DANMARKS OG GRØNLANDS GEOLOGISKE UNDERSØGELSE RAPPORT 2008/47

CO_2 SINK: WP 3.1 Rock-/Fluid Interactions. Laboratory Experiments

Deliverable D3.1-11, Preliminary caprock analysis data from new wells

Niels Springer

GEOLOGICAL SURVEY OF DENMARK AND GREENLAND
MINISTRY OF CLIMATE AND ENERGY

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Contents

Req. no.: 08307-534
Files: CO_2Sink_rep2.doc
Caprock Ktzi wells_poroperm-new.xls
Caprock_CT1.xls

1. Introduction

GEUS is a partner in the EU Project "CO_2 Sink : In-situ R&D Laboratory for Geological Storage of CO_2", and GEUS Core Laboratory participates in WP 3.1 "Rock-/ Fluid Interactions – Laboratory Experiments". The objective of this Work Package is to characterize the storage site and study the reactions taking place during injection and storage of CO_2 at the Ketzin demonstration site outside Berlin. Part of the obligations in WP 3.1 is to evaluate the quality of the caprock sealing the Ketzin structure, and the present Delivery D3.1-11 reports on the preliminary data recorded so far from the caprock section in two (of three) new wells drilled at Ketzin in mid 2007. No caprock core was retrieved from the third well.

This Delivery is a listing of routine core analysis data and screening work considered necessary to select caprock core material for the advanced caprock analyses about to be initiated.

2. Sampling of caprock core

During the period March-September 2007, 3 new wells were drilled at the Ketzin site outside Berlin. All wells were cored, but only 2 wells were cored in the caprock section. Details of measuring up and description of the core as well as measurements performed at the Ketzin core field laboratory has been given in a recent report by Norden and Förster (2008). Core analysis on the potential reservoir section was handled by a commercial laboratory, but by tradition analysis of caprock is normally not included with the routine core analysis work. It was therefore decided to take a limited number of caprock samples and analyze for routine porosity-permeability at GEUS Core Laboratory that should do the advanced core analysis work on the caprock anyway.

Sampling for the caprock study was performed at wellsite on June 12, and September 27, 2007. From the Ktzi-200 well 16 core pieces were picked and from the Ktzi-201 well 4 core pieces were picked and later shipped to GEUS in Copenhagen.

2.1 Plugging

From a previous study (Springer et al., 2006) it was known that the variegated, dolomite cemented mudstones of the Triassic Weser Fm (Oberer Gipskeuper) are sensitive to drilling and cleaning liquids. Cooling with formation brine or compressed air during plugging will often provoke sample fracturing. By using a light liquid paraffin (LLP) oil (Isopar-L™) as a coolant, a successful plugging programme was finally carried out. Samples were cylindrical plugs of 38mm diameter and varying length with the majority having vertical orientation rel. to the bedding; a few horisontal plugs were taken however, to estimate the k_v/k_h ratio of the caprock. A list of plug samples are given in Appendix 1.

2.2 Sample preparation

Plugs were trimmed to a suitable length. As the plugs could not stand cleaning with normal cleaning liquids or water, they were just left to dry at 60 ˚C in an oven for a couple of days before being passed on for routine core analysis. The LLP oil used during plugging is known to evaporate completely at room temperature leaving no trace of oil components and will therefore have no effect on the following analytical work.

2.3 Sample screening

After routine core analysis a number of plugs were selected for advanced caprock testing and sent to the X-ray CT-scanning facility at the Technical University of Denmark for imaging of the internal sample texture. X-ray images are included with Appendix 3.

2.4 Sample description

A visual presentation and short description of plugs are given in Appendix 2 and 3. During drilling of plugs it was observed that many core pieces were transsected of an apparently stochastic network of fine open fractures – no systematic orientation of the fractures could be determined, fig. 2.1 and 2.2. We believe this chaotic fracture network to be responsible for the fragile nature of the core samples making it very difficult to drill cylindrical plugs from the Ketzin caprock core. Cementation with anhydrite is widespread and appears as white spots and irregular, flaser like textures that are clearly seen in the X-ray images; an example is shown below in fig. 2.3.

Figure 2.1. Ketzin 200/2007, core A-9-3, depth 626.70 m (cf. Appendix 2, Fig. 9). After plugging, the volatile LLP oil evaporates from the surface of the core leaving the fracture network wet, and therefore visible to the naked eye.

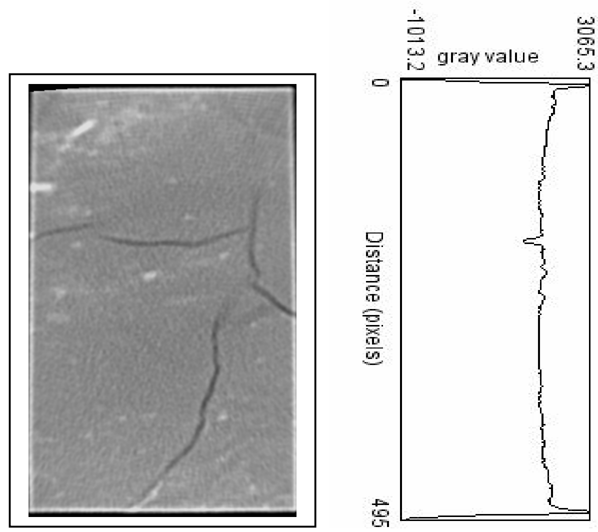


Figure 2.2. Ketzin 200/2007, core A-9-3, plug 9V (cf. Appendix 2, Fig. 9). Longitudinal X-ray CT-scan through plug 9V showing the chaotic fracture network. Fractures (high porosity) appear dark on the image and one is clearly visible from the profile plot through the middle of the image. The grey scale figure is in Hounsfield units; -1000 represents air (100% porosity) and appears dark on the images, +3000 is dense matrix (0% porosity) and appears white on the image.

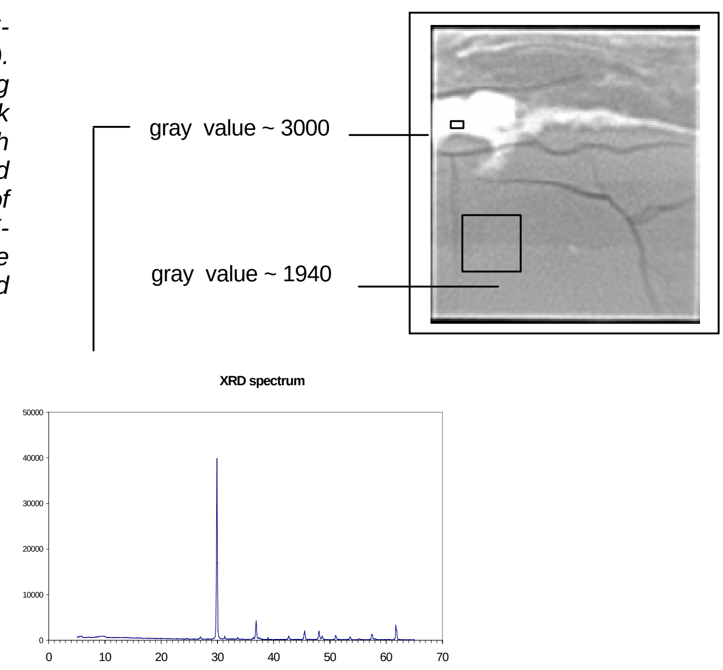


Figure 2.3. Ketzin 200/2007, core A-6-2b, plug 5V (cf. Appendix 2, Fig. 5). Longitudinal X-ray CT-scan through plug 5V showing the chaotic fracture network and flaser anhydrite. Cementation with anhydrite ($CaSO_4$) is seen as spots and flaser like textures in the mudstones of the Weser Fm. Anhydrite has a high X-ray attenuation and appear white on the image (gray scale figures around +3000).

3. Core analysis

3.1 Analytical

The samples were analyzed in a dry (but uncleaned) condition according to API recommended practice for core analysis procedure (API RP 40, 2nd ed. 1998). Porosity and grain density was measured by the Helium injection technique, and gas permeability was measured relative to dry N_2 gas at a confining stress of 2.8 MPa (400 psi) on the samples.

3.2 Conventional core analysis

Caprock material is notoriously difficult to clean due to the very low permeability of mudstones. The core analysis data listed for the Ketzin caprock samples relates to uncleaned and dried samples, and are therefore affected by salt precipitated in the pore space and some adsorped humidity. The samples were not heated to 110 ˚C, but only to 60 ˚C to prevent drying fractures. The effect will be a lower porosity and grain density than measured for cleaned and completely dried samples.

The first poro-perm measurements were obtained in November 2007. A later check measurement in June 2008 revealed that some plug samples may not have been completely dry when first analyzed. When this was recognized, the whole set of plugs were re-dried at 60 ˚C for a week with several checks on the dry weight to make sure that stable conditions had been obtained. Samples were then re-analyzed for poro-perm and the new set of data listed in Appendix 1. Compared with the November 2007 set of data there is an increase in porosity and grain density for most samples; gas permeability however, is less affected because it is mainly controlled by the fracture network present in the samples and core pieces right from the start.

Table 3.1. Ketzin 200 & 201 conventional core analysis data for uncleaned caprock material. Average poro-perm data measured for matrix mudstone plugs of the Weser Fm. , ref. Appendix 1. Observe that most plugs from the Ketzin 200 well have fine fractures and thus cannot contribute to the calculation of matrix permea-bility. Also the k_v/k_h ratio is very uncertain due to the scarce number of unbiased measurements.

Well	Mean		Mean		Average matrix	
ID	Por [%]	Sdev [%]	GD [g/cc]	Sdev [%]	Gas perm* [mD]	k_v/k_h ratio
Ktzi-200	11	3	2.74	0.04	0.004	0.33
Ktzi-201	14	1	2.75	0.02	0.018	0.63
All plugs	12	3	2.74	0.04	0.010	0.56

* Vertical plugs only

The caprock samples from Ketzin have ambient porosity values close to 12 % and slightly elevated grain densities due to the cementation with dolomite and anhydrite. There is no significant correlation between gas permeability and porosity, fig. A1.1; the distribution pattern seems to be controlled mainly by fractures and the dolomite / anhydrite cementation.

4. References

API RP 40, Recommended Practice for Core Analysis, second edition, 1998. Washington DC: American Petroleum Institute.

Norden, B. & Förster, A., 2008: WP 3.2 Integrated Reservoir and Caprock Characterization. Characterization of Caprock and Reservoir rock of the CO_2SINK Ketzin Site based on Well Logs and Laboratory Measurements on Cores. Delivery D3.2-1, GFZ internal report.

Springer, Niels; Lindgren, Holger; Fries, Kirsten: CO_2 SINK: WP 3.1 Rock-/Fluid Interactions. Laboratory Experiments. Caprock seal capacity characterization of old core material from the Ketzin structure.
Danmarks og Grønlands Geologiske Undersøgelse Rapport nr. 24, 2006

Appendix 1

A1.1 Core analysis data

Table A1.1. Ketzin 200/2007 conventional core analysis data for uncleaned caprock material. Most plugs from this well had fine fractures affecting the measured gas permeability.

Sample Number	Core Depth (m)	Core Run	Core Section	Gas Horizontal Permeability (mD)	Gas Vertical Permeability (mD)	Ambient He-Porosity (% of Vb)	Grain Density (g/ml)	Remarks
1H	610.70	3	1	0.006		8.2	2.798	
1V	610.70	3	1		0.002	6.6	2.801	
2V	612.70	4	1		0.007	5.3	2.767	
3V	614.60	4	3			12.9	2.742	Plug fractured
4V	616.60	5	3			15.1	2.737	Plug fractured
5H	618.70	6	2	2.52		11.6	2.682	Frac perm
5V	618.70	6	2		0.28	13.7	2.723	Frac perm
6V	620.53	7	1		0.003	12.0	2.721	
7H	622.64	7	3	14.0		10.6	2.695	Frac perm
7V	622.64	7	3		0.873	9.7	2.690	Frac perm
8V	624.60	8	2		0.007	12.5	2.701	
9H	626.70	9	3	0.370		12.4	2.746	Frac perm
9V	626.70	9	3		1.70	11.5	2.666	Frac perm
10V	628.70	10	2		0.002	7.7	2.815	
11V	630.52	11	1		45.9	15.5	2.782	Frac perm
12H	632.82	11	3	8.4		15.1	2.709	Frac perm
12V	632.82	11	3		3.5	14.1	2.728	Frac perm

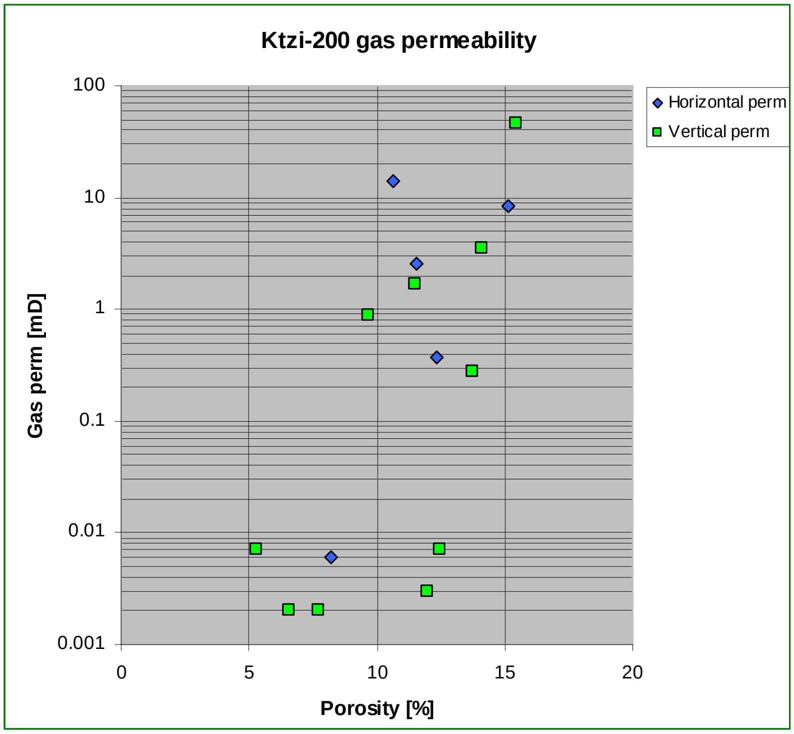
Table A1.2. Ketzin 201/2007 conventional core analysis data for uncleaned caprock material.

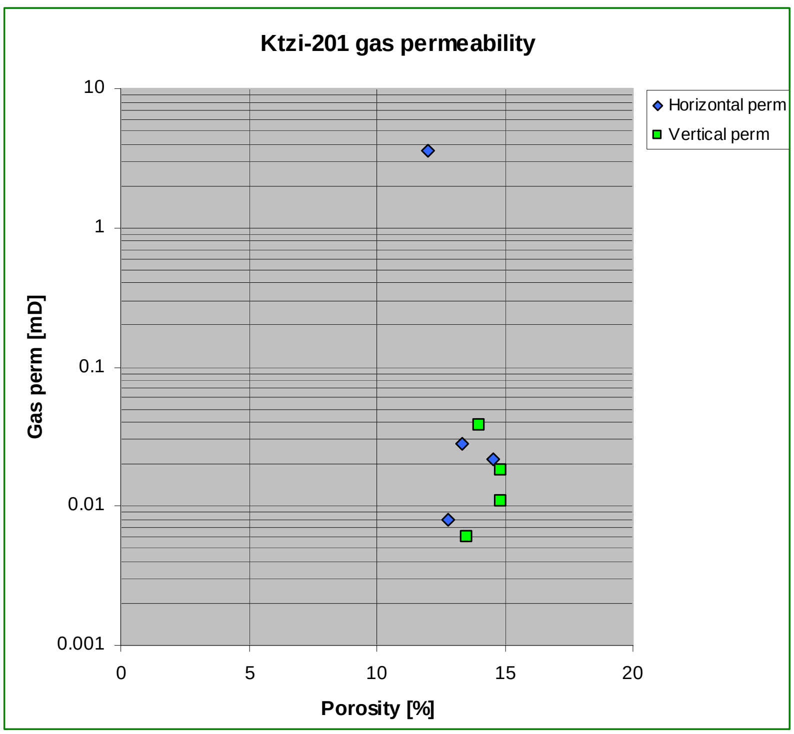
Sample Number	Core Depth (m)	Core Run	Core Section	Gas Horizontal Permeability (mD)	Gas Vertical Permeability (mD)	Ambient He-Porosity (% of Vb)	Grain Density (g/ml)	Remarks
20H	621.92	1	3	0.008		12.8	2.766	
20V	621.97	1	3		0.006	13.5	2.777	
21H	626.16	3	2	3.590		12.0	2.777	Frac perm
21V	626.15	3	2		0.038	14.0	2.749	
22H	628.09	4	2	0.028		13.3	2.740	
22V	628.03	4	2		0.018	14.9	2.741	
23H	632.33	5	3	0.022		14.6	2.733	
23V	632.32	5	3		0.011	14.8	2.738	

Analytical: Gas permeability was measured at a confining P ~ 2.8 MPa (400 psi), and at a mean N_2 gas pressure of ~ 1.5 bara (bar absolute) = 0.15 MPa (permeabilities below 0.05 mD was measured using a bubble flowmeter). He-porosity was measured unconfined.

"Frac perm" designates a gas perm reading believed to have been affected (increased) by visible fine fractures present in the plug.

Figure A1.1. Ketzin 200/2007 and 201/2007 distribution of gas permeability vs He-porosity.





Appendix 2

A2.1 Core and plug photo

Full core pieces of approx. 10 cm length was taken for the caprock study to allow plugging of a vertical and horizontal set of plugs from each core piece. The attached photos gives an impression of the material and state of plugs that was drilled from each piece. As appears the caprock material is brittle and breaks easily into bits and pieces, and not all plugs were drilled successfully. The sample identification is as follows:

core ID A-x-y : identifies a core piece from the Ktzi-200/2007 well
core ID C-x-y : identifies a core piece from the Ktzi-201/2007 well

x is the core run number and y is the core section number

Observe that most plugs shown in figure 1-16 were trimmed or cut into two pieces before being measured for porosity and permeability, and the offtrim used for grain size/mineralogy/mercury injection analysis.

Figure 1. *Plug 1H/V, core ID A-3-1, 610.70m. Variegated mudstone with flaser anhydrite.*

Figure 2. *Plug 2V, core ID A-4-1, 612.70 m. Red-brown, spotted mudstone.*

Figure 3. *Plug 3V, core ID A-4-3, 614.60 m. Grey to red-brown mudstone.*

Figure 4. *Plug 4V, core ID A-4-3, 616.60 m. Brown mudstone.*

Figure 5. *Plug 5H/V, core ID A-6-2b, 618.70 m. Grey to red-brown spotted mudstone with flaser anhydrite.*

Figure 6. *Plug 6V, core ID A-7-1a, 620.53 m. Red-brown mudstone with anhydrite spots.*

Figure 7. *Plug 7H/V, core ID A-7-3, 622.64 m. Red-brown mudstone with anhydrite spots.*

Figure 8. *Plug 8V, core ID A-8-2a, 624.60 m. Red-brown mudstone.*

Figure 9. *Plug 9H/V, core ID A-9-3, 626.70 m. Massive, dark red-brown mudstone.*

Figure 10. *Plug 10V, core ID A-10-2, 628.70 m. Light grey mudstone with flaser anhydrite.*

Figure 11. *Plug 11V, core ID A-11-1, 630.52 m. Massive, dark red-brown mudstone.*

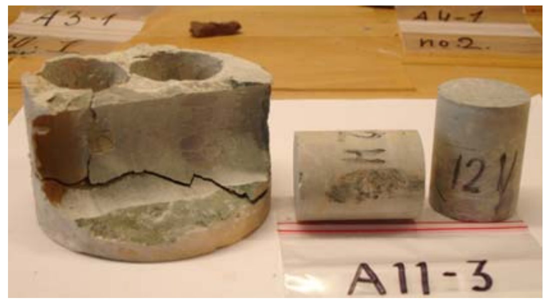


Figure 12. *Plug 12H/V, core ID A-11-3, 632.82m. Massive, variegated mudstone with anhydrite.*

Figure 13. *Plug 20H/V, core ID C-1-3, 621.9 m. Massive, dark red-brown mudstone.*

Figure 14. *Plug 21H/V, core ID C-3-2, 626.15 m. Red-brown mudstone with anhydrite.*

Figure 15. *Plug 22H/V, core ID C-4-2, 628.1 m. Variegated massive mudstone.*

Figure 16. *Plug 23H/V, core ID C-5-3, 632.32 m. Variegated anhydrite spotted mudstone.*

Appendix 3

A3.1 X-ray sample screening

Only vertical plugs were screened and only plugs that were considered potential candidates for the final caprock testing study.

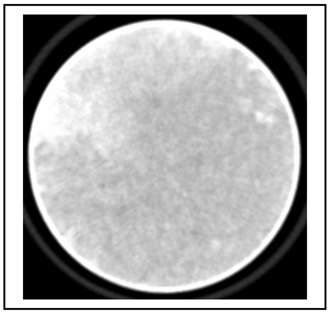
Instrumental settings:

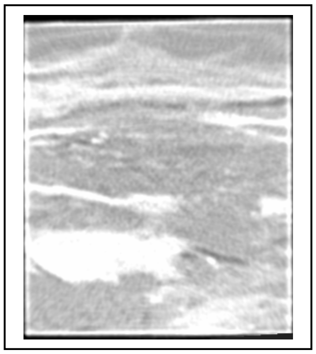
Sellar-ear	Ultra High
120 kV	330 mAs
Time=	2 s
Slice=	4 mm
Zoom=	8.0
IMG-files	16 bit signed
Width:	512
Height:	512

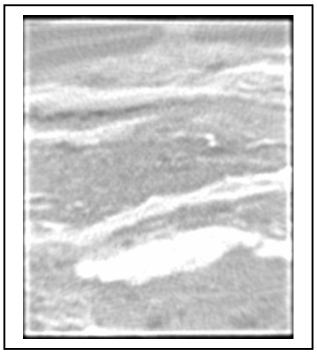
The images below are close to real plug size and represent 2 longitudinal cuts perpendicular to each other and one transversal cut through the plug sample. The thickness of each slice is 4 mm. The average grey tone figure and standard deviation for the longitudinal cuts is given in the info box; this figure is proportional to the sample porosity. The grey scale figures is in Hounsfield units; -1000 represents air (100% porosity) and appears dark on the images, +3000 is dense matrix (0% porosity) and appears white on the images.

Plug 1V Depth: 610.70 [m]

Avg. gray value	:	2494
Sdev	:	308
Porosity	:	6.6 [%]

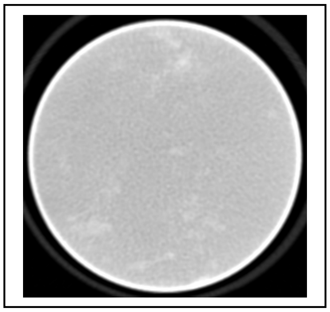


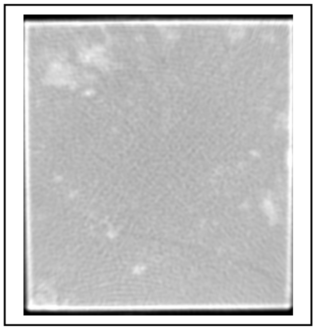


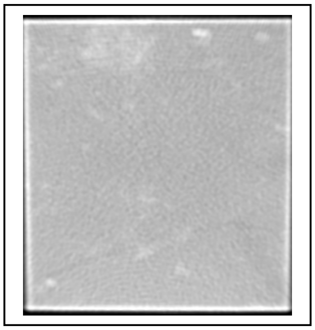


Plug 2V Depth: 612.70 [m]

Avg. gray value	:	2300
Sdev	:	101
Porosity	:	5.6 [%]

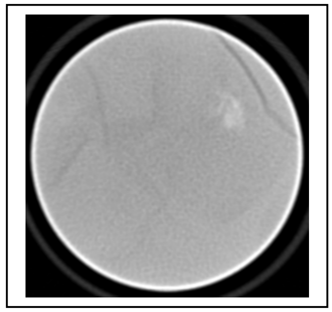






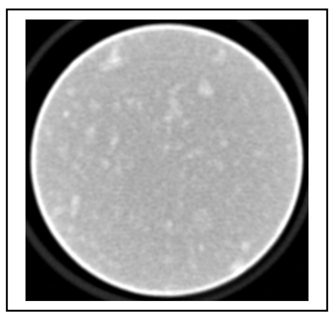
Plug 5V   Depth: 618.7 [m]

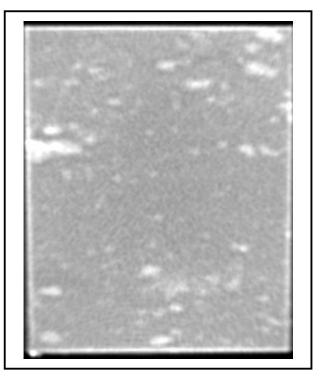
Avg. gray value	: 2042
Sdev	: 257
Porosity	: 13.7 [%]

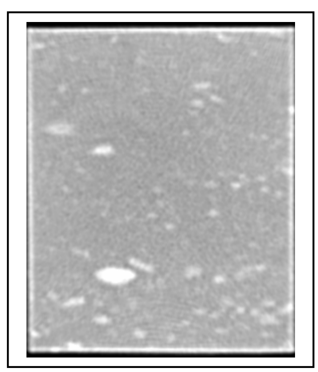


Plug 6V   Depth: 620.53 [m]

Avg. gray value	: 2173
Sdev	: 157
Porosity	: 12.0 [%]

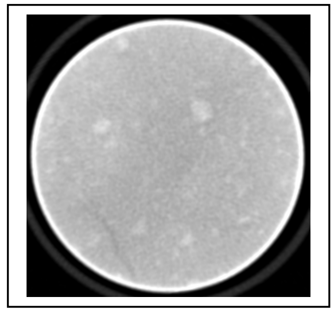


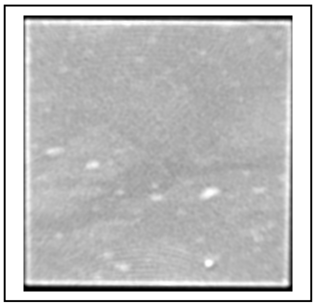


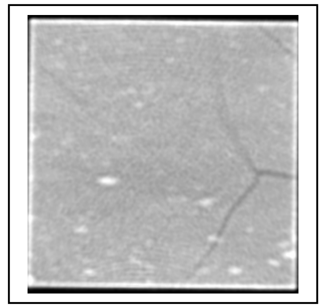


Plug 7V Depth: 622.64 [m]

Avg. gray value	:	2215
Sdev	:	108
Porosity	:	9.7 [%]

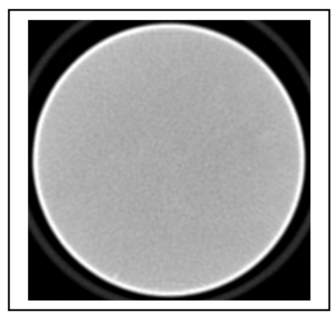


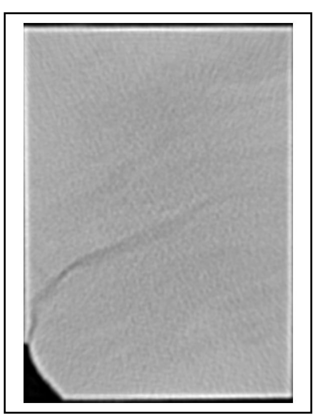


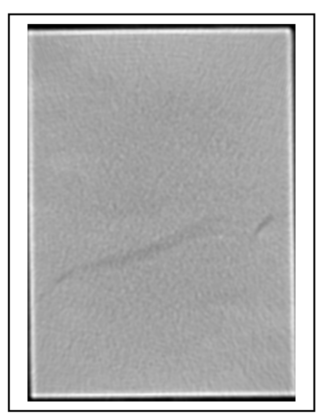


Plug 8V Depth: 624.60 [m]

Avg. gray value	:	2022
Sdev	:	72
Porosity	:	12.5 [%]

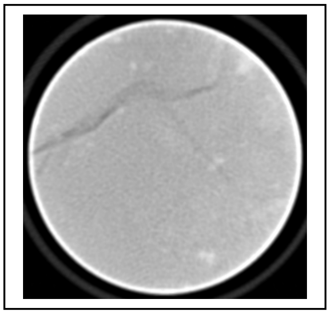


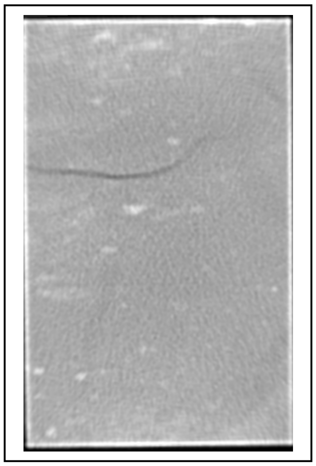


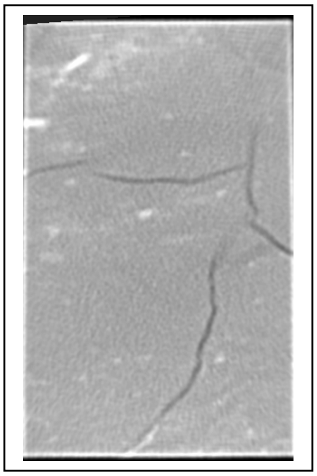


Plug 9V Depth: 626.70 [m]

Avg. gray value	:	2092
Sdev	:	126
Porosity	:	11.5 [%]

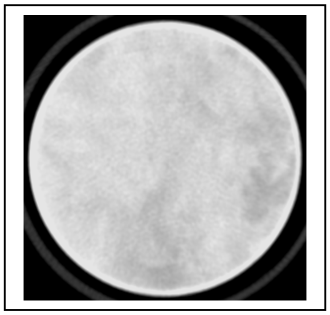


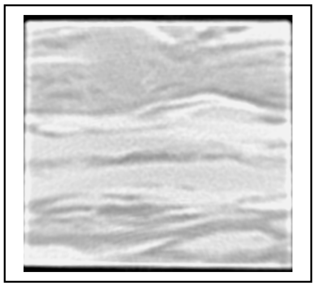


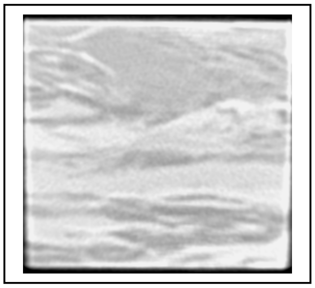


Plug 10V Depth: 628.70 [m]

Avg. gray value	:	2530
Sdev	:	308
Porosity	:	7.7 [%]

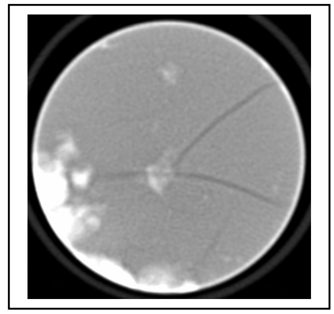


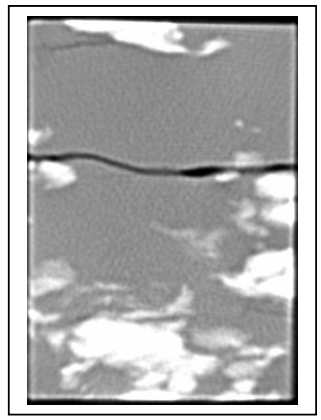




Plug 12V Depth: 632.82 [m]

Avg. gray value	: 1954
Sdev	: 390
Porosity	: 14.1 [%]

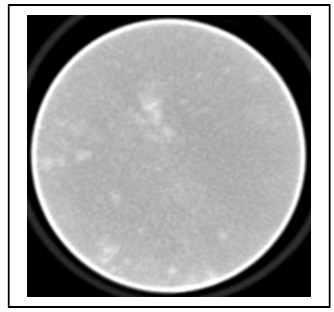


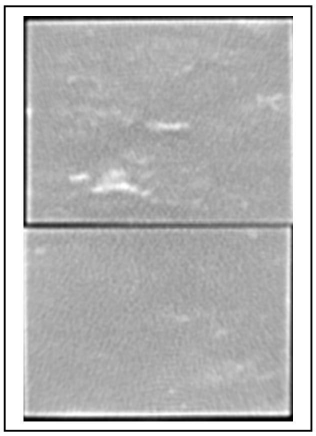


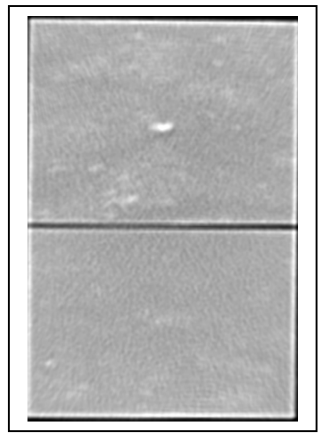
Plug 20V   Depth: 621.97 [m]

Avg. gray value	:	1954
Sdev	:	390
Porosity	:	13.5 [%]

Observe that this plug sample was split into two parts before is was CT-scanned.

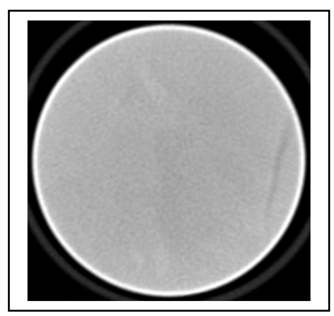


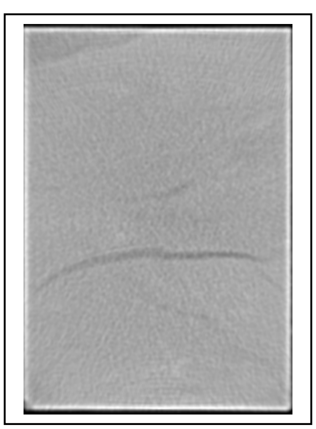




Plug 21V   Depth: 626.15 [m]

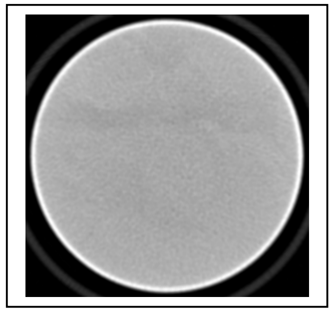
Avg. gray value	:	2016
Sdev	:	86
Porosity	:	14.0 [%]

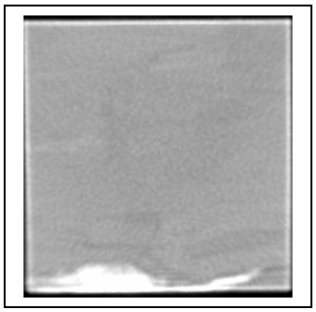


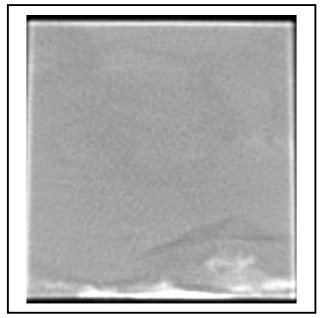


Plug 22V   Depth: 628.03 [m]

Avg. gray value	: 2042
Sdev	: 155
Porosity	: 14.9 [%]







Plug 23V   Depth: 632.32 [m]

Avg. gray value	: 2060
Sdev	: 146
Porosity	: 14.8 [%]