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eEarth Discussions

Interactive comment on "Morphology of the pore space in claystones – evidence from BIB/FIB ion beam sectioning and cryo-SEM observations" *by* G. Desbois et al.

Anonymous Referee #1

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Referee Comment

Morphology of the pore space in claystones –evidence from BIB/FIB ion beam sectioning and cryo-SEM observations.

General comments

This paper presents some novel results showing the capability of a powerful new technique for the characterisation of fluid-filled pore space in clay-rich rocks. The technique of sectioning using either a focused or broad ion beam followed by imaging using scanning electron microscopy has so far only been applied in a few studies to geological

materials. This paper is a first proof of concept to show that the technique can be applied to fine-grained claystones providing crucial direct information on the morphology and size distribution of pore spaces in this rock type. The results are of general interest because of the importance of claystones as sealing layers for crustal fluids and in this particular case as sites for radioactive waste disposal. This paper is sufficiently novel and interesting to be published in eEarth, although there are some points that should be addressed before the final version is accepted.

The main result of the paper is that the SEM measured porosity, in a small volume, is 20.4% in freeze dried samples, and 26% in pre-dried samples, compared to the mercury-porosimetry data, for larger volumes, of 24-27% in dried samples, and a water content porosity of 36%. On the basis of these numbers this study seems to show good agreement for porosity measured by direct SEM and indirect mercury injection methods. Yet the authors claim that their results call for re-interpretation of traditional pore distributions measured from mercury injection. This inconsistency should be corrected and discussed before the paper can be accepted for eEarth. See point 8 in the specific comments.

The authors note that the SEM technique has limited resolution so that pores below 10-20 nm diameter and nano-scale water films along grain boundaries and in clay layers will not be resolved. A key point is the difference in volume analysed in each technique. Some further work on a wider range of sample volumes will be needed to determine if the porosity values obtained from different techniques, are because of a scale dependence of porosity or because of possible artefacts with indirect methods. Another possibility that should be considered in future studies is the role of clastic grain content and degree of folding on porosity. The authors show that the largest pores are jagged shaped pores surrounding clast grains. Furthermore the largest pores tend to be connected so the clast content of the claystone should have a strong effect on the porosity and permeability of these rocks.

The methods and assumptions used in this study are clearly outlined, although, it would

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be useful to include some more details on the operating conditions used for ion sectioning and electron imaging.

Specific comments

1. Abstract. The authors claim that their findings call for re-interpretation of traditional pore size distributions calculated from mercury injection experiments. In the discussion it is shown that the SEM measured porosity can be described by a fractal distribution but no comparison is made with distributions from mercury injection experiments. The only comparison between SEM data and mercury data is for the porosity. A comparison with pore distributions from mercury injection should be included or the abstract should be re-worded.

2. Different terms are used for the ion beam sectioning in different parts of the paper, ion beam sectioning, ion beam excavation, ion beam cutting). It would be better to use one term to describe the removal of material by ion sputtering.

3. In section 3.1; on line 15 the word melting should be replaced by sublimes.

4. Section 3.2; how was the organic material, left in the pre space after sublimation, identified?

5. Section 3.3; line 10. It would be useful to indicate on the image, locations where the connection of pore throats to neighbouring pores can be seen.

6. Section 3.4; is the volume studied in detail representative of the clast content of the clay?

7. Section 4.1; in this section the authors note on line 2 that plunge freezing vitrifies the pore fluids. Yet on line line 11 they state that the vitrification of our samples is not fully validated. These statements appear to be contradictory.

8. Section 4.2; line 16, it is noted that the SEM-measured porosity (20.4% in freeze dried, 26% in pre-dried) is higher than the porosity based on mercury injection data.

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(24-27% for pre-dried samples). Is there a typing error here because the SEM value of 26% actually agrees quite well with the mercury value of 24-27%.

Interactive comment on eEarth Discuss., 4, 1, 2009.