

Visualization of phases inside porous medium with X-ray microCT

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Aims

One of the main goals of current study was to develop X-ray microCT based approach for investigation of phase distribution inside pore space of rock samples. This information has fundamental scientific interest (for instance, studying of fluids/ions migration, formation of properties) and can be used in practice in different areas of Oil-gas industry, e.g. for optimization of enhanced oil recovery (EOR) methods, analysis of mechanisms of reservoir formation damage etc.. Low X-ray attenuation coefficients of substances which typically fill the rock pores (oil/water/ice/clay and etc) in comparison with solid rock matrix¹ make the phases almost invisible in microCT experiments. At the same time, saturating phases with small amount (in most cases less than 1% of mass) of X-ray contrast agents allows sufficient improvement of saturating phases visibility in 3D and building the 3D phase distribution map inside porous rock. Current study shows results of different physico-chemical approaches to improve contrast of phases inside saturated rock.

Method

X-ray tomography investigations were carried out using 1172 X-ray micro Computed Tomography (SkyScan, Kontich, Belgium). Standard scanning parameters were: rotation step 0.2°, 8 frames averaging, angle of rotation was 360°. X-ray tube voltage was 100 kV and current was 100 uA, built-in Al+Cu filter was used.

For scanning of the ice/gas-hydrate saturated rocks the samples were placed into SkyScan's Peltie Cooling stage during whole microCT experiment. The stage holder temperature was -10°C to avoid ice melting.

3D sample reconstruction was performed using NRecon (SkyScan, Belgium) with InstaRecon software (InstaRecon, USA). Data were analyzed by CTAn software (SkyScan, Belgium).

The basis of the X-ray tomography method is the reconstruction of spatial distribution of linear attenuation coefficient (LAC) of X-rays in the flat layer of the object under test on the ground of computer mathematical processing of back projection received by X-ray in different directions along the investigated layer.

The value of LAC of every substance depends on chemical composition, material density and X-ray radiation energy:

$$\mu = \mu_m \rho$$

μ_m – mass attenuation coefficient at given X-ray radiation energy (cm²/g), ρ – material density (g/cm³).

The idea of correlation between the gray values distribution in X-ray CT image and material densities distribution is related to patent application².

Unfortunately, it is common situation when mediums of interest have weak X-ray contrast and cannot be spatially resolved with required accuracy. In this case different salts of heavy metals (X-ray contrasting agents) are used to improve X-ray contrast of medium of interest.

In this study we applied non-destructive approach of improving contrast of ice/gas hydrates and clay solution inside rock porous medium based on two different effects: solid state ions

(X-ray contrast agent) diffusion at low temperature and selective ions exchange in case of clays.

Results

Ions migration of chemical elements at low temperature (below 0°C) was previously studied at Geological faculty of Lomonosov Moscow State University³. The experiments were based on contrast method of interaction between samples of frozen soils or ice with water-based solution of various metals under isothermal conditions. Using Fick's law the ions diffusion coefficient in frozen soils and ice was determined. For instance, the effective diffusion coefficient of $\text{Pb}(\text{NO}_3)_2$ in ice is $0.2 \times 10^{-5} \text{ cm}^2/\text{sec}^{-1}$. The ionic permeability of ice does not vary much for different chemical elements; it is essentially determined by temperature conditions. Described approach is used to improve X-ray contrast for ice inside pore samples that allow accurate estimation of spatial distribution and ice concentration by analysis of X-ray cross-sections (Figure 1).

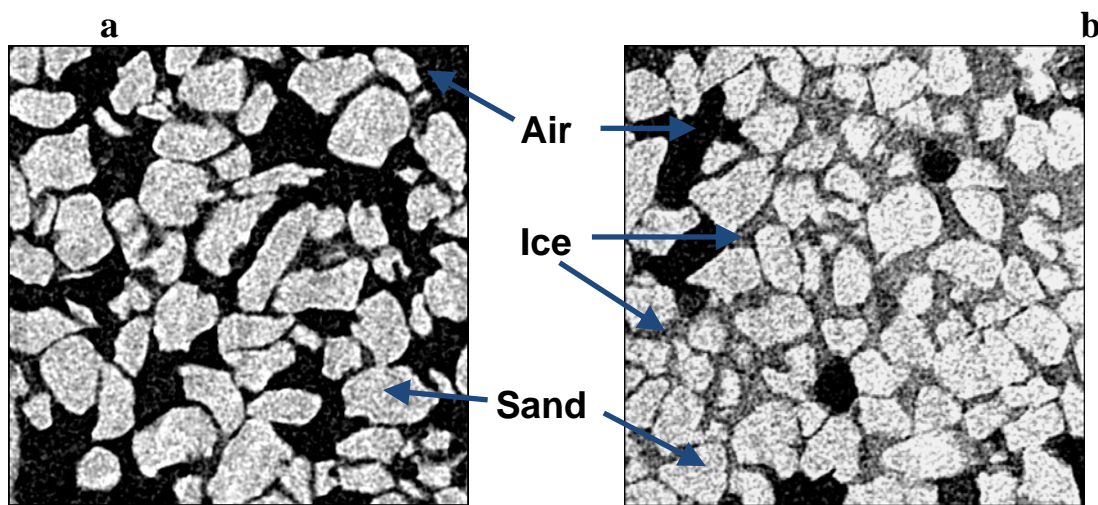


Figure 1: MicroCT images of ice contained sand samples without contrast agent (a) and saturated with lead ions (b)

Initially, rock sample contains ice without contrast agents; as a result it is almost invisible in microCT images (Figure 1a). Then the sample was contacted at -7°C with frozen water-based solution of lead nitrate. Leads ions diffusion allows clearly distinguish ice from air in porous structure (Figure 1b).

Conventional grayscale histogram for an ice-saturated sample without contrast agent has two main peaks (Figure 2, red curve): for air in pore space (grayscale index equals about 70) and solid rock matrix (grayscale index equals about 160). Ice has low adsorption of X-ray radiation⁴ that leads to absence of ice phase related peak on grayscale histogram (Figure 2, red curve).

The same ice-saturated sample histogram, after solid state diffusion of lead ions (at -7°C), reveals additional peak appearance between air in pore space and solid rock matrix (Figure 2, blue curve).

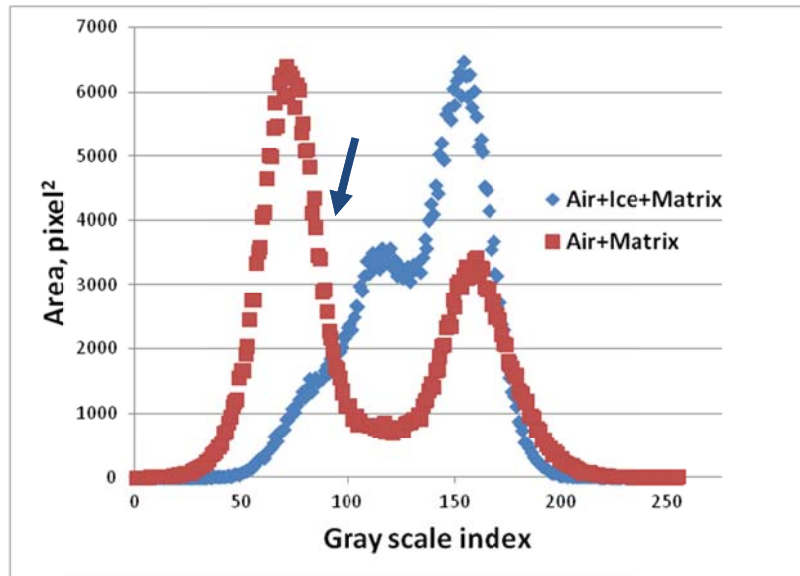


Figure 2: Characteristic gray scale histogram for pore ice contained sample with contrast agent and without. Additional peak of ice phase is marked by arrow

CTAn based calculation of pore size distribution, using “structure separation” function, has shown (Figure 3) that ice is accumulated mainly into pore range 25-50 microns (μm).

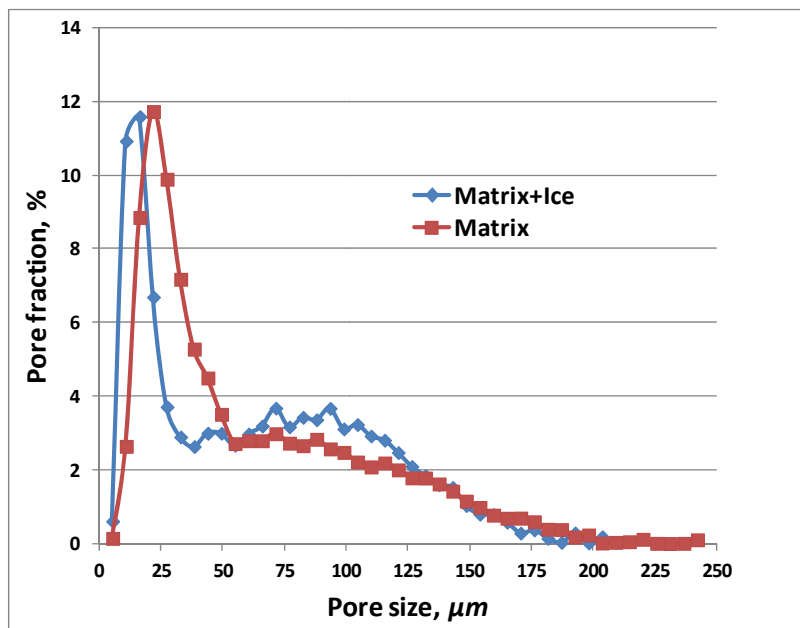


Figure 3: Pore size distribution for two cases: rock matrix porosity and remain porosity taking into account the pore ice

Analysis of ice (gas hydrates) distribution along rock core sample would help in investigation of frozen core samples and defining the mechanism of ions migration in frozen rock. X-ray contrast agents can be also used for increasing the contrast of clay or clay solution inside pore space (Figure 4). That allows better estimation of spatial distribution and concentration of clay by analysis of X-ray cross-sections of core sample.

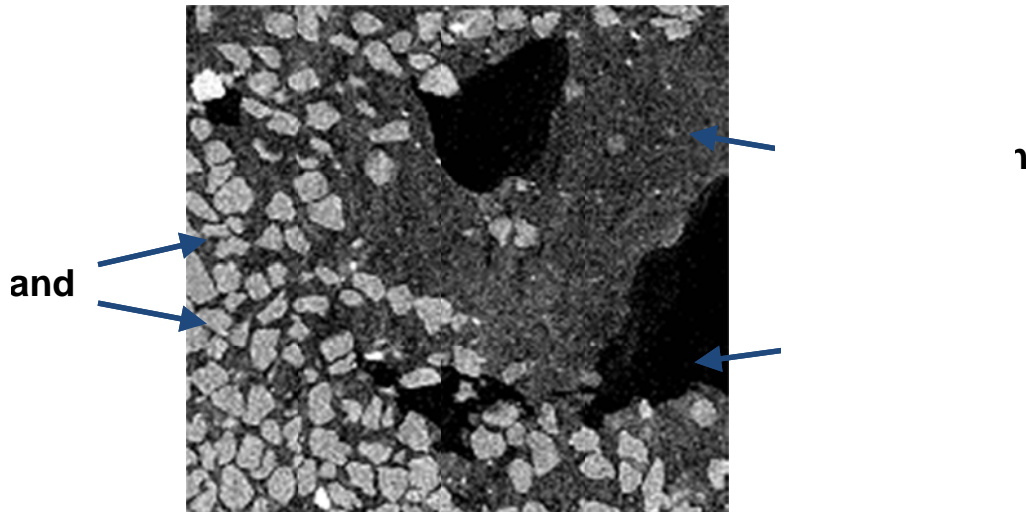


Figure 4: Accumulation of Barium ions in clay allow to spatially resolve air, 5% (in mass) water based clay solution and sand particles (for wet sample)

The current approach is focused on using of effect of accumulation of X-ray contrast agent due-to selective ion exchange and adsorption to improve contrast of clay. The soluble salts of metals with high atomic weight (Ba, Sr, Ra etc) are good compounds for this contrast additive.

Special interest of this approach is caused by problem of formation damage by mud component invasion during drilling the wellbores. The problem of formation damage due to the mud (or flush liquid) effect is a very important one (especially for long horizontal wells as most of them are completed open-hole, i.e. without a cemented and perforated liner).

Clay particles forms after core sample drying. The drying makes clay very high contrast inside pore medium (Figure 5).

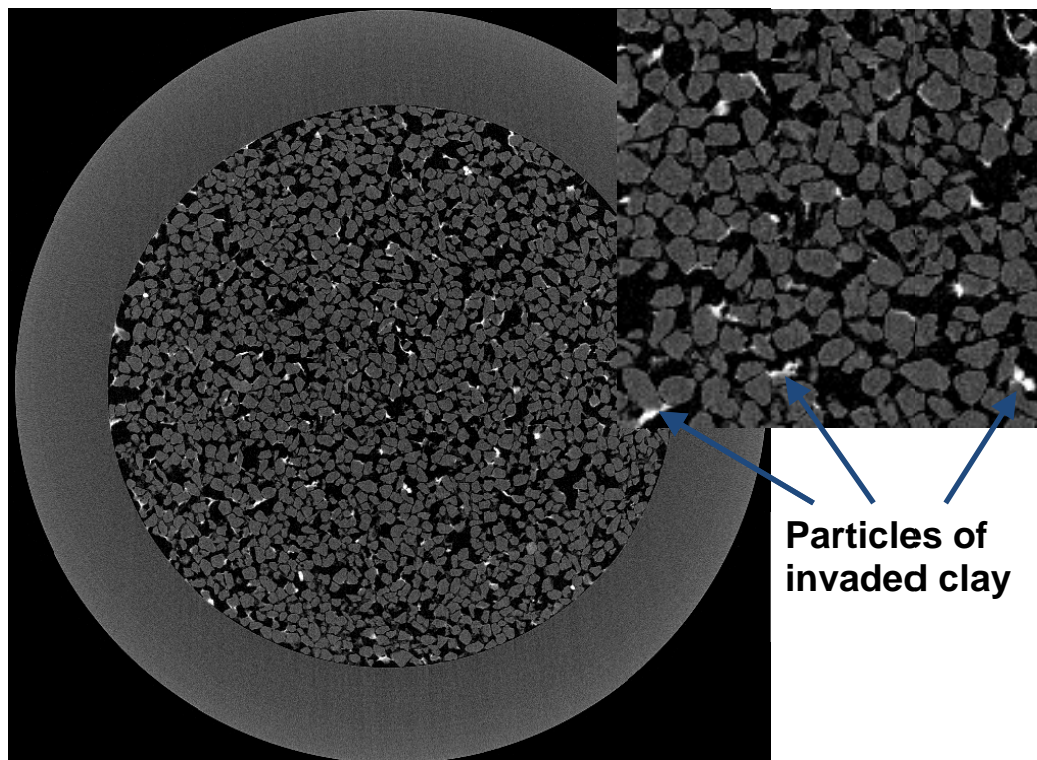


Figure 5: Visualization of clay particles invasion in dried sample. X-ray contrast of clay is improved by special contrast agent

Analysis of clay distribution along core sample would help to estimate mechanism of formation damage.

Conclusion

3D mapping of fluids distribution inside rock core samples is carrying a crucial information for fundamental science, for instance, solid material (clay, polymers and etc) transport in porous medium and it is also important for many practical applications including oil-gas wells production intensification. For unconsolidated or low consolidated reservoir rock, ice in porous medium is able to plays role of natural cement. The only chance to investigate petrophysical properties (porosity, pore size distribution, residual water saturation and etc) of such rocks is to improve ice contrast for correct matrix-ice segmentation in low temperature microCT measurements.

References:

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