# Caprock seal capacity evaluation of the Fjerritslev and Børglum formations

Contribution to the EFP-project AQUA-DK

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GEOLOGICAL SURVEY OF DENMARK AND GREENLAND MINISTRY OF CLIMATE AND ENERGY

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Req. no.: 08406-579 Files: CO<sub>2</sub>AquaDK\_rep.doc Excel-files The objective of the EFP07 project "Aqua-DK" has been to evaluate the effect of  $CO_2$  sequestration on potential seal and reservoir rocks onshore Denmark. During the project a series of laboratory experiments seeks to clearify how seal and reservoir rocks behave when subjected to  $CO_2$  injection, and the observations will be used as an input to thermo-dynamic modelling.

This report presents the analytical data measured for the potential seals (caprocks) of the Børglum and Fjerritslev Formations, ie. determinations of mineralogy, grain and pore size distributions, porosity, permeability and capillary properties, data that are necessary when evaluating the seal capacity to storage of supercritical  $CO_2$  in geological traps.

## 2 Sampling

The object reservoirs within the depth interval c. 800 – 3000 m in the Danish Embayment are the Middle Jurassic Haldager Sand Formation, the Upper Triassic-Lower Jurassic Gassum Formation and the less well known Lower Triassic Bunter Sandstone Formation. The most promising seals are the mudstones of the Fjerritslev Formation (capping the Gassum Formation) and the Børglum Formation (capping the Haldager Sand Formation). These are the two caprock formations that have been the subject for sampling and mineralogical-petrophysical characterization in this study.

### 2.1 Samples

Core material from the two formations has been sampled from a number of old onshore wells, table 2.1 and 2.2 below. The location of the wells are given on the map of the Danish Basin in fig. 2.1. The sample material is generally in a poor condition, ie. dry, heavily fractured or rubble and drilling of cylindrical plugs for core analysis was seldom possible. In this respect the sample taken from the Stenlille-2 well is a remarkable exception; the caprock core section of core #2 was preserved air tight and has kept the core in a fresh condition for more than 20 years. Therefore this is the only well that can be used for a reservoir condition test of liquid permeability and threshold pressure (entry pressure) to super critical  $CO_2$ . However, most dry core material or rubble can still be used for characterization with few reservations.

Figure 2.1. Map showing structural units of the Danish Basin and well locations ao. the location of the wells sampled during the caprock study. Most wells are located in the northern part of Jylland; the Stenlille wells are not given by name on the map but found as a triple well signature just NE of the Slagelse-1 well on Sjælland. After Nielsen (2003).



Well	Core #	Box #	Formation	Lithology	Sample	MD
					ID	[m]
Fjerritslev-1	1		Børglum	grey mudstone	F1	477.00
Børglum-1	8	1		grey lam mudstone	B8	986.03
Haldager-1	100			dark grey lam mudstone	H100	1041.20
Gassum-1	31			dark grey mudstone	G31	1189.90

Table 2.1. List of samples representing the Børglum Formation from 4 different wells in the Danish Basin.

Table 2.2. List of samples representing the Fjerritslev Formation from 3 different wells in the Danish Basin.

Well	Core	Box #	Formation	Lithology	Sample	MD
					ID	[m]
Vedsted-1	# 6		Fjerritslev	grey siltst	V6-21	1403.40
				grey shaly siltst	V6-20	1407.10
	# 8	1		shaly siltst	V8-15	1865.40
		4		dark grey siltst	V8-13	1868.60
Stenlille-1	#6	2		dark shale	St-1	1501.33
Stenlille-2	#2	5		dark shale	St-2	1484.70

## 2.2 Sample preparation

Sampling of 100-150 g of core material was sufficient for all tests performed during this caprock study.

*Core analysis:* Plugs – when possible – and pieces of rubble were extracted for salt in cold methanol, and then dried at 60 °C until constant weight had been obtained. They were then analyzed for Heporosity, gas permeability (if possible) and capillary pressure by mercury injection. The plug drilled from the preserved Stenlille-2 well was not cleaned and analyzed before the reservoir condition test, but kept under simulated formation brine in a cold store until further testing could be initiated. Preparation of a useful plug was extremely difficult due to a well developed cleavage in the mudstone. The preserved core piece and plug drilled for caprock testing are shown in fig. 2.2 and 2.3.

*Mineralogy:* Plug offtrims and irregular core pieces were used in the mineralogical characterization. Samples were hand-ground to pass a 0.250 mm sieve. Samples used for grain size analysis were then dispersed by ultrasonic treatment. Chemical pre-treatment involved removal of Fe and Al oxides using  $\sim$  50 mg of dithionite per g of sample (Mehra and Jackson, 1960). Chemical pre-treatment involved removal of CaCO<sub>3</sub> using 1 M NaAC Ph 5.5.



Fig. 2.2. Stenlille-2, C#2, B#5, 1483.8 – 1484.8 m. The photo shows the lower ~ 30 cm of the core box before several attempts to cut a cylindrical plug; top of box is towards the left. A vertical  $1\frac{1}{2}$ " (38 mm) plug was cut successfully from one of the bottom pieces of full core (arrow) at 1484.7 m. The well was drilled in 1987 and the caprock section of core #2 preserved with drilling mud and simulated formation water in the glassfiber liner tube. After 23 years of preservation the core appears as fresh a the day it was received in the laboratory.





Figure 2.3. Side and top of the vertical  $\emptyset \sim 38$  mm diameter plug sample drilled for the reservoir condition caprock test. A partly open, bedding plane parallel fracture is seen at the white arrow as formation water is slowly leaking out from the fracture. The fracture does not present a problem to the caprock test because the direction of flow is perpendicular to the fracture.

# 3 Core analysis

## 3.1 Analytical

The samples were analyzed in a cleaned and dried condition according to API recommended practice for core analysis procedure (API RP 40,  $2^{nd}$  ed. 1998). Porosity and grain density was measured by the Helium injection technique, and gas permeability was measured relative to dry N<sub>2</sub> gas at a confinig stress of 2.8 MPa (400 psi) on the samples.

## 3.2 Conventional core analysis

The sample material originated from old, dry core and was mainly present as rubble or heavily fractured core in the core boxes. Therefore only a Helium porosity was measured, table 3.1 and 3.2 below. A few 25 mm (1") diameter plugs were drilled from the Fjerritslev Formation wells and measured for gas permeability, but the results should be treated with causion due to micro cracks often being present in mudrocks containing smectite and/or mixed layer minerals. This may also have affected the Helium porosity measurement where absorption of He is sometimes observed, leading to an over estimation of the porosity and grain density. On the contrary, porosity measured by mercury injection tend to under estimate the true porosity due to the inability of Hg to penetrate the very small pore throats < 3 nm at the maximum injection pressure of 4 kbar (60,000 psi). For comparison sample porosity measured by both techniques are listed in the tables below.

Well	Sample	Use	MD	Porosity <sub>(He)</sub>	Porosity <sub>(Hg)</sub>	Grain density	Gas perm
	ID		[m]	[%]	[%]	[g/cc]	[mD]
Fjerritslev-1	F1-1	Min	477.00	27.29		2.680	nd
	F1-2	Hg	477.00		20.7		nd
Børglum-1	B8-1	Min	986.03	23.07		2.718	nd
	B8-2	Hg	986.03	20.23	14.9	2.683	nd
Haldager-1	H100-1	Min	1041.20	23.23		2.746	nd
	H100-2	Hg	1041.20	22.75	14.9	2.699	nd
Gassum-1	G31-1	Min	1189.90	20.70		2.666	nd
	G31-2	Hg	1189.90		13.3		nd

Table 3.1. Børglum Formation, conventional core analysis data. The core material was in a poor condition in all wells, and only pieces of rubble could be analyzed. No gas permeability was possible.

Table 3.2. Fjerritslev Formation, conventional core analysis data. Four plug size samples could be measured for gas permeability, the remaining samples were irregular pieces.

Well	Sample	Use	MD	Porosity <sub>(He)</sub>	Porosity <sub>(Hg)</sub>	Grain density	Gas perm
	ID		[m]	[%]	[%]	[g/cc]	[mD]
Vedsted-1	V6-22	Min	1403.33	14.93		2.680	0.055
	V6-21	Hg	1403.40	15.15	11.0	2.675	
	V6-20	Min	1407.10	17.58		2.697	nd
	V6-20	Hg	1407.10		11.7		
	V8-15	Hg	1865.40	8.31	7.1	2.681	nd
	V8-14	Min	1868.23	2.99		3.354	0.022
	V8-13	Hg	1868.60	13.18	8.0	2.697	~ 0.04
	V8-12	Min	1869.27	12.13		2.692	0.060
Stenlille-1	St-1	Min	1501.33	13.1		2.730	0.021
	St-1	Hg	1501.33		9.4		
Stenlille-2	St-2	Min	1484.70	17.5		2.680	0.015
	St-2	Hg	1484.70		13.2		

#### 3.3 Mercury injection data

Aliqouts of the Børglum and Fjerritslev Formation samples were used for air-mercury, drainage capillary pressure measurement. Besides a capillary pressure curve, mercury injection data also supplies information on the pore size distribution and thereby important data for an evaluation of the caprock seal capacity. Relevant diagrams are included with Appendix 2, and data extracted from the diagrams to be used in an evaluation of the seal capacity are shown in table 3.3.

Table 3.3. Seal capacity data read from mercury capillary pressure measurements on 10 samples from the Børglum and Fjerritslev Formations;  $r_{10}$  and  $r_{50}$  denotes the pore throat radius in nm (nano meter) where 10% and 50% respectively of the sample pore volume have been filled by injected mercury.

Well	Sample	Formation	MD	Porosity <sub>(Hg)</sub>	Entry P <sub>(air-brine)</sub>	Pore	radius
	ID		[m]	[%]	[bar]	r <sub>50</sub> [nm] r <sub>10</sub>	
Fjerritslev-1	F1-2	Børglum	477.00	20.7	40	21	31
Børglum-1	B8-2		986.03	14.9	35	16	36
Haldager-1	H100-2		1041.20	14.9	30	20	45
Gassum-1	G31-2		1189.90	13.3	75	10	18
Vedsted-1	V6-21	Fjerritslev	1403.40	11.0	60	10	38
	V6-20		1407.10	11.7	75	9	23
	V8-15		1865.40	7.1	110	5	150
	V8-13		1868.60	8.0	95	6	300
Stenlille-1	St-1		1501.33	9.4	65	9	200
Stenlille-2	St-2		1484.70	13.2	75	8	600

From inspection of the diagrams in Appendix 2 it is obvious that each formation has its own characteristic "Hg-fingerprint". To examplify the general trends selected diagrams from each formation are shown below in fig. 3.1.

The Børglum Fm samples have a sharp entry at a high pressure of ~ 2000 psi in the air/mercury capillary pressure plot and a narrow, uni-modal pore throat size distribution, pore throats exceeding 100 nm in radius are simply not present in the mudstones.

The Fjerritslev Fm samples have a gradual entry starting below 100 psi in the air/mercury capillary pressure plot and a bi-modal pore throat size distribution; a stable plateau is not reached until filling of ~ 20% of the pore volume representing the largest pore throat radii. However, it should also be observed that the majority of pores are very small with pore throat radii below 10 nm.

The difference between the two formations are also seen from table 3.3 above with the  $r_{10}$  radius generally being much greater for the Fjerritslev Fm due to the presence of the large pores in the upper tail of the mercury injection pore size diagrams.



Figure 3.1. Mercury injection diagrams characteristic for the two different pore systems, the Børglum Fm mudstones (left hand side) and the Fjerritslev Fm silty mudstones (right hand side).

#### injection of two fluids or a quick swap between fluids right against the end face of the plug. The core holder and piston cylinder with CO<sub>2</sub> is contained within a temperature controlled oven, fig. 3.3. Data on flow, volume, pressure and temperature vs. time are continously recorded during the experiment. In experiments with no back pressure a PC controlled analytical balance (Mettler®) was connected to the downstream end of the core holder. This allowed a very good control of material balance (fluid input / output data). In experiments at true reservoir conditions using scCO<sub>2</sub> one cylinder pair delivered the opstream pressure and the other cylinder pair controlled the (downstream) back pressure.

detection limits to between 30 and 40 MPa at the pore pressure and overburden pressure lines. An overview of the rig is shown in fig. 3.2. A computer controlled 5 cylinder metering pump (Quizix® SP5000 series) delivers a fluid flow at a controlled flow rate or pressure, and also controls the confining stress on the core holder. A heavy duty hydrostatic core holder (Core Laboratories®) able to handle 38 mm diameter plug samples is used in the study. Two needle valves in the upstream end piece allows

An experimental rig capable of handling pressures to 70 MPa is available and tested for leaks and

Table 3.4. Chemical composition and physical properties of the simulated formation water used in the present study. Data originates from Laier (2008), except density and viscosity data that was measured by GEUS Core Laboratory.

#### 3.4 Reservoir parameters

The following settings were used for the caprock test of the Stenlille mudstone sample:

Depth	: 1484.0 m
Total vertical stress	: 336 bar (33.6 MPa)
Pore pressure	: 168 bar (16.8 MPa)
Effective stress	: 168 bar (16.8 MPa)
Poisson's ratio (assumed)	: 0.3
Net effective laboratory hyd	lrostatic stress during testing : 105 bar (10.5 MPa)
Reservoir temperature	: 50 °C

Hydrostatic conditions was assumed for the reservoir and caprock before storage of natural gas.

#### 3.5 Reservoir fluid properties

During the dynamic experiment a simulated formation water composition for the Stenlille caprock have been used to saturate the sample and measure liquid permeability, table 3.4. The experiment ran at the reservoir temperature 50 °C; at this temperature CO<sub>2</sub> is above the critical point if the pressure

exceeds 7.4 MPa, ie. it is a supercritical fluid. Below 7.4 MPa CO<sub>2</sub> is a gas. At room conditions the interfacial tension (IFT) for the system  $CO_2$  – water is close to the IFT for the system air – water, approximately 72 mN/m. As pressure increases the IFT for a CO<sub>2</sub> -brine system falls rapidly and reaches a plateau at approx. 30 mN/m above 15 MPa pore pressure. The IFT increases slightly with increasing temperature and salinity. IFT data used in this study has been interpolated from Chalbaud et al. (2006).

Element	Concentration
	mg/l
Na total	58000
K+	370
Mg2+	1640
Ca2+	8600
Sr2+	650
Ba2+	
CI-	110260
HCO3-	70
TDS:	179590
Density:	1.117 g/ml @ 25⁰C
	1.106 g/ml @ 50⁰C
Viscosity:	0.82 cP @ 50°C

3.6 Rig design





Fig. 3.3. The experimental setup of the oven with the  $CO_2$  pressure cylinder in the foreground and the vertical core holder in the back-ground.

Fig. 3.2. Sketch showing the major components in the  $CO_2$  reservoir condition rig. The pumping system is a 5 cylinder computer controlled pump with one cylinder pair controlling the upstrean flow / pressure, and one cylinder pair controlling the downstream flow / back pressure under reservoir condition tests. The last cylinder controls the sample confining stress. Under effective stress conditions (ambient pore pressure) the down-stream line is connected to an electronic balance and serves as an independent material balance check on pump readings.

### 3.7 Experimental design

A conventional cap rock test is performed by increasing the upstream pore pressure of a non-wetting phase (normally oil or gas), or decreasing the downstream pressure until the threshold or entry pressure is exceeded, and the wetting phase (normally water) is displaced. The threshold pressure point is detected by fluid starting to move into the sample upstream side, and by observing wetting fluid being produced from the downstream side of the sample. Eventually a breakthrough will occur, and the non-wetting phase will be observed downstream.

The following experiment plan for the preserved caprock sample from the Stenlille-2 well has been decided for:

- 1. Determination of sample creep and porosity reduction at effective stress conditions
- 2. Determination of liquid permeability at effective stress conditions
- 3. Entry pressure to nitrogen gas (N<sub>2</sub>)
- 4. Entry pressure to carbon dioxide gas (gasCO<sub>2</sub>)
- 5. Entry pressure to super critical carbon dioxide fluid (scCO<sub>2</sub>)

Comments: liquid permeability is measured because there is a general lack of reliable data for very low permeability materials like clays and mudstones; entry pressure to N<sub>2</sub> is measured because the surface property product (surface tension × contact angle) is well known for the system N<sub>2</sub> – fluid – rock and this allows some comparisons to be drawn for the later measured entry pressures in the systems  $gasCO_2 / scCO_2 - fluid - rock$ , where data are still needed for different salinities, temperatures and pressures. Conversion data derived from such experiments may be later used in translations of mercury injection data to the relevant fluid systems.



Fig. 3.4. Liquid expel curve recorded on the electronic balance during loading of the caprock test plug from the Stenlille-2 well.

Legend: Pump 3 vol expl. – liquid volume delivered from the pump cylinder applying stress to the core holder. Rec downstream – liquid expelled from the sample and received on the electronic balance, recalculated to a volume

Conf P – confining stress applied to the core holder

Temp - temperature recorded in the oven during the experiment

Poly – 6<sup>th</sup> order polynominum fitted to the balance readings to help calculate the pore volume

#### 3.8 Porosity reduction

A hydrostatic confining stress of 105 bar was applied to the plug during a period of 8 hours and the setup left to settle for a total of ~ 17000 minutes (12 days), Fig. 3.4. From the liquid expel curve a pore volume reduction of 0.93 ml was calculated; creep was very low and asymptotically approaching zero because the consolidated Fjerritslev Fm mudstone is not a rate type material. The calculated porosity reduction at reservoir conditions is given in Table 3.5

#### 3.9 Liquid permeability

The Stenlille caprock sample turned out to be very tight and precise measurement of the liquid permeability was near the limit for the experimental setup. Pore pressure (Upstream P in Fig. 3.5) was applied in 3 steps of each 10 bar. The response of the formation brine injected upstream is clearly visible from the diagram at 20 and 30 bar upstream P. However, the liquid leaving the sample downstream is lagging somewhat behind – a hysteresis due to the very low permeability. This means that the sample expands upstream. This is followed by a stress relief in the top of the sample, eg. at 30 bar upstream P, the net effective stress in the top of the sample is no longer 105 bar, but 75 bar. In the downstream end of the sample the pore pressure is at ambient conditions, and therefore the effective confining stress is still 105 bar. The liquid permeability given in Table 3.5 is thus the permeability calculated from the balance data and valid for the downstream end of the sample that has been kept constant under 105 bar effective stress.



Fig. 3.5. Pressure and volume data recorded during the liquid permeability test. Perfect equilibrium data was never obtained although the test continued beyond 25000 minutes (19 days).
 Legend: Upstream P – pore pressure delivered from the metering pump
 Inj. Upstream – injected upstream from the metering pump

Refer to fig. 3.4 above for additional abbreviations.

Liquid permeability data calculated from the injected upstream pumping data is expected to overestimate the permeability, and this is exactly what we find, Table 3.5.

#### 3.10 Entry pressure to gasses

After finishing the liquid permeability programme, the upstream pore pressure lines were swopped to gasses  $N_2$  and later  $CO_2$ , and the input pressure increased stepwise to determine the entry pressure point. An estimate of the entry pressure for the two different gasses can be obtained from the mercury

injection data measured earlier. Airmercury data have been transformed to the gas systems using recent data for interfacial tension (IFT) in the N<sub>2</sub>brine and CO<sub>2</sub>-brine systems, Fig. 3.6. It should be observed, however,

Fig. 3.6. Air-mercury capillary pressure diagram for the Stenlille-2 caprock sample converted to gas/brine systems @ 50 bar and 50 °C:

 $IFT N_2$ /brine = 66 mN/m IFT gasCO<sub>2</sub>/brine = 46 mN/m

IFT data from Yan et al. (2001) and Chalbaud et al. (2006).





Fig. 3.7. Entry pressure to  $N_2$  is observed at 80 bar upstream pore pressure. Approx. 2000 min later breakthrough (BT) is recorded downstream. Observe that the 2 ml's produced from entry P to BT of  $N_2$  gas is due to gas blow down of the liquid standing in the downstream tubing to the electronic balance, and not an expression for the liquid that was drained from the sample in the core holder. Legend: N2 P – Nitrogen pore pressure delivered from the metering pump



Fig. 3.8. Entry pressure to gas  $CO_2$  is observed at 70 bar upstream pore pressure. This is seen from the upwards kink on the downstream liquid production curve.

Legend: CO2 cyl P – pore pressure delivered from the metering pump via a  $CO_2$  pressure cylinder, cf. Fig 3.3

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that data are only approximate as the exact IFT data for the reservoir temperature and salinity of the Stenlille brine cannot be found from tables or publications, they have to be measured under live conditions in the laboratory. From Fig. 3.6 the entry pressures for the gas-brine systems are expected at ~ 80 bar and ~ 60 bar for the N<sub>2</sub>- and CO<sub>2</sub>-brine systems. From Fig. 3.7 it is clearly seen that entry in the N<sub>2</sub>-brine system in fact occur at 80 bar upstream pressure, but only after a considerable waiting time of 600 min. In the gasCO<sub>2</sub>-brine system the entry does not occur until ~70 bar upstream pressure, Fig. 3.8, ie. the IFT used in the conversion of the air-mercury system, Fig. 3.6, is not valid for the Stenlille case. This is not unexpected as the salinity of the Stenlille brine is significantly higher than the range of salinities covered by the Chalbaud et al. (2006) paper, and besides is not a pure NaCl-brine.

## 3.11 Entry pressure to super critical CO<sub>2</sub>

The experimental rig was now changed to reservoir pressure conditions, ie. the pore pressure was increased to 168 bar upstream as well as downstream, and the confining stress was set at 168 + 105 = 273 bar. This means that the electronic balance was disconnected from the setup, and the computer controlled high pressure pump set to control the downstream back pressure in constant pressure mode (= 168.0 bar). The upstream pore pressure was changed from gasCO<sub>2</sub> to super critical CO<sub>2</sub> (scCO<sub>2</sub> as we are well above the critical point in the CO<sub>2</sub> phase diagram), and the pressure increased stepwise until withdrawal of liquid from the downstream side of the sample could be detected from the high-pressure pump, Fig. 3.9. The following observations can be made: the long period at a differential pressure dP = 45 bar (~ 1000 min) causes very little invasion of scCO<sub>2</sub>, presumably confined to the minor number of "large" pores seen to be present from the mercury injection diagrams. At dP = 50 bar the rate of invasion increases clearly and this is the entry pressure for the scCO<sub>2</sub> -brine system in the Stenlille caprock. At dP = 60 bar there is a permanent withdrawal of liquid from the downstream side of the sample, indicating that fluid BT is about to occur within days or weeks, not months.



Fig. 3.9. Entry pressure to  $scCO_2$  is observed at 50 bar upstream pore pressure. BT will occur at 60 bar after an extended period of time. This is seen from the downstream liquid withdrawal curve. Legend: Cyl#2B, vol – withdrawal volume recorded from the high-pressure metering pump.

Capillary entry / break-through data for the gas and fluid pairs are listed in Table 3.5 below.

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Table 3.5. Special core analysis data measured for the Fjerritslev Fm mudstone caprock from the Stenlille-2 well. Data measured at initial room conditions and at reservoir conditions, ie. 50 °C and 168 bar effective confining stress (equal to 105 bar hydrostatic laboratory pressure).

Sample	Depth	Li	Di	BVi	PVi	dPV <sub>rescon</sub>	initial Ø	rescon
ID	[m]	[cm]	[cm]	[cm <sup>3</sup> ]	[cm <sup>3</sup> ]	[cm <sup>3</sup> ]	[%	6]
St-2/1A	1484.70	2.99	3.77	33.52	5.44	0.93	16.2	13.8
Sample	ple Liq. perm @ rescon		N <sub>2</sub>		gasCO <sub>2</sub>		scCO <sub>2</sub>	
ID	[nD]		Pce [bar] P <sub>BT</sub>		Pce [bar] P <sub>BT</sub>		Pce [bar] P <sub>BT</sub>	
St-2/1A	2.6	~ 3 *	80	80	70	nd	50	60
Legend: L D B <sup>N</sup> P <sup>N</sup> dF	: ler : dia V : bu V : po PV : po	ngth ameter Ik volume re volume re volume re	P : Subscripts:	fluid pressu i = initial (ro rescon = res ce = capillal	re oom conditio servoir cona ry entry P	ons) litions		
Ø	: po	rosity	(-D) (0 <sup>9</sup>	D	-212	BT = break-	through P	

Liquid permeability unit: nano Darcy  $[nD] = 10^9$  Darcy  $\approx 10^{21}$  m<sup>2</sup> \* liquid permeability read from the metering pump

## 4.1 Analytical

After sample disintegration the grain size distribution was obtained from a particle size centrifuge (Slater and Cohen, 1962). The coarse and fine clay (size fractions 2-0.2  $\mu$ m and <0.2  $\mu$ m respectively) were investigated by X-ray diffraction (XRD) using a Philips 1050 instrument with pulse-height selection and  $\beta$ -filtered Co-K<sub> $\alpha$ </sub> radiation. Oriented specimens were prepared by the pipette method as follows:

Mg-saturated air dry Mg-saturated and glycolated (exposed to glycol vapour for three days at 60°C) Mg-saturated and glycerol treated (glycerol added to the suspension) K-saturated, air-dry and after heating for 1 hour at 300°C

No attempt was made to quantify the mineral phases because the degree of similarity between the sample minerals and available standard clay minerals is unknown. The identification of smectite and vermiculite is according to the recommendations of MacEwan and Wilson (1980). Peaks of ordered mixed-layer minerals were not significant. Less ordered mixed-layer minerals are difficult to identify in the presence of the large amounts of illite and chlorite in the samples. This identification and quantification requires that well-crystalline minerals are removed and diffraction patterns are modelled.

Specific surface area and pore volume was determined from adsorption isotherms measured on a Coulter SA 3100 gas adsorptometer using liquid N<sub>2</sub>. The samples were outgassed at room temperature until stable vacuum. The specific surface area was calculated according to the BET model (Brunauer et al., 1938). Gurvitsch pore volume in cm<sup>3</sup>/g dry sample was calculated from the adsorption isotherm by the amount of liquid gas adsorbed at a relative pressure of  $P/P_0=0,9$ .

Carbonate content was determined as the amount of calcite by titration.

## 4.2 Results

The granulometric analysis showed that hardly any sand-size grains are present in the two formations and that they both contain between  $\frac{1}{3}$  and  $\frac{2}{3}$  silt-size grains; Figures 4.1 and 4.2. In the sense of Lundegaard & Samuels (1980) they are classified as mudstones (non-laminated) or mudshales (laminated). However, there are distinct differences between the two formations. The Børglum Formation samples are slightly calcareous, contain more quartz and clay size grains, especially fine clay than seen for the Fjerritslev Formation, and this is also reflected in the specific surface area data (N<sub>2BET</sub>) of 30-40 m<sup>2</sup>/g for the Børglum Formation samples vs 25-35 m<sup>2</sup>/g for the Fjerritslev Formation samples, Table 4.1 and 4.2.

Clay mineral identification has been carried out for 4 samples, two samples representing the Børglum Fm and two samples from the Fjerritslev Fm; clay XRD-diagrams are shown in Appendix 1.

The Børglum Formation: The coarse clay fraction is in both Fjerritslev-1 and Haldager -1 composed of large amounts of illite and kaolinite and medium amounts of smectite and vermiculite, with small amounts of chlorite. However, in the Haldager-1 the relative proportion of kaolinite is larger compared to in the Fjerritslev-1, whereas illite is present in larger amounts in Fjerritslev-1 compared to Haldager-1. The fine-clay fraction is in Haldager-1 composed of large amounts of smectite and kaolinite with minor amounts of illite and chlorite, and in Fjerritslev-1 of large amounts of smectite, medium amounts of kaolinite and minor amounts of illite and chlorite.

Table 4.1. Børglum Formation, grain size and gas absorption data. Observe the high quartz content ~30 % of the bulk rock, and the high clay size fraction of 40-50 wt-% that explains the high specific surface area  $(N_{2BET})$  ~30-40 m<sup>2</sup>/g. G pores is the pore volume confined to pores  $\leq$  20 nm (Gurvitsch pore volume).

Sample	> 63	<u>63 – 20</u>	20 – 4	4 – 2	2 – 0.2	< 0.2	Σ	$\Sigma$ Clay	Quartz	N <sub>2BET</sub>	G pores
ID	μm	μm	μm	μm	μm	μm	wt-%	wt-%	%	m²/g	cm <sup>3</sup> /g
F1-1	1	3	36	12	14	32	97	46	30	40	0.047
G31-1	0	1	41	8	15	34	99	49	35	37	0.048
B8-1	3	12	37	9	17	21	99	38	25	23	0.027
H100-1	0	4	37	14	21	21	98	42	25	29	0.033



Figure 4.1. Histogram showing the grain size distribution for Børglum Formation mudstones sampled from 4 different wells. Observe the similarity among the samples contrary to the geographical distance between the wells.

The Fjerritslev Formation: The coarse clay fraction in Vedsted-1 is dominated by kaolinite, and illite, vermiculite, chlorite, and mixed-layer illite-smectite are present in small amounts. In Stenlille-2, the coarse clay consists of large amounts of smectite, medium amounts of kaolinite, and small amounts of illite and chlorite. The fine-clay fractions have in both Vedsted-1 and Stenlille-2 the same compositions as the corresponding coarse-clay fractions.

Minor amounts of carbonate was observed from the total XRD scans of the Børglum Fm samples, and the content was determined by titration, Table 4.3. Trace amounts of carbonate may be present also in the Fjerritslev Fm samples but no attempt was made to determine it.

Table 4.3. Determination of carbonate as amount of  $CaCO_3$  from the Børglum Fm samples.

Sample ID CaCO<sub>3</sub> [%]

F1-1	17
G31-1	5.5
B8-1	7.8
H100-1	7.9

Table 4.2. Fjerritslev Formation, grain size and gas absorption data. Observe the moderate quartz content ~20 % of the bulk rock, and the reduced clay size fraction of 20-35 wt-% that explains the lower specific surface area ( $N_{2BFT}$ ) ~30 m<sup>2</sup>/g. G pores is the pore volume confined to pores  $\leq$  20 nm (Gurvitsch pore volume).

Plug	> 63	<u>63 – 20</u>	20 – 4	4 – 2	2 - 0.2	< 0.2	Σ	$\sum$ Clay	Quartz	N <sub>2BET</sub>	G pores
ID	μm	μm	μm	μm	μm	μm	wt-%	wt-%	%	m²/g	cm <sup>3</sup> /g
St-1	0	29	34	13	11	11	98	22	18	22	0.023
St-2	0	8	40	14	17	18	97	35	19	35	0.035
V6-22	3	39	31	6	10	9	98	19	24	26	0.026



Figure 4.2. Histogram showing the grain size distribution for Fjerritslev Formation mudstones sampled from 3 different wells. Observe the greater variability among the samples, less clay size grains and more coarse silt fraction grains (compared to the Børglum Fm samples).

New and unexpected data from the core analysis and mineralogical characterization of potential caprocks in the Danish Basin has demonstrated that the Børglum Formation has very good sealing properties. Based on the results from the analysis of grain size distribution and mineralogy, and the late entry in the mercury injection diagrams, the Børglum Formation should be expected to be superior to the Fjerritslev Formation as a caprock. That is however not the case, mainly because of :

- the porosity of the Fjerritslev Formation is significantly lower than the porosity of the Børglum Formation, i.e. it is a tighter rock
- although an early entry is seen from the mercury injection diagrams, the majority of pore throats are very small in the Fjerritslev Formation, presumably because of a deeper burial and stronger diagenetic imprint on the Fjerritslev Formation.

The caprock test on a fresh state sample of the Fjerritslev Fm in Stenlille demonstrated excellent sealing properties of a quality normally only found for hydrocarbon seals. However, only one sample was tested and, considering the variability among the Fjerritslev Fm samples, results may not be unconditionally valid for the Fjerritslev Fm in the whole Danish Basin area.

A nominal seal capacity evaluation has been attempted for two different scenarios 1) the Børglum Formation case with CO<sub>2</sub> storage in 1000 m depth, and 2) the Fjerritslev Formation case (Vedsted, Stenlille) with CO<sub>2</sub> storage in 1500 m depth (table 5.1). Reservoir parameters have been taken from the present study and recent data from the litterature on interfacial tension (IFT) and density ( $\rho$ ) of supercritical CO<sub>2</sub>. Allowing for some variation in entry pore throat radius, the results demonstrate that both caprock formations will hold a CO<sub>2</sub> column of several hundred meters height, i.e. they have excellent sealing properties. The findings of the reservoir condition test performed on the Stenlille caprock can be compared with scenario 2) in the table below if it is assumed that the "effective pore throat radius r" is approx. 10 nano meter [nm].

Table 5.1. Evaluation of the seal capacity for a hypothetical storage site below a Børglum Formation caprock (1000 m depth) and below a Fjerritslev Formation caprock (1500 m depth). Observe that a doubling of the pore throat radius 'r' will halve the height ' $H_{max}$ ' of a potential column of supercritical CO<sub>2</sub> that can be safely stored below the caprock. It is assumed that the fluid-rock system is waterwet \*.

Depth	Pore P	ρ <sub>w</sub> (brine)	ρ <sub>nw</sub> (CO <sub>2</sub> )	IFT	'r' (radius)	P <sub>ce</sub> (entry P)	H <sub>max</sub> *
[m]	[MPa]	[kg/m <sup>3</sup> ]	[kg/m <sup>3</sup> ]	[mN/m]	[nm]	[MPa]	[m]

1000	10	1085	713	33	10	6.6	1809
					20	3.3	904
1500	17.2	1106	744	30	5	12	3379
					10	6	1690

Legend:

 $\rho_w$  = density of wetting phase (brine);  $P_{ce}$  = capillary entry pressure,  $\rho_{nw}$  = density of non-wetting phase (gas)

determined from :

r = pore radius  $\sigma$  = interfacial tension (IFT)

 $P_{ce} = \frac{2\sigma}{r}$  [Pa]

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X-ray diffractograms (XRD) recorded for the clay size fractions of mudstones of the Børglum and Fjerritslev Formations. For indexing of reflection peaks refer to table A.1 below.

Table A.1. Discrete mineral peak positions in the attached XRD diagrams can be identified from the table.

Mineral	Glycol/glycerol	Mg-air	K-300°C
Illite	10Å, 5Å	10Å, 5Å	10Å, 5Å
Smectite	17Å/18Å	14Å	10Å
Vermiculite	14Å	14Å	10Å
Chlorite	14Å, 7Å, 4.7Å, 3.5Å	14Å, 7Å, 4.7Å, 3.5Å	14Å, 7Å, 4.7Å, 3.5Å
Kaolinite	7Å, 3.5Å	7Å, 3.5Å	7Å, 3.5Å

Positions of scattering intensities of the less ordered mixed-layer minerals are:

Mineral	Glycol/glycerol	Mg-air	K-300°C

Illite-smectite	5-6Å	10-14Å	10Å
Chlorite-vermiculite	14Å	14Å	10-14Å
Chlorite-smectite	14-18Å	14Å	10-14Å





Core Laboratory



25



4000



26





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Mercury injection drainage capillary pressure data measured on cleaned and dried samples of the Børglum and Fjerritslev formations.

#### Well : Fjerritslev-1 Børglum Fm GEUS Core Lab and SKM Services, 18.08.2010

Plug ID	F1-2	
Sample Depth	477	[m]
Plug Permeability (Air)	n/a	[mD]
Plug Porosity (He)	0,279	[fraction]
Injection Sample Porosity	0,207	[fraction]
Injection Sample Pore Vol	0,503	[cc]
Injection Sample Bulk Vol	2,430	[cc]
Injection Sample Weight	5,120	g
Mean Hydraulic Radius	0,012	[µm]
Swanson's Parameter	0,002	
FZI		









#### Well : Fjerritslev-1 Børglum Fm

GEUS Core Lab and SKM Services, 18.08.2010

Plug ID	F1-2	
Sample Depth	477	[m]
Plug Permeability (Air)	n/a	[mD]
Plug Porosity (He)	0.279	[fraction]
Injection Sample Porosity	0.207	[fraction]
Injection Sample Pore Vol	0.503	[cc]
Injection Sample Bulk Vol	2.430	[cc]
Injection Sample Weight	5.120	g
Mean Hydraulic Radius	0.012	[µm]
Swanson's Parameter	0.002	
FZI		







#### Well : Børglum-1 Børglum Fm GEUS Core Lab and SKM Services, 29.06.2010

Plug ID	<b>B8-2</b>	
Sample Depth	986,03	[m]
Plug Permeability (Air)	n/a	[mD]
Plug Porosity (He)	0,202	[fraction]
Injection Sample Porosity	0,149	[fraction]
Injection Sample Pore Vol	0,277	[cc]
Injection Sample Bulk Vol	1,859	[cc]
Injection Sample Weight	4,220	g
Mean Hydraulic Radius	0,015	[µm]
Swanson's Parameter	0,001	
FZI		
Injection Sample Bulk Vol Injection Sample Weight Mean Hydraulic Radius Swanson's Parameter FZI	1,859 4,220 0,015 0,001	[cc] g [µm]









Core Laboratory

#### Well : Børglum-1 Børglum Fm GEUS Core Lab and SKM Services, 29.06.2010

Plug ID	B8-2	
Sample Depth	986.03	[m]
Plug Permeability (Air)	n/a	[mD]
Plug Porosity (He)	0.202	[fraction]
Injection Sample Porosity	0.149	[fraction]
Injection Sample Pore Vol	0.277	[cc]
Injection Sample Bulk Vol	1.859	[cc]
Injection Sample Weight	4.220	g
Mean Hydraulic Radius	0.015	[µm]
Swanson's Parameter	0.001	
FZI		





#### Well : Haldager-1 Børglum Fm GEUS Core Lab and SKM Services, 29.06.2010

Plug ID	H100-2	
Sample Depth	1041,20	[m]
Plug Permeability (Air)	n/a	[mD]
Plug Porosity (He)	0,147	[fraction]
Injection Sample Porosity	0,149	[fraction]
Injection Sample Pore Vol	0,399	[cc]
Injection Sample Bulk Vol	2,677	[cc]
Injection Sample Weight	5,980	g
Mean Hydraulic Radius	0,017	[µm]
Swanson's Parameter	0,001	
FZI		







2000 1600 1600 1200 1200 1000 800 600 400 200 0 1 0,8 0,6 0,4 0,2 0 Mercury Saturation (fraction)

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GEUS

## Well : Haldager-1 Børglum Fm

GEUS	Core	Lab	and	SKM	Services,	29.06.2010

Plug ID	H100-2	
Sample Depth	1041.20	[m]
Plug Permeability (Air)	n/a	[mD]
Plug Porosity (He)	0.147	[fraction]
Injection Sample Porosity	0.149	[fraction]
Injection Sample Pore Vol	0.399	[cc]
Injection Sample Bulk Vol	2.677	[cc]
Injection Sample Weight	5.980	g
Mean Hydraulic Radius	0.017	[µm]
Swanson's Parameter	0.001	
FZI		







#### Well : Gassum-1 Børglum Fm GEUS Core Lab and SKM Services, 18.08.2010

Plug ID	G31-2	
Sample Depth	1189,90	[m]
Plug Permeability (Air)	n/a	[mD]
Plug Porosity (He)	0,207	[fraction]
Injection Sample Porosity	0,133	[fraction]
Injection Sample Pore Vol	0,383	[cc]
Injection Sample Bulk Vol	2,871	[cc]
Injection Sample Weight	6,470	g
Mean Hydraulic Radius	0,006	[µm]
Swanson's Parameter	0,001	
FZI		



Air/Brine system [bar]





1000



1

0,8

0000

## Well : Gassum-1 Børglum Fm

GEUS	Core	Lab	and	SKM	Services,	18.08.2010

Plug ID	G31-2	
Sample Depth	1189.90	[m]
Plug Permeability (Air)	n/a	[mD]
Plug Porosity (He)	0.207	[fraction]
Injection Sample Porosity	0.133	[fraction]
Injection Sample Pore Vol	0.383	[cc]
Injection Sample Bulk Vol	2.871	[cc]
Injection Sample Weight	6.470	g
Mean Hydraulic Radius	0.006	[µm]
Swanson's Parameter	0.001	
FZI		







#### Well : Stenlille-1 Fjerritslev Fm GEUS Core Lab and SKM Services, 04.09.2009

Plug ID	St-1	
Sample Depth	1501,33	[m]
Plug Permeability (Air)	0,021	[mD]
Plug Porosity (He)	0,136	[fraction]
Injection Sample Porosity	0,094	[fraction]
Injection Sample Pore Vol	0,238	[cc]
Injection Sample Bulk Vol	2,525	[cc]
Injection Sample Weight	6,050	g
Mean Hydraulic Radius	0,760	[µm]
Swanson's Parameter	0,006	
FZI	0,143	









#### Well : Stenlille-1 Fjerritslev Fm GEUS Core Lab and SKM Services, 04.09.2009

Plug ID	St-1	
Sample Depth	1501,33	[m]
Plug Permeability (Air)	0,021	[mD]
Plug Porosity (He)	0,136	[fraction]
Injection Sample Porosity	0,094	[fraction]
Injection Sample Pore Vol	0,238	[cc]
Injection Sample Bulk Vol	2,525	[cc]
Injection Sample Weight	6,050	g
Mean Hydraulic Radius	0,760	[µm]
Swanson's Parameter	0,006	
FZI	0,143	







#### Well : Stenlille-2 Fjerritslev Fm GEUS Core Lab and SKM Services, 04.09.2009

Plug ID	St-2	
Sample Depth	1484,70	[m]
Plug Permeability (Air)	0,015	[mD]
Plug Porosity (He)	0,151	[fraction]
Injection Sample Porosity	0,132	[fraction]
Injection Sample Pore Vol	0,261	[cc]
Injection Sample Bulk Vol	1,979	[cc]
Injection Sample Weight	4,440	g
Mean Hydraulic Radius	2,352	[µm]
Swanson's Parameter	0,022	
FZI	0,070	









#### Well : Stenlille-2 Fjerritslev Fm GEUS Core Lab and SKM Services, 04.09.2009

Plug ID	St-2	
Sample Depth	1484,70	[m]
Plug Permeability (Air)	0,015	[mD]
Plug Porosity (He)	0,151	[fraction]
Injection Sample Porosity	0,132	[fraction]
Injection Sample Pore Vol	0,261	[cc]
Injection Sample Bulk Vol	1,979	[cc]
Injection Sample Weight	4,440	g
Mean Hydraulic Radius	2,352	[µm]
Swanson's Parameter	0,022	
FZI	0,070	
FZI	0,070	







#### Well: Vedsted-1 Fjerritslev Fm GEUS Core Lab and SKM Services, 04.09.2009

Plug ID	V6-21	
Sample Depth	1403,40	[m]
Plug Permeability (Air)	0,055	[mD]
Plug Porosity (He)	0,135	[fraction]
Injection Sample Porosity	0,110	[fraction]
Injection Sample Pore Vol	0,340	[cc]
Injection Sample Bulk Vol	3,076	[cc]
Injection Sample Weight	7,130	g
Mean Hydraulic Radius	0,159	[µm]
Swanson's Parameter	0,001	
FZI	0,178	









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#### Well: Vedsted-1 Fjerritslev Fm GEUS Core Lab and SKM Services, 04.09.2009

V6-21	
1403,40	[m]
0,055	[mD]
0,135	[fraction]
0,110	[fraction]
0,340	[cc]
3,076	[cc]
7,130	g
0,159	[µm]
0,001	
0,178	
	V6-21 1403,40 0,055 0,135 0,110 0,340 3,076 7,130 0,159 0,001 0,178









#### Well: Vedsted-1 Fjerritslev Fm GEUS Core Lab and SKM Services, 04.09.2009

Plug ID	V6-20	
Sample Depth	1407,10	[m]
Plug Permeability (Air)	n/a	[mD]
Plug Porosity (He)	0,125	[fraction]
Injection Sample Porosity	0,117	[fraction]
Injection Sample Pore Vol	0,827	[cc]
Injection Sample Bulk Vol	7,053	[cc]
Injection Sample Weight	16,480	g
Mean Hydraulic Radius	0,176	[µm]
Swanson's Parameter	0,001	
FZI		









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#### Well: Vedsted-1 Fjerritslev Fm GEUS Core Lab and SKM Services, 04.09.2009

Plug ID	V6-20	
Sample Depth	1407,10	[m]
Plug Permeability (Air)	n/a	[mD]
Plug Porosity (He)	0,125	[fraction]
Injection Sample Porosity	0,117	[fraction]
Injection Sample Pore Vol	0,827	[cc]
Injection Sample Bulk Vol	7,053	[cc]
Injection Sample Weight	16,480	g
Mean Hydraulic Radius	0,176	[µm]
Swanson's Parameter	0,001	
FZI		







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#### Well: Vedsted-1 Fjerritslev Fm GEUS Core Lab and SKM Services, 04.09.2009

Plug ID	V8-15	
Sample Depth	1865,40	[m]
Plug Permeability (Air)	n/a	[mD]
Plug Porosity (He)	0,062	[fraction]
Injection Sample Porosity	0,071	[fraction]
Injection Sample Pore Vol	0,210	[cc]
Injection Sample Bulk Vol	2,973	[cc]
Injection Sample Weight	7,250	g
Mean Hydraulic Radius	0,507	[µm]
Swanson's Parameter	0,002	
FZI		









#### Well : Vedsted-1 Fjerritslev Fm GEUS Core Lab and SKM Services, 04.09.2009

Plug ID	V8-15	
Sample Depth	1865,40	[m]
Plug Permeability (Air)	n/a	[mD]
Plug Porosity (He)	0,062	[fraction]
Injection Sample Porosity	0,071	[fraction]
Injection Sample Pore Vol	0,210	[cc]
Injection Sample Bulk Vol	2,973	[cc]
Injection Sample Weight	7,250	g
Mean Hydraulic Radius	0,507	[µm]
Swanson's Parameter	0,002	
FZI		







#### Well: Vedsted-1 Fjerritslev Fm GEUS Core Lab and SKM Services, 04.09.2009

Plug ID	V8-13	
Sample Depth	1868.60	[m]
Plug Permeability (Air)	0.040	[mD]
Plug Porosity (He)	0.099	[fraction]
Injection Sample Porosity	0.080	[fraction]
Injection Sample Pore Vol	0.280	[cc]
Injection Sample Bulk Vol	3.520	[cc]
Injection Sample Weight	8.470	g
Mean Hydraulic Radius	0.804	[µm]
Swanson's Parameter	0.004	
FZI	0.258	









#### Well: Vedsted-1 Fjerritslev Fm GEUS Core Lab and SKM Services, 04.09.2009

V8-13	
1868,60	[m]
0,040	[mD]
0,099	[fraction]
0,080	[fraction]
0,280	[cc]
3,520	[cc]
8,470	g
0,804	[µm]
0,004	
0,258	
	V8-13 1868,60 0,040 0,099 0,080 0,280 3,520 8,470 0,804 0,004 0,258







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