm. CT images are stored in a DICOM (Digital and Imaging Communications in Medicine) format. Scans were used a modified dual-scanning approach (Balazs et al., 2018). Usually, rock samples are dried in a vacuum oven at temperatures of 120 to 210 °F (50 to 100 °C). Drying is terminated when the samples reach a stable weight (Soeder, 1986). After six hours of vacuuming the sample, all pore water was removed, and CT measurements were acquired (scan of the dry core). The next phase was pumping water in the whole dried sample (saturation process). After an hour of relaxation, those slices that went under vacuumed were flooded and re-scanned. CT images were stored in a DICOM (Digital and Imaging Communications in Medicine) format. A DICOM file contains the scanning parameters, i.e., Pixel Spacing and Slice Thickness attributes, in its metadata. These metadata are essential for geoscientific applications as they record each voxel's dimension (in millimeters) in the x, y, and z directions. This image format is a standard in medical applications and can be easily read by 'classical' 3D volume rendering software (e.g., VOXLER).

METHODOLOGY

Three major scopes were followed in this research: Pre-processing, Data mining, and REV calculations.

- **Pre-processing step**

A 3D-nearest neighbour algorithm was used to build the 3D volumes of the scanned dry core sample. This process resulted in two Hounsfield lattices, one for the vacuum dried and one for the saturated core volumes.

Although the output of computed tomography lends itself to straightforward interpretation, so-called scanning artifacts may obscure details of interest or cause the CT value of a single material to change in different parts of an image. The most commonly encountered artifact in CT scanning is beam hardening. Various methods have been developed to reduce or remove the effects of beam hardening. One of these is the so-called "subset" CT volumes in which the image's outer edges are removed, and only central volumes of the original three-dimensional images are used for quantitative analysis.

For identification of rock-forming components of the core sample, dry CT measurements were classified by PURAM to characterize rock types of the BCF. The intervals studied were as follows: detrital fragments (coarse siltstone): <2700 HU, fine siltstone: 2700-3150 HU, claystone: 3150-3300 HU, calcite and /or dolomite: 3300-3600 HU, and albite: >3600 HU. For illustration, the rock-forming components were compared with macroscopic core descriptions. Calculating the relative percentages of the rock-forming components for the dry-vacuum scan was also necessary to determine the dominant rock-forming components and lent the name of the rock type.

The voxel porosities of the scanned slices were computed from both lattices by normalizing the results of the dual scans subtraction via flooded HU values (Moss et al., 1990, Hove et al., 1988). Each voxel of the 3D CT volume was accordingly described with three variables: dry CT number, saturated CT number, and voxel porosity.

- **Data mining**

The spatial patterns of the voxels with similar effective porosity and textural characters were assorted by a generalized k-means clustering algorithm of data-mining techniques. Our work deals with Chebyshev distances; 10 (v-value) cross-validation and 20 iterations were applied in each approximation.

A hypothesis test, then, has been applied to evaluate and compare the calculated CT pore volume mean percentages with other conventional lab measurements, such as helium and mercury.

- **REV calculations**

Representative Elementary Volume (REV) was efficiently utilized in quantifying the inhomogeneity of CT densities of rock-forming components of the Boda Claystone Formation. The REV calculation was established using the Autoregressive Integrated Moving Averages, Statistical Process Control (ARIMA SPC) method.

The aforementioned process was also applied to study the REV of voxel porosity. Since the REV heavily relies on the spatial position of the initial volume, five different starting positions were pointed out. The ARIMA SPC, therefore, was five times iterated. Each time the initial voxel volume started from a new position in the BCF core sample (upper left, upper right, lower left, lower right, and middle). Consequently, we could compare the averages of the computed REVs and determine the adequate starting point position for REV calculations.

Monte Carlo simulation was used to simulate the long-run properties of REV and the normalized REV of both HU-density and voxel-porosity assessments.

RESULTS AND CONCLUSIONS

- **Results of Evaluating 3D small-scale lithological heterogeneities and pore distribution of the Boda Claystone Formation using X-ray Computed Tomography images (CT)**

    - The spatial patterns of the voxels with similar effective porosity and textural characters were divulged by a generalized k-means clustering algorithm, accordingly, three clusters had been defined: the micro-porosity, the macro-porosity, and no-porosity regimes.

    - The mean average of the matrix cluster porosity is 3.39, whereas the average of the macro-pores regime is about 10.77, and zero for the no-porosity one. while, the ratios of voxel-porosity of the three clusters are as follows: 30.37% for the matrix cluster, 14.65% for the macro-pores cluster, and 55% for the no-porosity cluster.
    - Each porosity cluster has different rock-forming components ratios. For example, the no-porosity cluster shows a very dense texture; it consists of about 19% fine siltstone, 23% Claystone, 13% carbonate, 0.16% albite, and is almost lacking detrital fragments. Compared with matrix cluster, the fine siltstone forms around 20%, and claystone is decreased to 8.44%. The carbonate content is also reduced to 1.66%, with no albite, and the detrital fragments component appears as 0.05%. macro pores cluster involves around 12% fine siltstone, 2.33%

claystone, 0.27% carbonate, and no albite. The detrital fragment increases to 0.23%.

- The total ratios of voxel porosity in each rock component over the three clusters are as follows: 0.29% detrital fragments, 50.74% fine siltstone, around 34% claystone, and 15% carbonate. Albite has the lowest porosity ratio summation along with three clusters at 0.17%.

- The He porosimetry measurements show quite close value for the calculated porosity mean of the CT scans image (2.51%), while the Hg measurement gives smaller porosity mean values at 2.02%. This can be attributed to the laboratory method's limitations concerning the inaccessibility of small pores to mercury penetration.

- In conclusion, authigenic minerals play an influential role in decreasing the pore volume and plugging the pore throats via swelling and migration of fine particles. Moreover, Cementation and the authigenic clay content are considered to be the main pore volume-controlling factors of the BCF core sample. In addition, Sedimentary structures, i.e., convolute structures, could also be a significant porosity-improving factor.

- **Results of Assessing the representative elementary volume of rock types by X-ray computed tomography (CT)**

  - The application of the Autoregressive Integrated Moving Averages, Statistical Process Control (ARIMA SPC) method to define Representative Elementary Volume (REV) of CT densities (Hounsfield unit values) affirmed the following results: 1) the highest REV values corresponded to the presence of sedimentary structures or high ratios of siltstone constituents (> 60%). 2) the REV average of the clayey siltstone was (5.86 cm^3) and (6.54 cm^3) of the fine siltstone. 3) normalized REV percentages of the clayey-siltstone and fine siltstone on the scale of the core volume studied were 19.88% and 22.84%, respectively. 4) whenever the corresponding layer did not reveal any sedimentary structure, the normalized REV values would be below 10%.

- As conclusion, the presence of structures (sedimentary structures or cracks) within the BCF core sample is more commonly interpreted as a type of heterogeneity; the higher the heterogeneity, the higher REV. notwithstanding, internal void space in the layers' sedimentary features might be the real reason for developing the high HU density contrast (increasing REV). We believe that the drying process of the core sample could essentially affect the available pore space and cause erroneous higher-porosity measurements (by collapsing clay), especially at sedimentary structures and cracks, where authigenic clay is expected to occur primarily.

- **Calculating the representative elementary volume of porosity of BCF using X-ray computed tomography.**

  - The REV calculation was established using, again, the (ARIMA SPC) technique. This method (ARIMA SPC) was five times iterated by varying positions of starting voxel volume in the core sample: upper left, upper right, lower left, lower right, and middle. The average range of REV values was from 16.56 cm^3 to 46.26 cm^3.

- Monte Carlo simulation with 1,000 runs was used to simulate the long-run properties of REV and the normalized REV of voxel-porosity. The simulated REV values were 64.80 cm^3 in the upper left position, 61.44 cm^3 for the upper right, 55.59 cm^3 for the lower right, 53.77 cm^3 for the lower left, and 42.23 cm^3 for the middle. According to the simulation of the normalized REVs, their volume percentages were as follows: 40 % for the upper left, 41.34 % in the lower right, and 23.86 % for the lower left. Lastly, the upper right and the central (middle) positions had almost the same percentage volumes, around 15.5 %.

- As the initial volume started from any core sample's corners, the porosity REV range would be between 2.47 % and 2.58 %, which is close to the average voxel porosity calculated of BCF (~2.5%). However, a deviant (higher) porosity REV value was attained from the center initiated volume (~ 2.8 %).

- The remarkable result associated with the highest porosity REV was studied deeply by applying further quantitative porosity analysis. Accordingly, the following conclusions were derived: 1) both detrital fragments and albite constituents might influence the averaged porosity proportions of the studied layers by enhancing the presence of macro-porosity ratios (7-25%). Conversely, the absence of detrital fragments and albite might intensify the matrix-porosity ratios (0-7%). Matrix (throat)-pores are almost related to dense proportions of claystone- siltstone components. 2) although the detrital fragments might be a factor in the macro-pores presence, the albite almost plays a significant part in developing macro-porosity. 3) The saturation process of the core sample studied was the key to interpreting the dual role of the albite. Hence, the albite particles could release and re-deposited downstream in pore throats during the core sample saturation process. Compiling its particles might be causing throats to be plugged (decreasing porosity). Otherwise, releasing albite particles from pore volume might generate an extra macro-pore space (increasing porosity ratio).

- As a result, when the initial voxels started from a medium where the macro-porosity is expected to exist primarily (detrital fragments and albite took place), the higher porosity REV was gained (~2.8%). Alternatively, when the volume of the initial voxel occurred within the matrix porosity medium (barely presence of albite and detrital fragments), the REV porosity ratio would be < 2.8%. That is to say, the middle part of the studied sample might not be adequate to be considered as an initial voxel volume of the REV calculation because its porosity REV value was significantly higher (~2.8%) than the average of the effective voxel-porosity of the BCF (~2.5%).

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List of publications used in the dissertation

Saja Mohammad Abutaha (MTMT author ID: 10082129)

- **Abutaha** S.M., Geiger J., Gulyas S. & Fedor F., 2021a: Evaluation of 3D small-scale lithological heterogeneities and pore distribution of the Boda Claystone Formation using X-ray computed tomography images (CT). *Geologia Croatica*, 74/ 3, 305–318. DOI: 10.4154/gc.2021.17. (impact factor: 1.273).

- **Abutaha** S.M., Geiger J., Gulyas S. & Fedor F., 2021b: Assessing the representative elementary volume of rock types by X-ray computed tomography (CT) – a simple approach to demonstrate the heterogeneity of the Boda Claystone Formation in Hungary. Geologos, 27/ 3, 157 152. DOI: 10.2478/ logos-2021-0018. (impact factor: 0.784).

- **Abutaha** S.M., Geiger J., Gulyas S. & Fedor F., 2022: Calculating the representative elementary volume of porosity using X-ray computed tomography: Boda Claystone Formation core sample/Hungary. Acta Geologica Slovaca (AGEOS), 14/1, 25–36. (impact factor: 0.5).

Other publications

- **Abutaha** S., Atallah M., Abed A. M., 2019: Structural Evolution of Wadi Hudaydun in Wadi Shueib Area, NW Jordan. Jordan Journal of Earth and Environmental Research (JJEES), 10/3, 152-160. ISSN 1995-6681

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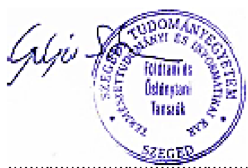
Conferences abstracts

- **Abutaha** S.M., Geiger J., Gulyas S. & Fedor F. (2022). Assessing the representative elementary volume of rock types by X-ray computed tomography (CT) – a simple approach to demonstrate the heterogeneity of the Boda Claystone Formation in Hungary, conference abstract, GeoMATES ’22”, International Congress on Geomathematics in Earth- & Environmental Sciences. Pécs, Hungary, P 30.

Statement of the supervisor

I, **Dr. Sándor Gulyás,** as supervisor, declare that the thesis written by Saja Mohammad Ahmed Abutaha (Neptune code: I1WS0J) titled **Quantifying variability of physical /petrophysical properties of Boda Claystone Formation (BCF) using X-ray computed-tomography scan images (CT)** is her own writing prepared under my supervision; the candidate's contribution to the results used in the discussion of the thesis is approved. I also declare that the thesis meets the formal and professional requirements of the Doctoral School of Geosciences of the University of Szeged and the Faculty of Science and Informatics/ Department of Geology and Paleontology; thus, I support its submission.



Szeged, 14/07/2022

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(supervisor)

Co-authors' waivers

We as co-authors of the publication entitled „ ***Evaluation of 3D small-scale lithological heterogeneities and pore distribution of the Boda Claystone Formation using X-ray computed tomography images (CT)***" published in the Journal "***Geologia Croatica***" officially declare that the jointly published results in the thesis and the publication are greatly contributed by the candidate and was not or will not be used in the past or in the future, respectively, for the purpose of acquiring an academic degree or title.

Date 14/07/22

Name and signature of co-authors

János Geiger ……………………………………………

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Ferenc Fedor ………………………………………………

We as co-authors of the publication entitled "***Calculating the representative elementary volume of porosity using X-ray computed tomography: Boda Claystone Formation core sample/Hungary***" published in the Journal "***Acta Geologica Slovaca (AGEOS)***" officially declare that the jointly published results in the thesis and the publication are greatly contributed by the candidate and was not or will not be used in the past or in the future, respectively, for the purpose of acquiring an academic degree or title.

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