Integration of X-ray micro tomography and fluorescence for applications on natural building stones

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Abstract. X-ray computed tomography (CT) is an excellent, non-destructive analysis tool for characterising many different materials. In geosciences, 3D visualisation is becoming of prime importance in characterising internal structures of various rock types. It enables new approaches in petrophysical research of rock components, including pore and mineral distribution. Although CT provides a lot of information, this technique is limited concerning information on chemical element distribution. X-ray fluorescence (XRF) on the other hand is an excellent technique to obtain the missing information on chemical properties. At the recently established “Centre for X-ray Tomography” of Ghent University (UGCT) a micro- and nanoCT scanner has been constructed. It is expected that by combination of high-resolution CT and XRF it will be possible to characterise the spatial mineral and element distribution. The combination of both techniques has been applied on natural building stones, in order to get a better insight into some geological parameters (porosity, pore structure, mineral distribution, colour, grain orientation, etc.). Afterwards, the integration of the Morpho+ software tool provides us a 3D quantification of the resulting data.

1. Introduction
All monuments built with natural building stones are sooner or later affected by weathering processes. Most of the stones are formed under relative high temperature and pressure conditions in the earth’s crust. Placement of those stones in buildings results into a thermodynamically unstable situation. Exposure of the stone to chemical, biological and physical weathering disintegrates the material and can lead to problems concerning the stability of the building. The use of alternative stone or replacing stone might solve problems of stone decay, but is not always a desired solution. Geological parameters that differ from those of the original stone may result in dissimilar weathering processes and thus different patina. Understanding the properties of natural building stone on micro-scale and the knowledge of its mineralogy are crucial for the assessment of weathering degree. To obtain important information on natural building stones, non-destructive tools as X-ray computed tomography (CT) and X-ray fluorescence (XRF) can be applied [1]. Without any form of sample preparation it is possible to

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obtain a full 3D model of the samples’ internal structure within a few minutes. The mineral distribution of the Balezjem sandstone and the Massangis Roche Claire limestone has been visualized and quantified. Iron bearing minerals are potential spot producers, due to the oxidation of the iron; so its distribution is an important aspect in the evaluation of natural building stones.

2. Methods and instrumentation
To perform CT, digital radiographs of the sample are made from different orientations by rotating the sample along the scan axis from 0 to 360 degrees. The sample, in this case a natural building stone, is positioned between the X-ray source and the detector. In only one radiograph, the information of the sample is summed along the rays of the X-ray beam. This can already reveal some information, but to obtain a full 3D model, 800 radiographies were taken. Afterwards the projected data has been reconstructed, which produces horizontal cross-sections of a sample [2]. X-ray CT is based on the X-ray transmission information and from Beer’s law it follows that:

\[ I_\text{x} = I_0 \cdot e^{-\mu x} \]  

where \( I_\text{x} \) is the measured intensity of the X-rays, \( I_0 \) is the emitted intensity, \( \mu \) is the linear attenuation coefficient and \( x \) is the thickness of the object [3]. The attenuation of X-rays is strongly increasing with higher mass numbers and a few centimetres of natural building stone can already attenuate the entire X-ray beam intensity. The resolution we can obtain depends on the resolution of the detector, the spot size of the X-ray source and the magnification. The latter is expressed as the ratio of the source detector distance (SDD) to the source object distance (SOD) and depends on the largest diameter of the sample. In case of natural building stone, with a diameter of 8 mm, we obtain a resolution of 15 µm. Despite constant improvement of CT research and its valuable contribution to detect physical properties (like porosity changes), there is still missing information on chemical properties. To obtain those missing information add-ons like XRF can be applied.

The experiments were carried out at the Centre for X-ray CT at the Ghent University (Belgium) [4]. The X-ray tube of this high-resolution CT scanner is an open-type device with dual head (Feinfocus®, FXE-160.51). The head can produce a voltage between 10-160 kV, but for natural building stones a voltage of 130 kV with a power of 8 W was chosen, with an Al-filter to block the low energetic X-rays. As X-ray detector, a Varian 2520V Paxscan was used. The CT data is processed with the in-house developed reconstruction software Octopus. 3D morphological analysis was performed using the software Morpho+ which is able to segment the volume using advanced tresholding and to label, classify and separate tools. For 3D volume rendering, VGStudioMax from Volume Graphics was used. XRF analyses are made with the X-PIPS® Detector (Canberra). The flexible set up of the scanner area at the UGCT allows using the same tube of the CT scanner as source for the XRF measurements.

3. Experimental data and results
With the aid of Morpho+ it is possible to obtain 3D data from the reconstructed 2D slices. The iron bearing minerals (glauconite) inside the Balegem sandstone and the Massangis Roche Claire limestone were segmented and labelled. Since the iron bearing minerals have similar grey values, they can be segmented as one object. Because the scans were performed with a resolution of 15 µm, fine dispersed grains cannot be visualized. Our 3D analysis shows that the limestone consists of 0.007% iron bearing minerals and the Balegem sandstone of 0.13%. The visualization of the mineral distribution is shown in figure 1. The XRF analyses were done with an integration time of 500 s and a tube voltage of 60 kV. Only surface detection of iron was possible, because the secondary X-rays cannot penetrate through the stone. The graphs in figure 2 clearly reveal the high iron concentration originating from the glauconite in the Balegem sandstone and the calcium rich content in the Massangis Roche Claire limestone. Due to their different mineral composition, the two natural building stones will have different weathering patterns.
4. Conclusions

An X-ray CT system is a powerful tool to visualize the internal structure of natural building stones, in a non-destructive way. To obtain missing information concerning chemical properties, XRF can be applied. CT provides the possibility to compare some geological parameters of the replacing stone with the original material, to assist the restoration in welfare to our historical buildings. Materials with high glauconite content will have weathering patterns different from other stones. Due to the conversion of glauconite into goethite, the Balegem sandstone acquires a rust-colour pattern, while the Massangis Roche Claire remains white. Topics like porosity inside stone samples can be visualized and quantified with the assistance of Morpho+. Mineral distribution of replacing materials can be compared with the original material in a non-destructive way and new building materials can be characterized and their weathering qualities assessed.

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