Microstructural, Petrophysical and Anisotropy Analysis of a Posidonia Shale Analogue

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By

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Abstract

It is well-known that conventional gas resources are diminishing, forcing experts to consider exploration of unconventional. But commercially viable production of unconventional gas requires applying the reservoir stimulation technique hydraulic fracturing. One of the formations classified so far as potential interesting formation for shale gas exploration in the Netherlands is the Lower Jurassic Posidonia Shale Formation (PSF). There are vast numbers of data published regarding the characterization of the PSF, however nothing on the variation and heterogeneity. Since knowledge on this is crucial for proper hydraulic fracturing, this thesis elaborates on the variety in certain characteristics and on the anisotropy within the shale. As up to date exploration drilling is prohibited in the Netherlands, material to conduct research on is scarce. Therefore research is conducted on a time and depositional analogue of the PSF: the Whitby Mudstone Formation (WMF) in the U.K. Porosity and matrix densities are quantified with variation in the section, as well as mineral composition analysis based on XRF analysis. Velocity measurements are also conducted at multiple heights in the section and in multiple directions to elaborate on anisotropy of the material. Attenuation anisotropy is incorporated as well as Thomsen’s parameters combined with elastic parameters, e.g. Young’s modulus and Poisson’s ratio, to quantify the elastic anisotropy. The aim of this thesis is to assess whether the integrated results prove to be promising for gas exploration in the PSF-analogue, and if so, what the most promising horizons within this formation could be.

The results show that the WMF is highly anisotropic and that none of the horizons prove to be obviously favorable for gas exploration. However, based on changes in the characteristics, 4 subsections are obtained. The upper and lower part of the WMF do show relatively enhanced prospects compared to the middle. But this is a cautiously taken conclusion as some characteristics seem to contradict each other and some less decisive parameters point towards other favorable subsections.

The pessimistic results might be a distorted view caused by the many induced fractures or weathering, but one thing is clear, this time analogue of the Posidonia Shale Formation is no Barnett Shale equivalent.
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Introduction

Since conventional gas resources in Western Europe are diminishing, experts are forced to look into the tough gas reservoirs (e.g. tight carbonates and shales). Because this decline is also noticeable in the Netherlands, experts there are currently analyzing their potential resources. According to TNO, Netherlands Organization for Applied Scientific Research, the Groningen gas field will hardly be producing any gas by 2025-2030 and gas production from the smaller fields will also decline (Zijp, 2012). For this reason TNO was instructed by EBN (Energy Management Netherlands) to do exploratory research. This exploratory research confirmed gas content in the Dutch shales (mud logs) (Zijp, 2013). But due to the low permeability in shales, hydraulic fracturing is inevitable in order to produce shale gas economically viable. Extensive horizontal lamination, which is confirmed by visual observation of samples, complicates production even more. The lamination strongly influences the height of the hydraulic fractures because of the difference in rock mechanical properties parallel and normal to the bedding (Waters et al., 2011). According to that same paper, prediction of fracturing heights from logs requires knowledge of these mechanical property differences (anisotropy). Zhubayev & Barnhoorn (2013) also mention that models which neglect anisotropy may fail to predict the behavior of hydraulic fractures.

Although the discussion about shale gas is very active in the Netherlands, relatively little is actually known about the occurrence of shale gas or shale oil in the Dutch subsurface. Two intervals have been classified so far as a potential interesting formation for shale gas exploration in the Netherlands. The shallowest horizon is the Lower Jurassic (Toarcian) Posidonia Shale Formation (PSF) and the other candidate is the deeper Upper Carboniferous (Namurian) Geverik Formation. The shallow Posidonia Shale has been given the most attention and is also the intended horizon for the first shale gas well that Cuadrilla, a UK based exploration and production company, would like to drill in Boxtel, a town in the southern Netherlands. But studies focused on Dutch shales have been rare so far. In addition, very little wells have cored the shale intervals of interest for shale gas exploration. A total of 9 wells cored the Posidonia formation of which only 5 are usable (Zijp, 2013) (these were not available for this thesis). Up to date, drilling exploration wells is prohibited in the Netherlands and the Posidonia shale is not outcropping either. There are some recent papers available about characterization of the Posidonia Formation in the Netherlands (Zijp et al., 2014; Bouw & Lutgert, 2012; Van Bergen et al., 2013), but none of these papers elaborates on anisotropy of the shale. So it is clear that information about these formations, especially on the variability and heterogeneity, is scarce. Knowledge on the anisotropy and heterogeneity is crucial for accurate sweet spot determination. Hence for the research conducted in this thesis, samples of the Jet Rock member of the Lower Toarcian Whitby Mudstone Formation (WMF) have been collected along a coastal section in Whitby, United Kingdom, as analogue for the Dutch Posidonia shales. Figure 1 shows the location where the samples were collected. On these samples petrophysical, microstructural, geomechanical and anisotropy analysis will be conducted to determine what the most promising localities and horizons within the formation for future shale gas exploration could be.

Based on this information, the scientific question of this thesis is as follows:

Based on the petrophysical, microstructural and anisotropy analysis, are the empirically determined characteristics promising and useful for gas exploration in the Posidonia Shale Formation? And if so, what are the most promising horizons within the formation?
Theoretical background

As previously mentioned, experts are considering to shift towards unconventionals e.g. shale gas, to make up for the diminishing conventionals. The USA is one of the major players when it comes to the commercially viable production of shale gas. In 2012, 39% of all dry natural gas produced in the USA was shale gas (U.S. Department of Energy, 2013). Some of their largest shale plays comprise the Barnett, Haynessville, Marcellus, Fayetteville etc. (U.S. Department of Energy, 2014). In the Netherlands, unconventional activity has been going on for quite some time too, but only for conventional gas: 200 wells fracked (mostly vertical) and 22 frack jobs between 2007-2011 (9 onshore, 13 offshore) (Zijp, 2013). Because unconventional production from shale is relatively new in the Netherlands, it is important to conduct proper research first.

The Posidonia shale
According to Verreussel et al. (2013) and Zijp (2013), the Posidonia Shale Formation is a so-called “black shale”, an organic rich shale, deposited in the early Jurassic (Toarcian) approximately 180 million years ago during a widespread oceanic anoxic event. The Posidonia Shale Formation is distributed from the United Kingdom (Jet Rock Member) to Germany (Posidonienschiefer or Ölschiefer) with a uniform character and thickness (30-60m) suggesting that it was deposited during a period of high sea level and restricted basin-floor circulation (Van Bergen et al., 2013).

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1 Modified from Google Maps
This formation acts as the main source rock for oil in the Netherlands (approximately 30-50 m thick) and is also a known oil source rock in Germany (Wong, 2007). As visible in Figure 2, it occurs primarily in the West Netherlands Basin (onshore, depth between 830 and 3055 m) and the Dutch Central Graben (offshore) (Verreussel et al., 2013; Van Bergen et al., 2013).

According to TNO (Zijp, 2013), 30 wells have logged the PSF and as already mentioned, only 5 usable cores are available. The main reason for this is that the Posidonia Formation has never been the target formation. TNO produced an assessment of GIP based on detailed seismic interpretation (for area, depth and rock volume), basin modelling (for maturity distribution, temperatures and pressures), palynology, organofacies, well log interpretations (for horizontal and lateral distribution of prolific zones and gas saturation), and core analysis (for porosity and permeability) (Verreussel et al., 2013). But to date no well test data are available for shale gas in the Netherlands, so this estimate is not accurate. Also because shale gas reservoirs are such complex, heterogeneous geological systems, having a large GIP is not enough to guarantee economically viable production (Chalmers et al., 2012). Hydraulic fracturing is also necessary.

Because oil and gas exploration is conducted on a large scale in the Netherlands, there is a large geological database available, hence several important properties could be derived from this available data. Source rock characterization indicates an overall Type II kerogen, with an average TOC (Total Organic Carbon) content of about 5–7% (can be up to 14%) (Van Bergen et al., 2013). Measurements conducted on cuttings of the Posidonia shale available from 11 wells, resulted in an average TOC content of 5.73% (Van Bergen et al., 2013). So there is quite some information available about the PSF characteristics, but nothing on the heterogeneity and anisotropy of the material. Zhubayev & Barnhoorn (2013) have conducted research on anisotropy, but on the analogue of the PSF collected in Whitby. Their research was focused on the quantification of elastic anisotropy and seismic attenuation (Q−1) anisotropy. They also determined certain characteristics and

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2 Modified from Zijp (2013)
geomechanical properties such as porosity, Young’s moduli and Poisson’s ratios. Zijp et al. (2014) also used the analogous Whitby Mudstone Formation (WMF) to perform research on, but excluded anisotropy. The aim of their study was to elaborate on the fraccability and productivity of the shale. They also compared a spectral gamma ray log measurement taken in the Dutch subsurface to a measurement taken in Whitby. In general, the results showed that the Dutch subsurface values are lower, but they have similar variation patterns in gamma radiation (Th, U and K).

Research on shale in general
Gas in shale can be trapped as free gas in natural fractures and intergranular porosity, as sorbed gas into kerogen and clay-particle surfaces, or as gas dissolved in kerogen and bitumen (Curtis, 2002). According to Sayers (2013), many shales can be described, to a good approximation, as being transversely isotropic with an axis of rotational symmetry aligned perpendicular to the bedding ($x_3$-axis). For an illustration, see Figure 3.

Anisotropy in shales is often caused by partial alignment of clay particles, kerogen inclusions, microcracks, and layering (Sayers, 2013).
Several papers describe the importance of the quantification of anisotropy in shale. To produce reliable information on reservoir fluid, lithology and pore pressure from seismic data, and to understand time-to-depth conversion errors, quantification of shale anisotropy is unavoidable (Sayers, 2005). According to Banik (1984), who studied 21 datasets from the North Sea, there is an excellent correlation between the occurrence of depth errors from surface seismic data and the presence of shale in the subsurface. He also suggested a method to correct the seismic depth, which requires information about the presence of shale. Thus proper characterization and anisotropy quantification of the shales is necessary.
For elastic anisotropy, Thomsen (1986) defined three anisotropy parameters which are based on P-wave and S-wave velocities (Figure 7). Several papers have published these parameters for all kinds of shales. For attenuation anisotropy, Toksöz et al. (1979) presented a laboratory method. Attenuation coefficients represent attenuation (loss of energy) of seismic waves in rocks.

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3 Source: Zhubayev & Barnhoorn (2013)
Focus of this thesis

Analogous samples of the Posidonia Shale Formation will be used to conduct research in this thesis. The samples are collected at an outcrop in Whitby, approximately 8m in height. In Figure 4, the 8m WMF section is depicted with the collected samples (green numbers), which were not only collected for the purpose of this thesis, but for the Tough Gas Project, of which this thesis is being part of. Just below the 4th meter of height in the figure, Whale Stones (WS) are visible, which are lens / oval shaped concretions between the shale. Throughout this thesis, the WS will be used as a reference.

Figure 4: Posidonia Shale Formation time analogue section in Whitby, UK

Petrophysical analysis on the shale will comprise porosity measurements and matrix density measurements at different heights in the WMF section. For the microstructural analysis of the shale, XRD and XRF analysis will be conducted, to elaborate on the mineralogy of the Posidonia Shale Formation and its variation. To elaborate on anisotropy, velocities are measured in multiple directions. Seismic anisotropy is estimated based on a paper by Johnston & Christensen (1995) and attenuation anisotropy is quantified using the laboratory method described by Toksöz et al (1979) while Thomson’s notation (Thomsen, 1986) is used to quantify elastic anisotropy. The last two methods were already applied by Zhubayev and Barnhoorn (2013), but their experiments were carried out at ambient conditions. For the purpose of this thesis, the same experiments are performed, but the samples were subjected to axial stress to try to mimic downhole reservoir conditions. Based on the results from the ultrasonic experiments, dynamic geomechanical properties, i.e. Poisson’s ratios and Young’s moduli are also calculated. The aim is to determine if these characteristics are promising for gas exploration and if so, what the most promising horizons within the formation could be.

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Modified from Van Laerhoven, L. (TNO)
1. Methodological approach

To elaborate on the properties of the PSF analogue and the variety within this formation, several measurements and analysis are conducted. To do this, sample preparation is of great importance. In this chapter sample preparation as well as the conducted measurements and analysis will be explained in further detail, in the following order:

- Sample preparation
- Grain density and porosity measurements
- XRD / XRF analysis
- Velocity measurements and seismic anisotropy calculations
- Attenuation measurements and attenuation anisotropy calculation
- Elastic properties and elastic anisotropy (Thomsen’s parameters)

1.1 Sample preparation

Due to fragility and water-phobic nature of the material (Zhubayev & Barnhoorn, 2013), the drilling technique which was initially tried to prepare cores, proved to be unsuccessful, therefore a sawing technique is utilized to prepare rectangular shaped samples. These samples were then polished manually to obtain the cylindrical shape. To avoid deviations in the results due to different geometries, the aim is to prepare samples with equally sized diameter and length, i.e. diameter = 4 cm and height = 4 cm. To ensure correct height which is required for velocity measurements, measuring equipment is used to ensure deviation is no more than 1/8 mm. To include anisotropy, sample series consisting of a sample parallel (//), at 45° angle and perpendicular (⊥) to the layering are prepared as visualized in Figure 5. Throughout this thesis, the samples will be referred to as vertically, diagonally or 45° and horizontally layered samples respectively.

![Image of samples](image)

*Figure 5: Example of one set after preparation (left) and a sample which broke during preparation (right)*

Powdered sample preparation will be mentioned in paragraph 1.3.

Encountered difficulties while preparing

Sample preparation proved to be very time consuming, therefore not all sets are complete and of the same height and diameter. Because of the fragility of the material, samples often break along the layering as illustrated in Figure 5, on the right.
1.2 Porosity and matrix density measurements

(Based on the Pycnometer Manual)

Matrix density and porosity measurements are performed using the Ultrapycnometer 1000 version 2.12. The Ultrapycnometer measures volume and density of solid objects. To maximize the accuracy, helium is used as the displaced fluid because of its small atomic dimensions. The pycnometer measures the volume of the solids in the sample placed in the sample holder, so the result gained from the pycnometer is equal to the matrix volume of the sample (\(V_m\)). Since the total mass of the matrix (\(M_m\)) can be determined easily, the matrix density \(\rho_m\) can be calculated using:

\[
\rho_m = \frac{M_m}{V_m}
\]

Both cylindrical and powdered samples are subjected to matrix density measurements. The samples were powdered using a mortar & disc mill, whereupon they were oven dried at a low temperature (70 °C) for at least 24 hours.

From the results of the non-powdered samples, porosities (\(\phi\)) are estimated. The total volume of the sample (\(V_t\)) consists of void space (\(V_v\)) and matrix volume \(V_m\), so \(V_t = V_v + V_m\). Since only cylindrical samples are prepared, \(V_t\) can be calculated from the measured height and diameter. To increase accuracy, the average of a minimum of 12 measurements was used. With \(V_m\) and \(V_t\) known, \(\phi\) can be calculated using the following equation:

\[
\phi = \frac{V_v}{V_t} = \frac{V_t - V_m}{V_t}
\]

Because samples are manually polished, the total volume calculated assuming a perfect cylinder (\(V_t = \pi r^2 h\)) deviates slightly from the “true” total volume of the sample. To estimate the deviation, a micro-CT scanner was used to obtain sample volumes. Micro-CT bulk volumes differ \(\approx 1\%\) (2 samples) from the calculated volume results. As bulk volume increases, porosity also showed slight increase. But due to beam hardening, a phenomenon also encountered and described by Ravestein (2014), this process turned out to be time consuming and also not totally accurate. Furthermore, bulk volume change is estimated at \(\approx 1\%\) thus it was therefore not applied to all samples.

1.3 XRD / XRF analysis

This paragraph is based on the reports “Identification and semi quantification of minerals in clay powders” and “Axios Results Quantification of samples Joella” by Ruud Hendrikx.

Accurate quantitative mineral analysis is important to elaborate on the variation within the formation. For this purpose X-ray diffraction and X-ray fluorescence (XRD and XRF) analysis were conducted at the X-RAY FACILITIES department of Materials Science & Engineering (3ME), Delft University of Technology. XRD is an analytical method used for determining the presence and absolute amounts of mineral content in the shale samples. XRF supplies information about the elemental composition of the shale samples.
1.3.1 The setup

The analyzed specimens were powdered using a mortar & disc mill, whereupon they were oven dried at a low temperature (70 °C) for at least 24 hours.

For XRF analysis the measurements were performed using a Panalytical Axios Max WD-XRF spectrometer and data evaluation was done with the SuperQ5.0i/Omnian software.

The XRD patterns were obtained using the Bruker D5005 diffractometer Bragg-Brentano geometry with Huber incident-beam monochromator and a Braun position sensitive detector was used. For data evaluation Bruker software Diffrac.EVA 3.1 was used.

1.3.2 Processing the data

Semi-quantitative XRD and XRF conducted at the faculty 3ME at TU Delft

For the semi-quantitative analysis, relative intensity values obtained by integrating certain peaks of the components are used. The semi-quantitative analysis of the phases is performed by scaling the intensities of the diffraction phase of the different samples to one diffracted peak intensity of a certain reference phase, in this case corundum. Figure 6 shows an example of the recorded XRD patterns in black. The colored sticks give the peak positions and intensities of the identified phases, such as found using the ICDD PDF-4 database (advanced database from the International Center for Diffraction Data designed for both phase identification and quantitative analysis). The patterns are background-subtracted, meaning that the contribution of air scatter and possible fluorescence radiation is subtracted.

![Figure 6: Example of XRD patterns shown in black](image-url)
XRF is the emission of characteristic "secondary" (or fluorescent) X-rays (form of electromagnetic radiation) from a material that has been excited by bombarding with high-energy X-rays. The emitted radiation has energy characteristic of the atoms present. The phenomenon is used for elemental analysis of solids, powders or liquids.

Mineral composition from Basica (software supplied by Dr. Karl-Heinz Wolf)
By using Basica, one can determine which minerals might be present in the rock sample with associated weight percentages (wt%), based on (and in consistency with) the elemental composition of the rock sample derived from the XRF analysis. So the software determines the mineral composition by regressive calculation from the XRF elemental analysis.

1.3.2 Encountered difficulties

Whereas mineral identification is relatively simple and unambiguous if modern software and good databases are available, accurate quantitative analysis of clays remains a challenge (Srodon et al., 2001). A major source of error is the platy habit of clay crystallites resulting in a tendency for a preferred orientation (Srodon et al., 2001). Another source of error is the nonexistent $I/I_{cor}$ value for certain clay minerals in the database (ICDD-PDF4).

1.4 Velocity and seismic anisotropy measurements

To elaborate on anisotropy of the shale, compressional (P-wave) and shear (S-wave) velocities (see Figure 7) will be measured for different samples, in multiple directions. The samples will be subjected to axial pressure to try to mimic downhole conditions (for visualization of the set-up, see Figure 8). With the calculated velocities, anisotropies will be determined. Section 1.4 will elaborate on the velocity measurements, while section 1.4.2 will elaborate on the seismic anisotropy calculation.

![Figure 7: P-wave and S-wave propagation](https://sites.google.com/site/xrayfacilities/)

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5 https://sites.google.com/site/xrayfacilities/
6 Source: http://www.earth.northwestern.edu/people/seth/B02/lectures/Seismology/p&swaves.htm
1.4.1 Velocity measurements

To measure velocity and seismic anisotropy, both P-wave S-wave transducers were used with 1 MHz central frequency. However, these could not be mounted to the sample at the same time thus the S-wave measurements were conducted first, followed by the P-wave measurements. The schematic of the set-up is depicted on the left in Figure 8, while the real set-up during measurements is seen on the right.

![Figure 8: A schematic (left) and picture (right) of the setup](image)

Processing the data

Considering that the sample length is known and the arrival time of the P-wave or S-wave can be determined from the recorded signals, the velocities are computed for associated stresses. With increasing stress, the sample length decreases. This shortening is also incorporated in the velocity computations. Figure 9 illustrates an example of the arrival of S-wave traces. Each trace represents a measurements recorded at a different stress level.

![Figure 9: Example of recorded signals for shale sample #70 (S-wave)](image)

---

7 Modified from Zhubayev & Barnhoorn (2014)
If the arrival time is picked from Figure 9 (red dashed line), the velocities are calculated with varying stress. An example of the calculated velocities is shown in Figure 10.

![Figure 10: Example of velocity vs. axial stress sample #70 (S-wave)](image)

1.4.2 Seismic anisotropy calculations

With the P- and S-wave velocities, the seismic anisotropies are estimated using the following definition (Johnston & Christensen, 1995):

\[
\frac{V_{\text{max}} - V_{\text{min}}}{V_{\text{max}}} 
\]

(1.3)

\(V_{\text{max}}\) is defined as the velocity traveling parallel to the layering, whereas \(V_{\text{min}}\) is defined as the velocity traveling perpendicular to the layering. Because the WMF shales are highly laminated, the velocity travelling parallel to the layering always exceeds the velocity normal to the layering. Data in the Results chapter will confirm this.

1.5. Attenuation and attenuation anisotropy measurements

This paragraph elaborates on seismic attenuation measurements with attenuation anisotropy in dry WMF samples. Section 1.5.1 explains how the attenuation values are obtained, whereupon section 1.5.2 elaborates on the attenuation anisotropy.

1.5.1 Attenuation measurements

A pulse transmission technique by Toksöz et al. (1979) is used to determine the quality factor Q. The amplitude decay (attenuation) of seismic signals traveling through samples is measured. Attenuation (Q-1) is measured relative to a reference sample with very low attenuation. In this case aluminum is used as reference (Q=150.000) (Toksöz et al., 1979).

The setup

The samples used are the ones described in paragraph 1.1, cylinders with \(d = h = 4\) cm. The shale and aluminum samples have the same geometry and approximately the same size. Since attenuation calculations are based on the results of the velocity measurements, the same set-up shown in Figure 8 is used for these measurements. The polarization of the shear source and receiver was always aligned parallel to each other.
Processing the data

Recorded acoustic data is read in as csv files. If possible, the stress decrease part is preferred above the stress increase part, because as Alimzhan Zhubayev explains (personal communication, February 2014), experience tells that the decreasing stress part produces more accurate results than the increasing stress part.

The amplitude ratio of the shale and aluminum signals, assuming constant Q, is calculated using: (Toksöz et al., 1979)

\[
\ln \left( \frac{A_1}{A_2} \right) = (\beta_2 - \beta_1) x F + \ln \left( \frac{G_1}{G_2} \right)
\]  

(1.4)

- \(A_1\) = Fourier amplitude of the reference sample (aluminum)
- \(A_2\) = Fourier amplitude of the shale sample
- \(F\) = Frequency
- \(x\) = Propagating distance
- \(G_i\) = Scaling factor due to spherical divergence (independent of frequency)
- \(\beta\) = A constant related to Q
- \(V\) = Seismic velocity

If the same geometry is used for both sample and reference, \(G_1\) and \(G_2\) are frequency-independent scale factors. If \(G_1/G_2\) is independent of frequency, \((\beta_2 - \beta_1)\) can be determined by estimating the slope of the line that fits the \(\ln(A_1/A_2)\) versus frequency graph. When Q of the reference sample is high (\(\text{Q}_1 \approx \infty\)), \(\beta_1 = 0\) and \(\beta_2\) of the shale can be determined using the slope. Finally, Q can be calculated using equations (1.4) & (1.5).

\[
Q = \frac{\pi}{\beta V}
\]

(1.5)

By applying a time window around the peak amplitude of the first arrival, an amplitude spectrum is calculated (Zhubayev & Barnhoorn, 2013) (see Figure 11 on the left). With the amplitude spectrum, the amplitude ratio \(\ln(A_1/A_2)\) (see Figure 11 on the right) is calculated and plotted versus frequency.

![Figure 11: Example of a calculated amplitude spectrum (left) with associated natural logarithm of the spectral ratio (right) as a function of frequency for sample #70 (S-wave)]
From the slope of the best fit line shown in red in Figure 11, $Q$ and therefore $Q^{-1}$ can be determined. An example is shown in Figure 12.

![Figure 12: Example of $Q^{-1}$ calculated for sample #70](image)

1.5.2 Attenuation anisotropy

With the obtained attenuation values, the attenuation anisotropy can be estimated using the following definition:

$$\frac{Q^{-1}_{\perp} - Q^{-1}_{//}}{Q^{-1}_{\perp}}$$

(1.6)

$Q^{-1}_{//}$ is defined as the attenuation measured parallel to the bedding, while $Q^{-1}_{\perp}$ is defined normal to the bedding.

1.6 Thomsen’s notation and elastic constants

To elaborate on the elastic anisotropy of the shale, the so-called Thomsen’s notation is used. In terms of elastic anisotropy, shale can be considered to be transversely isotropic (TI), meaning shale samples show change in the vertical direction, but are more or less constant in any horizontal direction i.e. regardless of azimuth. The axis of symmetry in shale ($x_3$) is aligned perpendicular to the bedding as illustrated in Figure 4.

A TI sample is specified by 5 independent elastic moduli: $C_{11}$, $C_{12}$, $C_{13}$, $C_{33}$ and $C_{44}$ and $C_{11} = C_{22}$, $C_{12} = C_{21}$, $C_{13} = C_{31}$, $C_{23} = C_{32}$ and $C_{44} = C_{55}$. With $x_3$ specified as mentioned above, the stress-strain relation according to Mavko et al. (2009) becomes:
\[ \begin{bmatrix} \sigma_{11} \\ \sigma_{22} \\ \sigma_{33} \\ \sigma_{12} \end{bmatrix} = \begin{bmatrix} C_{11} & C_{12} & C_{13} & 0 & 0 & 0 \\ C_{12} & C_{11} & C_{13} & 0 & 0 & 0 \\ C_{13} & C_{13} & C_{33} & 0 & 0 & 0 \\ 0 & 0 & 0 & C_{55} & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & C_{66} \end{bmatrix} \begin{bmatrix} \varepsilon_{11} \\ \varepsilon_{22} \\ \varepsilon_{33} \\ \varepsilon_{12} \end{bmatrix} \] (1.7)

\( \sigma_{ij} \) and \( \varepsilon_{ij} \) signify stress and strain respectively in the j-direction working on the i-plane.

The five components of the stiffness tensor for a TI material are obtained from five velocity measurements: \( V_p(0^\circ) \), \( V_p(90^\circ) \), \( V_p(45^\circ) \), \( V_{SH}(90^\circ) \) and \( V_{SV}(0^\circ) = V_{SV}(0^\circ) \) (Mavko et al., 2009). These five velocities are seismic velocities measured as explained in paragraph 1.4 of this thesis. \( V_p(0^\circ) \), \( V_p(90^\circ) \), \( V_p(45^\circ) \) are P-wave velocities propagating perpendicular, parallel and at a 45° angle respectively to the bedding. \( V_{SH}(0^\circ) \) refers to the S-wave velocity propagating perpendicular to the bedding and the plane of polarization is in the \( x_1-x_3 \) or \( x_2-x_3 \) plane, while \( V_{SH}(90^\circ) \) refers to the S-wave velocity propagating parallel to the bedding with the plane of polarization in the symmetry \( x_1-x_2 \) plane (Zhubayev & Barnhoorn, 2013).

The 5 independent elastic moduli are calculated as follow (Mavko et al., 2009):

\[ C_{11} = \rho V_p^2(90^\circ) \] (1.8)
\[ C_{12} = C_{11} - 2\rho V_{SH}^2(90^\circ) \] (1.9)
\[ C_{33} = \rho V_p^2(0^\circ) \] (1.10)
\[ C_{44} = \rho V_{SH}^2(0^\circ) \] (1.11)
\[ C_{13} = -C_{44} + \sqrt{4\rho^2 V_p^4(45^\circ) - 2\rho V_p^2(45^\circ) (C_{11} + C_{33} + 2C_{44}) + (C_{11} + C_{44})(C_{33} + C_{44})} \] (1.12)

\( C_{66} \) is formed as a combination of other moduli:

\[ C_{66} = \frac{(C_{11} - C_{12})}{2} \] (1.13)

With these constants, the Thomsen's anisotropy parameters \( \varepsilon, \delta \) and \( \gamma \) can be calculated with the following equations (Thomsen, 1986):

\[ \varepsilon = \frac{C_{11} - C_{33}}{2C_{33}} \] (1.14)
\[ \gamma = \frac{C_{66} - C_{55}}{2C_{55}} \] (1.15)
\[ \delta = \frac{(C_{11} + C_{55})^2 - (C_{33} - C_{55})^2}{2C_{33}(C_{33} - C_{55})} \] (1.16)

Thomsen (1986) defined the weak to moderate anisotropy range as \( \varepsilon, \delta \) and \( \gamma < 0.2 \).
A TI medium can be characterized by 2 independent Young’s moduli namely $E_1=E_2$ and $E_3$ and 2 Poisson’s ratio’s namely $\nu_{21}=\nu_{12}$ and $\nu_{31}=\nu_{13}$. $E_1$ is the Young’s modulus parallel to the bedding ($x_1$-axis), while $E_3$ is perpendicular to the bedding ($x_3$-axis). $\nu_{12}$ corresponds to the elastic strain in the $x_2$-direction due to stress applied in the $x_1$-direction while $\nu_{13}$ corresponds to the elastic strain in the $x_3$-direction due to stress applied in the $x_1$-direction.

Poisson’s ratios are calculated using seismic velocities (Mavko et al., 2009):

$$\nu_{12} = \frac{(V_p(90^\circ)/V_s(90^\circ))^2 - 2}{2(V_p(90^\circ)/V_s(90^\circ))^2 - 1} \quad (1.17)$$

$$\nu_{31} = \frac{(V_p(0^\circ)/V_s(0^\circ))^2 - 2}{2(V_p(0^\circ)/V_s(0^\circ))^2 - 1} \quad (1.18)$$

The Young’s moduli are calculated as follow (Zhubayev & Barnhoorn, 2013):

$$E_1 = 2C_{66}(1+\nu_{12}) \quad (1.19)$$

$$E_3 = 2C_{44}(1+\nu_{31}) \quad (1.20)$$

$E$ and $\nu$ can give an indication of the brittleness of a rock. Poisson’s ratio (low values preferred) reflects the rock’s ability to fail under stress, while Young’s modulus (high values preferred) reflects the ability to maintain a fracture (Rickman, 2008). Based on these 2 characteristics, the Brittleness Index (BI) can be estimated, but this thesis will not elaborate on the BI, because this topic is discussed extensively in the thesis of Thomas Ravestein (his thesis is also part of the Tough Gas project).
2. Results

In this chapter the results will be presented and interpreted.

2.1 Porosity and grain density measurements

In this paragraph, the porosities and matrix densities derived from the helium pycnometer, are depicted. Matrix densities were estimated from powdered and cylindrical samples.

Figure 13: $\phi$ and $\rho_m$ vs. height in the Whitby section (upper 2 and lower right) and $\phi$ vs. $\rho_m$ (lower left)\(^8\)

Figure 13 depicts porosities that vary between 0.26% and 5.82%, matrix densities measured on cylindrical samples that vary between 2.25 g/cc and 2.55 g/cc, and matrix densities measured on powdered samples that vary between 2.21 g/cc and 2.80 g/cc. The porosity results show no correlation between height in the section and porosity. The aim was to conduct porosity measurements on at least 2 cylindrical samples (d=4cm; h=4cm) taken from the same block (block size varies from approximately 20x20x5 cm to 30x30x10 cm). As depicted here, even samples taken from the same block and therefore the same height, have a varying porosity, suggesting that there is variation in porosity on cm scale. To analyze the variation within the section, it was

\(^8\) 2 points (h = 3.5m) are results of measurements by Thomas Ravestein
divided into 2 parts: the first part above the Whale Stones (WS) and the second part below the WS. But there is no clear correlation between the upper and lower part of the section either. The data is scattered. The matrix densities measured on cylinders taken above the WS seem more clustered than below the WS, suggesting that the upper part of the section might be more homogeneous in terms of matrix density. Dr. Maartje Houben, a Post-Doctoral Researcher from the University of Utrecht who also conducts research on the WMF, confirmed that some of the characteristics she is working on also show more homogeneity in the upper part of the section (personal communication, August 2014). The matrix densities measured on cylinders and on powdered samples both seem to decrease towards the WS, but this phenomena will be discussed later on in this thesis. The lower left graph in Figure 13 shows that there is no correlation between porosity and matrix density. The lower right graph shows matrix densities measured on powdered samples. A noticeable point from this graph is that matrix densities from powdered samples exceed the matrix densities from cylindrical samples on all of the corresponding heights. This will be discussed in paragraph 3.1. The tabulated results with associated sample number and height in the WMF section, are shown in APPENDIX A.

2.2. XRD / XRF analysis

In this paragraph the results of the XRF analysis will be presented and the results of the XRD analysis will be discussed. Based on the elemental analysis, the mineral composition of the different samples was determined with associated weight percentages (wt%) by using Basica, a program supplied by Dr. Karl-Heinz Wolf.

The XRD results obtained at the faculty 3ME were not be used for the purpose of this thesis. The PDF-4 database used to compare the RIR values doesn’t contain clay minerals. They still provide weight percentages for kaolinite and illite, but these are based on similar cards in the database. For montmorillonite, they didn’t have a similar card, so calculated weight percentages were normalized to 100%, resulting in large errors in the percentages. However the XRD results did help to confirm which minerals are in the samples, only not in determining the wt%.

The XRF results obtained at the faculty 3ME proved to be very useful in analyzing mineral composition when used in combination with the software supplied by Dr. Karl-Heinz Wolf. The XRF results contain an average error of ±4.5%. The values were normalized to 100%. APPENDIX B contains the tabulated XRF results and the mineral composition obtained using Basica.

Figure 14 depicts the estimated clay, calcite, quartz and pyrite content respectively with varying height in the section. The results show that the quartz content is more or less constant. The values are not very high. Clay content on the other hand is very high. All values are higher than 45%. Clay and calcite content show a negative correlation. Pyrite content shows a positive correlation with calcite content therefore a negative correlation with clay content. Close to the WS, pyrite content increases. Note that the horizontal axes have the same increment and relative range, but not the same values.
Figure 14: Clay, calcite and quartz content of the WMF with height in the section

Other minerals assumed to be in the WMF shales are goethite and chlorite with averages of 5-6%. APPENDIX B contains a table showing the calculated percentages per sample. It is clear that the samples are very clay-rich (ductile), though the clay content seems to decrease towards the WS. In paragraph 3.2, the collected results will be compared to other obtained characteristics to try to explain this phenomenon. The clay and quartz content will also be compared to other interesting shales, to place the WMF in the bigger picture.

2.3 Velocity and seismic anisotropy measurements

In this paragraph the results of the P- and S-wave velocity measurements conducted in multiple directions, will be presented (section 2.3.1). 11 samples are subjected to measurements at zero-stress (=0 MPa) conditions of which 6 are later on also subjected to increased stress. With these velocities, the seismic anisotropy of the shales will be estimated (section 2.3.2) as described in the Methodology chapter. In APPENDIX C the sample positions in the WMF section are shown.
2.3.1 Velocity measurements

Figure 15: Velocities measured ⊥ to the bedding vs. height in the WMF (zero-stress). For clarification, the sample orientation is shown on the right.

Figure 15 depicts the variation in S- and P-wave velocity. S-wave velocities seem to decrease slightly towards the Whale Stones, but this trend is not clear in the P-wave velocities measured above the WS. Velocities below the WS seem to be less scattered than above the WS. Note that the horizontal axes have the same increment and relative range, but not the same values.

Figure 16: S- and P-wave velocities with increasing stress in horizontally layered samples

Figure 16 shows increasing S- and P-wave velocities with stress. It is assumed to be due to the closing of the microcracks and fractures present in the samples (Winkler & Nur, 1982) (Figure 17), causing the mineral frame to tighten up. As at least 30 MPa was reached for all of the horizontally layered samples, the P-wave velocities don’t seem to level out yet, while the S-wave velocities seem to level out nicely. Note that the vertical axes
have the same increment and relative range, but not the same values (this also holds for Figure 18 & Figure 19).

---

**Figure 17:** Fractures are present in the sample before subjecting it to stress

**Figure 18:** S- and P-wave velocities with increasing stress in vertically layered samples

The same conclusions from Figure 16 can be drawn from Figure 18. The S-wave also flattens out while this is not the case for the P-wave.

**Figure 19:** S- and P-wave velocities with increasing stress in samples with 45° layering

Measurements conducted on samples with 45° layering, proved to be much more difficult. Samples tended to break along the layering at lower stresses. But these results also show the same aforementioned trends. Since the P-wave travels faster than the S-wave, a steeper gradient is expected in the P-wave velocities. But to
confirm whether this change in gradient is only caused by faster traveling P-waves, the ratios $V_p/V_s$ were calculated at minimum and maximum stress and compared. The results are shown in Table 1.

<table>
<thead>
<tr>
<th>Sample #</th>
<th>$V_p/V_s$ min. stress</th>
<th>$V_p/V_s$ max. stress</th>
<th>Sample #</th>
<th>$V_p/V_s$ min. stress</th>
<th>$V_p/V_s$ max. stress</th>
</tr>
</thead>
<tbody>
<tr>
<td>47-H</td>
<td>1.47</td>
<td>1.55</td>
<td>47-V</td>
<td>1.79</td>
<td>1.73</td>
</tr>
<tr>
<td>56-H</td>
<td>1.75</td>
<td>1.74</td>
<td>56-V</td>
<td>1.63</td>
<td>1.63</td>
</tr>
<tr>
<td>70-H</td>
<td>1.89</td>
<td>1.92</td>
<td>70-V</td>
<td>1.71</td>
<td>1.75</td>
</tr>
<tr>
<td>29-H</td>
<td>1.65</td>
<td>1.79</td>
<td>29-V</td>
<td>1.67</td>
<td>1.68</td>
</tr>
<tr>
<td>2-H</td>
<td>1.47</td>
<td>1.57</td>
<td>2-V</td>
<td>1.55</td>
<td>5.26</td>
</tr>
<tr>
<td>45-H</td>
<td>1.59</td>
<td>1.64</td>
<td>45-V</td>
<td>1.67</td>
<td>1.68</td>
</tr>
</tbody>
</table>

Table 1: $V_p/V_s$ ratios at minimum ($\approx$0 MPa) and maximum stress

9 out of 12 measurements conducted on horizontally and vertically layered samples, $V_p/V_s$ amplifies with increased stress, indicating that the steeper gradient is not only caused by faster traveling P-waves. The leveling of the S-wave velocities may be explained by the fact that S-wave measurements are conducted first, followed by P-wave measurements on exactly the same sample. Samples are stressed during the S-wave measurements, causing extra fractures and microcracks during relaxation. The samples probably contain more fractures and microcracks when subjected to stress again for P-wave velocity measurements, leading to greater velocity increases.

According to Mavko (n.d.), pressure dependence of velocities can be determined from core measurements. By normalizing the velocities of each sample by the high pressure value, the curves can be clustered at the high pressure point. The equation of the dashed trendline through the cloud of lines can provide an estimate of velocities at higher stresses. Figure 20 shows the Whitby shale velocity data with the best fit trendlines.

![Figure 20: Pressure dependence of velocity velocities from core measurements for P- and S-wave](image-url)
2.3.2 Seismic anisotropy measurements

Based on the calculated velocities, the seismic anisotropy was calculated as mentioned in section 1.4.2.

![Figure 21: S- and P-wave anisotropy along the Whitby section](image)

<table>
<thead>
<tr>
<th>Anisotropy min. stress</th>
<th>Anisotropy max. stress</th>
</tr>
</thead>
<tbody>
<tr>
<td>P-wave [%]</td>
<td>S-wave [%]</td>
</tr>
<tr>
<td>32.9</td>
<td>18.4</td>
</tr>
<tr>
<td>28.7</td>
<td>33.4</td>
</tr>
<tr>
<td>24.4</td>
<td>31.5</td>
</tr>
<tr>
<td>36.5</td>
<td>36.1</td>
</tr>
<tr>
<td>34.2</td>
<td>30.9</td>
</tr>
<tr>
<td>P-wave [%]</td>
<td>S-wave [%]</td>
</tr>
<tr>
<td>26.5</td>
<td>17.8</td>
</tr>
<tr>
<td>22.7</td>
<td>27.3</td>
</tr>
<tr>
<td>25.2</td>
<td>31.9</td>
</tr>
<tr>
<td>30.2</td>
<td>34.4</td>
</tr>
<tr>
<td>28.4</td>
<td>26.8</td>
</tr>
</tbody>
</table>

Table 2: P- and S-wave anisotropy of the Whitby shale at min. and at max. stress

The results presented in Figure 21 confirm that the Whitby shales are highly anisotropic with values ranging from 18 to almost 37%. Table 2 shows that anisotropy decreases with increasing stress. A reasonable explanation for this phenomenon can be that aligned microcracks are closing and that grain contacts consolidate, resulting in the decrease in anisotropy.

2.4 Attenuation measurements

This paragraph elaborates on seismic attenuation measurements (section 2.4.1) and attenuation anisotropy (section 2.4.2) in dry WMF samples. A pulse transmission technique is used to estimate the attenuation of seismic signals traveling through the samples. To determine from which part of the Whitby section the chosen samples are, an image of the section with corresponding heights and colors is given in APPENDIX D.
2.4.1 Attenuation measurements

Figure 22 reveals decreasing attenuation with increasing stress for both P- and S-waves measured ⊥ to the bedding. At low stresses, attenuation decrease rates are higher, but are leveling off with increasing stress. An explanation for the high decrease rate can be fracture and microcrack closure, allowing signals to travel without too much amplitude loss at increased stress. Some of the samples show a strange increase in attenuation at low stress, before the rapid decline begins. This phenomenon will be discussed in paragraph 3.4.

In this paragraph only the attenuation values measured ⊥ to the bedding are presented, because values of attenuation measured // and at a 45° angle to the bedding show the same trend. The figures are shown in APPENDIX D. An important point to be noted is that the 45° results are not as consistent as the other results, but most of them too show the same declining trend. An explanation for this can be that the samples prepared at a 45° angle showed considerable more fractures due to sample preparation. These samples were much more difficult to prepare because they tended to break easily along the layering. The applied maximum stresses are therefore lower.

From the 18 samples subjected to measurements, 10 samples consistently showed $Q_P^{-1} > Q_S^{-1}$ at all the measured stress values (± 11 measured stress values/sample). To support this, paragraph 3.4 contains a figure showing P-wave attenuation vs. S-wave attenuation at minimum and maximum stresses. Toksöz et al. (1979) measured ultrasonic attenuation on dry, gas- or water-saturated sandstone and limestone samples. They found that $Q_P^{-1} > Q_S^{-1}$ for dry or methane-saturated rocks. This result is in consistency with most of the Whitby shale results because the Whitby samples are “room dry”.

...
2.4.2 Attenuation anisotropy measurements

This section will elaborate on attenuation anisotropy i.e. attenuation measured parallel to the layering versus attenuation measured normal to the layering.

![Figure 23: P- (left) and S-wave (right) attenuation parallel to the layering. Red triangle: at maximum applied stress. Red dashes and black crosshairs: at ambient conditions (stress ≈ 0). The grey lines are the isotropic lines.](image)

The right chart in Figure 23 depicts the S-wave attenuation measured perpendicular to the bedding, calculated at minimum and maximum applied stresses. The black crosshairs display measurements by Zhubayev & Barnhoorn (2013) on Whitby samples, conducted at ambient conditions. His results compared to the results obtained for the purpose of this thesis (at minimum stress) fall within the same range, but are less scattered. At minimum applied stress, the self-obtained data is scattered above and below the 45° line, meaning that it is not clear in which direction ultrasonic attenuation is larger, either parallel or perpendicular to the bedding. But at maximum applied stress, 5 out of 6 cases show that attenuation perpendicular to the bedding is larger than attenuation parallel to the bedding. The left chart of Figure 23 shows the P-wave attenuation measured perpendicular to the bedding at minimum and maximum applied stresses. In this graph, there is no clear dominating anisotropy direction in either the low or high stress regime. The attenuation values by Zhubayev and Barnhoorn (2013) at ambient conditions are lower and the attenuation anisotropy is less, since their values are closer to the 45° line. Another point to be noted is that compressional anisotropy is lower than shear wave anisotropy as the data points are closer to the 45° line. Paragraph 3.4 will elaborate on this.

---

9 Modified from Zhubayev & Barnhoorn (2013) (black crosshairs)
2.5 Thomsen’s anisotropy parameters and elastic properties

This paragraph will give the results of the elastic anisotropy analysis. Based on velocities and elastic stiffness coefficients, Thomsen’s parameters are calculated as well as Young’s moduli and Poisson’s ratios.

Figure 24 depicts cross-plots of δ, γ and ε above and below (filled points) the Whale Stones in the WMF section. The upper 3 graphs were measured at ambient conditions. Zhubayev and Barnhoorn (2013) also conducted measurements on the WMF samples at ambient conditions, so their results (green points) were added to the data collected for the purpose of this thesis, to get a more extensive dataset of the WMF.

![Graphs showing Thomsen's anisotropy parameters](image)

The results in Figure 24 show that the Whitby shales are highly anisotropic. The exact ranges measured at ambient conditions (no stress applied), are shown in the left part of Table 3. For shales, the high anisotropy values (ε and γ) are expected, because of the fine layering visible in the samples. The WMF is classified as highly anisotropic since \( \varepsilon, \gamma \approx 0.2 \) (Thomsen, 1986). For the other parameter (δ), 10 out of 12 calculated values are negative. Because literature reports lab measurements of both positive and negative values, the sign of this parameter is poorly understood (Sayers, 2005). It will be discussed in paragraph 3.5.

The lower 3 graphs of Figure 24 show the anisotropy parameters measured at maximum stress (averages of 32 MPa for horizontally layered, 25 MPa for vertically layered and 12 MPa for 45° layered samples). Table 3

---

10 Modified from Zhubayev & Barnhoorn (2013) (green points)
shows the ranges from minimum to maximum $\varepsilon$, $\delta$ and $\gamma$ measured on the different samples at low (left) and high (right) stress. The values decrease as stress is increased.

<table>
<thead>
<tr>
<th>At minimum stress (=0MPa)</th>
<th>Epsilon [-]</th>
<th>Delta [-]</th>
<th>Gamma [-]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.38 to 0.74</td>
<td>-0.24 to -0.11</td>
<td>0.25 to 0.72</td>
</tr>
<tr>
<td>At maximum stress</td>
<td>Epsilon [-]</td>
<td>Delta [-]</td>
<td>Gamma [-]</td>
</tr>
<tr>
<td></td>
<td>0.39 to 0.53</td>
<td>-0.37 to -0.20</td>
<td>0.24 to 0.66</td>
</tr>
</tbody>
</table>

Table 3: Thomsen’s parameter ranges at minimum (left) and maximum (right) stress

The Young’s moduli and Poisson’s ratios are also estimated at ambient conditions (no stress) (Figure 25, upper 2 graphs) and at maximum applied stresses (Figure 25, lower 2 graphs) (maximum applied stresses coincide with the maximum applied stresses mentioned for the Thomsen’s parameters).

$E_1$ measured parallel to the bedding $> E_3$ measured normal to the bedding for all samples. The self-generated results at ambient conditions show that in 3 out of 4 cases $v_{31} < v_{12}$ (stress applied normal to the bedding while strain is measured parallel ($v_{31} = v_{13}$) $<$ stress applied parallel to the bedding while strain is measured normal to the bedding). But as stress is increased, the $v_{31}$ increased more than $v_{21}$. The data points shift closer to or above the 45° line ($v_{31} > v_{12}$). Sayers (2013) predicted that $E_1 > E_3$ and $v_{31} > v_{12}$ for shales. But he also concluded that

$^{11}$ Modified from (Zhubayev & Barnhoorn, 2013) (green points)
results sometimes prove otherwise: $\nu_{31}$ can be greater than, equal to or less than $\nu_{12}$. The WMF shale values will be compared to other shales in paragraph 3.5.

<table>
<thead>
<tr>
<th>$E_3$ min</th>
<th>$E_3$ max</th>
<th>$E_1$ min</th>
<th>$E_1$ max</th>
<th>$\nu_{12}$ min</th>
<th>$\nu_{12}$ max</th>
<th>$\nu_{31}$ min</th>
<th>$\nu_{31}$ max</th>
</tr>
</thead>
<tbody>
<tr>
<td>15.43</td>
<td>20.02</td>
<td>27.00</td>
<td>32.17</td>
<td>0.22</td>
<td>0.20</td>
<td>0.05</td>
<td>0.10</td>
</tr>
<tr>
<td>12.64</td>
<td>13.27</td>
<td>25.57</td>
<td>27.29</td>
<td>0.19</td>
<td>0.21</td>
<td>0.25</td>
<td>0.26</td>
</tr>
<tr>
<td>11.96</td>
<td>13.40</td>
<td>29.41</td>
<td>30.02</td>
<td>0.17</td>
<td>0.18</td>
<td>0.16</td>
<td>0.22</td>
</tr>
<tr>
<td>13.22</td>
<td>15.70</td>
<td>28.72</td>
<td>29.77</td>
<td>0.17</td>
<td>0.18</td>
<td>0.13</td>
<td>0.16</td>
</tr>
</tbody>
</table>

Table 4: $E_3$, $E_1$, $\nu_{12}$ and $\nu_{31}$ at min. and max. applied stress

The lower 2 charts of Figure 25 depict results at maximum applied stresses. For clear comparison, Table 4 depicts values of Young’s moduli and Poisson’s ratios at minimum (columns with min) and maximum applied stresses (columns with max). For all of the individual measurements, the Young’s moduli increased with applied stress and for all but 1 measurements the Poisson’s ratios also increased. Because increased stress compacts the sample, $E$ is expected to increase. Increased Poisson’s ratio means that with increased stress, radial deformation of the samples is large compared to the axial deformation.
3. Discussion

In this chapter, the results presented in chapter 2 will be discussed and compared to results presented by authors of other literature. The aim is to decide whether the collected results are favorable for shale gas production based on current or past production data from the other shales, e.g. if the Whitby shale results prove to be similar to the Barnett shales, it might be promising.

3.1 Porosity and matrix density measurements

In this paragraph the porosity and matrix density results will be discussed and compared to other shale formations to determine whether the WMF values are promising or not.

The matrix densities presented in Figure 13 display higher values for powdered samples than for cored samples. This is due to the fact that more pore space is filled with helium in the powdered specimens, causing a decrease in matrix volume measured, which again causes an increase in matrix densities on powder. This indicates that helium doesn’t reach all the pores in the cored samples, underestimating the porosity. But on the other hand, porosities calculated based on matrix density measurements by the helium pycnometer are assumed to be overestimations of the pore volumes for methane because the kinetic diameter of methane (0.38 nm) (Baker, 2004) exceeds helium (0.26nm) (Chalmers et al., 2012). This means that methane pores will not be able to fill all pores that helium would be able to fill.

![Figure 26: Porosity and matrix density ranges compared to other shale formations](image)

According to Figure 26, the porosity range measured is lower compared to other shale formations, suggesting that the porosities might be less promising. The matrix density range measured shows a wide density variation in the WMF compared to other shales, which is also not favorable. The wide range could suggest variation in mineral composition leading to increased heterogeneity within the formation, complicating gas production. The effect of heterogeneity on hydraulic fracture propagation is explained earlier.

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12 Source other data points: Sone & Zoback (2013)
3.2 XRD / XRF analysis

The main observation from paragraph 2.3 was that the WMF contains high clay content. To check the validity of this observation, the data is compared to data obtained by Dr. Maartje Houben (MH) (Houben et al., 2014). She also analyzed the mineral composition of the WMF, but she used a different technique (Scanning Electron Microscope, SEM). The results she found and the results obtained for the purpose of this thesis are compared. Average values found above and below the Whale Stones (WS) are shown in Table 5 and confirm that the measurements are consistent, both showing high clay content.

<table>
<thead>
<tr>
<th></th>
<th>Average above WS</th>
<th>Average below WS</th>
</tr>
</thead>
<tbody>
<tr>
<td>JL</td>
<td>55.5</td>
<td>60.6</td>
</tr>
<tr>
<td>MH</td>
<td>63.9</td>
<td>63.4</td>
</tr>
</tbody>
</table>

Table 5: Comparison of the mineral composition obtained for this thesis (JL) to the results obtained by Dr. Maartje Houben (MH) above and below the Whale Stones (WS)

Another observation concerns the increase of pyrite close to the WS. Zijp et al. (2014) measured TOC content in the WMF and they also analyzed the corresponding mineral composition. Based on anticipated results, their suggestion was that increased pyrite content corresponds with increased TOC values, therefore the observed increase in pyrite content close to the WS may indicate an increase in TOC relative to the other parts of the WMF section.

Furthermore, the weakness of Basica, the software used to extract mineral composition from the XRF results will be discussed as well as the error related to this issue. The amount of iron detected by the XRF could either come from siderite (carbonates) or goethite (iron oxyhydroxide). In the presented results, the choice fell on goethite because siderite and goethite are both not detected in the XRD analysis conducted at 3ME. Since goethite is an iron bearing hydroxide mineral, it is difficult to be detected by the XRD, while siderite is assumed to be easily detected (Dr. Karl-Heinz Wolf, personal communication, August 2014). Because both siderite and goethite are not detected, goethite is more likely to be the one to be missed because of its structure. The choice to go with goethite, causes an enormous decrease in the amount of carbonates (average of ≈ 2.5x over all of the samples). But the most important minerals (clay, quartz and calcite, combined ≈80% of the shale) don’t change much (max. 1.07% change in all of the samples). So the uncertainty in the quartz and clay content due to this issue is limited.

For shale gas plays, quartz-rich shales are preferred, because those usually are more brittle and have high Young’s moduli, making it prone to natural fractures (Bai et al., 2013). To determine whether the WMF quartz content is in the high or low region, it will be compared to other already developed shales. The WMF data is compared to data from a paper by Bai et al. (2013) and a report by the U.S. Department of Energy (Bruner & Smosna, 2011) which summarized multiple papers on the Barnett and the Marcellus shale.

13 Source other data point: (Houben et al., 2014)
Based on Figure 27, one can predict that the WMF would not be as brittle as other shales because of its very low quartz content and high clay content. This is not favorable for the fraccability of the WMF.

To determine whether there is a correlation between porosity and clay content, these two parameters are plotted against one another in Figure 28.

The figure shows that clay content increases as porosity increases. Though the coefficient of determination \( R^2 \) is very low, the trend is visible. But because of the limited dataset, the relationship between these two parameters is not assumed to be representative. If the dataset is enlarged, a more representative correlation is expected. This would mean that one could for example estimate clay content from porosity logs using the obtained relationship. Marion et al. (1992), who measured velocity and porosity at various confining pressures and at various sand-clay mixtures, also determined that in shale and sandy shale, clay content increases as porosity increases.

\[ y = 0.7136x + 57.171 \]
\[ R^2 = 0.02 \]

Figure 27: Quartz and clay content compared to other shales

Figure 28: Cross-plot of porosity vs. clay content

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14 Source other data ranges: Bai et al. (2013) and Bruner & Smosna (2011)
3.3 Velocity and seismic anisotropy measurements

In paragraph 2.3, the velocity results are presented with possible explanations. As mentioned, a possible explanation for the velocity decrease with increasing stress in Table 2, might be the closure of microcracks and fractures. On average, the maximum stress applied on the different samples is 22 MPa for the vertical samples and 32 MPa for the horizontally layered samples. In this stress range the samples are likely still in the elastic deformation regime. Jones & Wang (1981) and Kaarsberg (1959) explained that for the shales they conducted research on (stress range 0.1-400MPa, thus probably reaching the plastic deformation regime), velocity anisotropy increases with burial depth i.e. increasing bulk density. By examining their samples under a scanning electron microscope (SEM), they confirmed that increased preferred orientation of mineral grains with increasing bulk density causing this phenomenon. In the Whitby samples (stresses up to max. 37MPa), anisotropy decreases with increasing stress. Assuming that bulk density also increases with increasing stress, this is contrary to the results from the last 2 papers. Because their anisotropy increases with increasing stress and the WMF anisotropy decreases with increasing stress, it suggests that the cause of the anisotropy is not the same in both cases. This discrepancy can confirm that preferred orientation is not the dominating cause of anisotropy in the WMF, but rather that microcracks and fractures are the main cause at this achieved stress level (elastic regime).

In Figure 29, anisotropy data from the Whitby shale is compared to data taken from Johnston & Christensen (1995). Their data points comprise measurements on samples obtained from the Chattanooga Shale, the New Albany Shale and the Antrim shale (US shales). To visualize a trend, 3 data point were taken from the Whitby shales (0-10-25 MPa) and the 3 data points available for the other shales (10-50-100 MPa).

Almost all of the measurements show a declining trend with increasing stress. But the Whitby shales show slightly steeper decline in the low stress part, especially for the P-wave data. This might indicate that the Whitby shales contain more aligned fractures and microcracks in the applied stress range, which are closing under stress. Because the samples are taken from an exhumed outcrop which is subjected to weathering, and samples were detached with a significant force from the formation using a hammer and a chisel, it is possible that some of the fractures are artificially created. This might create a distorted view on the WMF.

15 Source other data points: (Johnston & Christensen, 1995)
To determine whether there are correlations between certain petrophysical parameters, cross-plots are generated. To check the velocity dependence on porosity, these 2 parameters are plotted against each other in Figure 30.

**Figure 30: Cross-plot showing porosity vs. velocity**

Figure 30 depicts a positive correlation between porosity and both compressional and shear wave velocity. Note that the vertical axes have the same increments, but different ranges. Because of the scarcity of data points, the obtained relationships are no good representatives of the true data ($R^2$ is low). But as already explained at Figure 28, this relationship is expected to enhance with increased numbers of data points. Such obtained relationships can be handy when data is scarce.

Marion et al. (1992) stated that there is a negative correlation between clay content and compressional velocity. To check whether this holds for the WMF, a cross-plot is generated (Figure 31).

**Figure 31: Cross-plot showing clay content vs. P-wave velocity**

This figure confirms that there is a negative correlation between these 2 parameters with a relatively good fit. Based on this relationship, one might provide an estimate of clay content from compressional velocity logs. But just like all the other obtained relationships, this one should also be confirmed with more data points before it is actually used.
Note that 2 data points in Figure 31, Figure 30 (right) and Figure 28 look like they could be outliers, but the deviation is noticed in clay content and in P-wave velocity measurements. These 2 measurements are conducted independently of one another, therefore these points are assumed to be valid data points instead of erroneous points. These measurements correspond with samples 56 and 70, both close to the WS (56 just above and 70 just below the WS). Paragraph 3.2 also mentioned high pyrite content in this part of the WMF section thus presumably high TOC (Zijp et al., 2014). If this is assumed to be true, it might have possibly caused the deviation from the other data points.

The data is not plotted together with data from other papers (e.g. Marion et al. (1992)) because it was difficult to find data that is produced under the same conditions as this study does. Most of the data results from saturated samples or sandstone samples, which are no good representatives of the measured conditions of this study.

3.4 Attenuation and attenuation anisotropy measurements

Paragraph 2.4 mentions the strange increase in attenuation at low stresses noticed in Figure 22, but does not explain this. It is possible that small cracks are induced because of the applied stress. When the sample is placed in the set-up and ready for measurements (stress is close to zero), the pressure bench is halted for about 5 minutes to let the sample settle. Cracks might be formed in the meantime, which are closed again as stress increases. According to Alimzhan Zhubayev (personal communication, October 2014), another possible explanation can be found in the processing technique itself. Uncertainty in calculation of the spectral ratio is high at low stresses.

An important observation from the S-wave results in Figure 23 was that 5 out of 6 data points taken at maximum stress, showed that attenuation perpendicular to the bedding is larger than attenuation parallel to the bedding. According to Zhubayev & Barnhoorn (2013) and Lucet & Zinszner (1992), the increased values can result from scattering attenuation due to heterogeneities like the presence of fine layers (≈1-3 mm), which are of the same order of dimension as the wavelength of the S-wave in the experiment (≈3mm). At the same frequency, wavelength of the P-wave is greater than S-wave wavelength, therefore the P-wave is not influenced by scattering, but S-wave is. With average maximum stresses ranging from 22 to 32 MPa, the samples might still contain cracks. This hypothesis can be confirmed or rejected by subjecting horizontally layered samples to even higher stresses. For some of the samples, it was clear that the maximum applied stresses could be increased further. But for the purpose of this thesis, a pressure bench was used with a maximum achievable force of 50 kN. Alimzhan Zhubayev (personal communication, May 2014) tested 1 horizontally layered sample on another pressure bench, reaching more than 50 MPa before completely breaking the sample. So there is a certain possibility that there are still cracks present in the sample at the maximum applied stresses. The fact that there is no increase in attenuation noticed at the maximum applied stresses, also supports this (no new fractures created yet).

Another important observation from Figure 23 was that compressional anisotropy is lower than shear anisotropy. Table 6 confirms that 4 out of 5 samples show this trend. Figure 32 shows the data in a graph compared to data collected by Zhubayev and Barnhoorn (2013).
Anisotropy

<table>
<thead>
<tr>
<th></th>
<th>Min. stress</th>
<th>Max. stress</th>
</tr>
</thead>
<tbody>
<tr>
<td>P-wave S-wave</td>
<td>0.27 -0.42</td>
<td>-0.11 -1.15</td>
</tr>
<tr>
<td>P-wave S-wave</td>
<td>0.21 0.42</td>
<td>0.33 0.65</td>
</tr>
<tr>
<td>P-wave S-wave</td>
<td>0.21 0.16</td>
<td>-0.29 0.11</td>
</tr>
<tr>
<td>P-wave S-wave</td>
<td>-0.38 -0.68</td>
<td>0.40 0.54</td>
</tr>
<tr>
<td>P-wave S-wave</td>
<td>-0.12 -0.88</td>
<td>-0.06 0.78</td>
</tr>
</tbody>
</table>

Table 6: Anisotropy values for P- and S-waves at min. and max. stress

Lucet & Zinszner (1992), who conducted attenuation measurements on limestone and sandstone samples, explained that shear wave splitting may account for this. They explain that this occurs when an arbitrary polarized shear wave entering an anisotropic medium, splits into 2 polarizations. Each of these 2 phases travels with a different velocity. At the other end of the sample, these are combined into another shear wave that can have a different shape (frequency content and amplitude) and has lost energy, causing an apparently strong attenuation in the results (Lucet & Zinszner, 1992).

Another explanation is that S-wave scattering causes the increase in S-wave anisotropy.

The negative values in Table 3 indicate that attenuation // to the bedding is larger than attenuation ⊥ to the bedding, which is already explained earlier.

Because of the extremely friable nature of shale and consequently the difficulty to prepare suitable samples (Jones & Wang, 1981), there are very few laboratory measurements of attenuation on shale. Most papers elaborate on attenuation measurements conducted on limestone and sandstone (Lucet & Zinszner, 1992; Toksöz et al., 1979; Best et al., 2007).
Table 7: Attenuation values from papers to compare to Whitby shale values

<table>
<thead>
<tr>
<th>Sample</th>
<th>Stress [Mpa]</th>
<th>P-wave</th>
<th>S-wave</th>
</tr>
</thead>
<tbody>
<tr>
<td>Whitby</td>
<td>0</td>
<td>0.076</td>
<td>0.061</td>
</tr>
<tr>
<td></td>
<td>32</td>
<td>0.044</td>
<td>0.033</td>
</tr>
<tr>
<td>Barea Sandstone</td>
<td>0</td>
<td>0.050</td>
<td>0.045</td>
</tr>
<tr>
<td></td>
<td>35</td>
<td>0.010</td>
<td>0.009</td>
</tr>
<tr>
<td>Sandstone</td>
<td>5</td>
<td>0.042</td>
<td>0.071</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>0.011</td>
<td>0.024</td>
</tr>
<tr>
<td>Limestone</td>
<td>5</td>
<td>&lt;0.005</td>
<td>0.050</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>0.008</td>
<td>0.010</td>
</tr>
</tbody>
</table>

The values in Table 7 show a comparison of other rock types to the Whitby shale values. None of the other examples show higher attenuations than the Whitby samples. This seems reasonable, because none of the other mentioned rocks are expected to contain as many fine layers and cracks oriented // to the bedding as shale has.

### 3.5 Thomsen’s anisotropy parameters and elastic properties

The main observation from Figure 24 was that the high values for $\varepsilon$ and $\gamma$ were caused by the fine layering in shale. To support this conclusion, literature was consulted. Best et al. (2007) also found that that $\varepsilon$ and $\gamma$ are higher in rocks with visible lamination. They found values ranging from 8 - 40% measured on sandstones with clay lamination, strongly laminated siltstone and sandstone with clay lenses, while visually isotropic sandstones and limestones show very low values in the range -5 – 2%. Since almost all of the computations result in $\varepsilon, \gamma > 0.4$, the Whitby shales are classified as highly anisotropic.

Another observation was that almost all calculated $\delta$ values are negative, but this will be discussed later.

The lower 3 charts of Figure 24 show $\varepsilon, \delta$ and $\gamma$ at maximum applied stress. Zhubayev & Barnhoorn (2013) explained that the significant elastic anisotropy they measured in the WMF, could be due to the fact that the experiments were conducted at ambient conditions, where a considerable amount of open fractures could influence the results. Comparing the self-generated results at minimum stress to the results at maximum stress (lower 3 charts of Figure 24), one can conclude that stressing the sample did have much influence on the parameters. Table 3 shows that $\varepsilon$ and $\gamma$ values decrease with increased stress, but since Thomsen (1986) defined the weak-to-moderate anisotropy range as $\varepsilon, \delta$ and $\gamma <0.2$, the WMF samples are still highly anisotropic at the maximum measured stresses. It is not clear from literature how $\delta$ is expected to change with applied stress (Alimzhan Zhubayev, personal communication, October 2014). In the WMF samples, the values decrease as well as stress values are increased ($\delta$ becomes more negative).

To place the WMF in the bigger picture with other shales of current interest, the Thomsen’s parameters are plotted together with data from various papers in Figure 33.

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16 Source other data points: Barea Sandstone (Toksöz et al., 1979); Sandstone #175a and limestone 184 (Best at al., 2007)
As already concluded, this figure also shows that $\varepsilon$ and $\gamma$ are very high. The values coincide with the highest values measured for the other shales. Another point to be noted is that almost all of the experiments on the WMF resulted in negative $\delta$ values, while most of the other shale results presented in Figure 33 show positive values. Because clay minerals (illite, chlorite and kaolinite) are characterized by negative or small positive $\delta$'s (Sayers, 2005), a possible explanation can be the presence of clay minerals. Because of the high clay content (XRF analysis showed a minimum of 47% in all samples) in the WMF samples, the negative $\delta$'s might be consistent.

Sayers (2005) also attempted to explain why some results in papers contain such large positive $\delta$ values. He found that $\delta$ is sensitive to (1) the contacts between clay particles and (2) to the degree of disorder in the orientation of the clay particles. (1) When the ratio of normal to shear compliance of the contact areas reaches a critical value, $\delta$ increases due to the presence of these regions. This may cause the $\delta$ sign to change to positive, but will also result in much larger values of $\varepsilon$ and $\gamma$ (Sayers, 2005). Since the 2 positive $\delta$ values do not correspond with higher $\varepsilon$ and $\gamma$, this is likely not the case for the WMF. (2) He also mentioned that misalignment of clay particles might be another possible cause for the positive $\delta$ values and that this shouldn’t lead to increased $\gamma$ and $\varepsilon$. This explanation sounds more reasonable for the WMF results. For further explanation of these mentioned reasons, the paper by Sayers (2004) which elaborates on this, can be consulted.

The high anisotropy values are not favorable for production because as mentioned in the introduction, increased anisotropy complicates the hydraulic fracturing process (Waters et al., 2011; Zhubayev & Barnhoorn, 2013). But it is important to know these values, because the high anisotropy values can be taken into account during exploratory research. According to Thomsen (1986), $\delta$ is the only anisotropy parameter necessary to understand important features in the seismic analysis, e.g. the difference between small-offset NMO (Normal Moveout) velocity and vertical velocity (a short explanation is provided in APPENDIX E).

To try to predict anisotropy, it is plotted against porosity and clay content. These 2 parameters are chosen because according to literature, there is a correlation between the characteristics. According to Wang (2002), anisotropy in shales should be affected by porosity. He explains that young shales usually contain high porosity and tend to be less laminated, while older, i.e. compacted shales have lower porosity and clay platelets which are preferentially oriented, leading to increased anisotropy. To determine whether the porosity measurements can give an indication of anisotropy in the WMF, the Thomsen’s parameters are plotted versus porosity in Figure 34.

![Figure 34: Anisotropy related to porosity](image)

As predicted (Wang, 2002), there a declining trend visible for ε and γ, but the number of measurements is too small to try to predict anisotropy from porosity.

![Figure 35: Cross-plot gamma vs. clay wt% for the WMF (red) and the Barnett shale (blue)\(^1\)](image)

Figure 35 shows a cross-plot between Thomsen’s gamma and clay wt%. The blue data points are measurements on the Barnett shale (mineralogy from a geomechanical logging tool) (Waters et al., 2011). According to Waters et al. (2011), it is common to have a correlation between these 2 parameters in organic shales. It shows that clay content is important in the development of transverse isotropy. Higher clay content

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\(^{18}\) Blue data points (Waters et al., 2011)
causes higher anisotropy, i.e. higher \( \gamma \). But in the WMF there is no inclining trend visible. It is clear though that the Whitby shales contain much more clay compared to the Barnett and that the samples are highly anisotropic.

Figure 25 shows the dynamic \( E \) and \( \nu \) results. An important point noted from this figure was that Young’s modulus parallel to the bedding always exceeded Young’s modulus normal to the bedding. A possible explanation for \( E_1 > E_3 \) (Stress_1 \( \times \) Strain_1 > Stress_3 \( \times \) Strain_3) is that if stress is applied normal to the bedding (Stress_3), the sample deforms (shortens) more (Strain_3 > Strain_1) due to the closure of cracks, leading to lower \( E_3 \). Another point noted from the figure concerns the Poisson’s ratios. \( \nu_{31} > \nu_{12} \) indicates that a horizontally layered sample that is stressed normal to the bedding, deforms more in radial direction than a vertically layered sample stressed parallel to the bedding. Ravestein (2014) whose static results show the same trend, explained that horizontally layered samples show more radial strain due to the layering itself. The friction of the contact with the metal on the top and bottom surface is only valid for the upper and lower layer of the sample. The other layers in the sample are not subjected to friction, causing them to deform easily in radial direction. Vertically layered samples are all in direct contact with the upper and lower metal surface causing friction on all of the individual layers. These samples deform differently, most radial deformation is only in the middle of the sample. Not close to the edges where friction is very high.

Again, to place the WMF in the bigger picture with other shales, the Young’s moduli and Poisson’s ratios are plotted together with data from various papers in Figure 36.

![Figure 36](image)

This figure shows both static and dynamic values of different shales compared to WMF shales. It is clear that for most shales \( E_1 > E_3 \), and that there is no clear correlation for the Poisson’s ratios. The complete dataset of the WMF shales (combined with data from Zhubayev and Barnhoorn (2013)) however nicely show \( \nu_{31} > \nu_{12} \) for almost all of the measurements. This is also predicated by Sayers (2013).

Compared to other shales, \( E \) is not in the high range. This is not favorable for the rock’s ability to maintain the created fractures (Rickman, 2008). \( \nu_{31} \) is somewhat high but definitely not in the top range and \( \nu_{12} \) is in the lower range of the dataset. This is favorable for the rock’s ability to fail under stress (brittle) (Rickman, 2008).

\(^{19}\) Modified from (Sayers, 2013)
3.6 Integration of all results

In this paragraph, the discussed results will be combined for better understanding of the WMF section. Based on the integration of the results, an attempt is made to point out which part of the section is the most promising for shale gas exploration. The number of data points is limited, so the interpolation between these points contains large uncertainty. Because data gathering is time consuming, a limited number of measurements have been performed.

According to Britt & Schoeffler (2009), a prospective shale contains among others less than 40% clay, has dynamic to static Young’s Modulus ratios that correlate with clastic reservoirs and looks isotropic on core scale thus only few laminations should be visible. The WMF does not satisfy these criteria since the mineral composition analysis showed a minimum clay content of 45% in all measured samples and lamination is clearly visible with the naked eye, as depicted in Figure 17 (below the fracture). The highest measured dynamic E is 32 GPa (Table 4) and according to Ravestein (2014) the highest static E measured is 17 GPa. If this optimum value is compared to Britt & Schoeffler’s (2009) range, it correlates with the lowest possible prospective shales.

The results generated for the purpose of this thesis will be combined in Figure 37 and Figure 38 to determine whether there is a correlation with height in the section and which part of the section proves to hold the best prospective for gas exploration.

A note on the Brittleness Index graph visible in Figure 37 (4th from left) and Figure 38 (2nd from left): Paragraph 1.6 explains that it is possible to calculate BI from E and ν, but that this would not be used in this thesis. However, only to elaborate on which part of this section is more “fraccable” (combination of high E and low ν), the dynamic BI is calculated and plotted versus height in the WMF section. BI is calculated as implied by Rickman (2008):

\[
V_{\text{Brittle}} = \frac{V - V_{\text{max}}}{V_{\text{min}} - V_{\text{max}}} \\
E_{\text{Brittle}} = \frac{E - E_{\text{min}}}{E_{\text{max}} - E_{\text{min}}} \\
BI = \frac{E_{\text{Brittle}} + V_{\text{Brittle}}}{2}
\]

In the performed calculations, \(E_{\text{min}}\) and \(E_{\text{max}}\) correspond to the minimum and maximum measured Young’s moduli and \(V_{\text{min}}\) and \(V_{\text{max}}\) correspond to the minimum and maximum measured Poisson’s ratios on WMF samples for this thesis.

According to Rickman (2008) BI gives an indication of the distinction between brittle and ductile shale. The higher BI is, the higher the odds that the shale is brittle. Furthermore, brittle shale is more likely to comprise natural fractures and is also expected to develop more fractures when hydraulically stimulated (Rickman, 2008). For extensive explanation regarding this topic, consult Thomas Ravestein’s thesis (Ravestein, 2014). Dynamic BI is calculated using the dynamic ν and E (from ultrasonic experiments) instead of static, as his thesis did.
Comparison of all the obtained data, leads to the conclusion that there might be a slight trend in the data. It is not visible in all characteristics, but in a lot of them, it is clear that there is a change towards the Whale Stones.

![Figure 37: A trend towards the Whale Stone?](image)

There is no clear explanation for the correlations among the characteristics. As matrix density decreases, velocity is expected to decrease as well, which is visible in Figure 37. But low clay content is expected to correlate with low BI (low E resulting from high clay content). This is not the case in Figure 37. Furthermore, Zijp et al. (2014) also noticed a trend towards the WS. Their spectral gamma ray log at Port Mulgrave shows that Potassium and Uranium values decrease towards the WS, causing a decrease in his total gamma ray towards the WS. This seems to correlate with the decrease in clay content detected by the XRF measurements from this thesis.
Clay content, quartz content and BI give an indication of the fraccability of the rock, i.e. its ability to form fractures when stimulated. Based on changes in the characteristics, the section is divided into four subsections. But from these subsections, none of them prove to be favorable for all characteristics. Some of them even turn out to be contrary to the expectations, e.g. the sections with the highest BI have the highest clay content (section 4) and vice versa. But because BI only incorporates E and ν, this is possible. Pointing out which part of the WMF is most favorable for gas exploration, is not straightforward. Based on BI and porosity, subsection 1 and 4 would be most favorable. S-wave anisotropy also shows slightly lower values in these parts of the WMF section. Though they have the highest

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20 Some of the P- and S-wave anisotropy data points and Brittleness Indices (ν and E) are modified from (Zhubayev & Barnhoorn, 2013)
P-wave anisotropy and clay content, there are no other parameters than porosity and BI that proof to be decisive. Therefore a cautious conclusion can be drawn from this data that the upper and lower most part of the WMF are more favorable for gas exploration. Because of the high clay content in the lowest section, the upper section (section 1) is preferred. But this is relative, because compared to other shales, most parameters don’t proof to be favorable. Thomas Ravestein, who focused more on the fraccability of the shales, shows that the best parts of the section for hydraulic fracturing are indeed the upper and lower most parts of the WMF.

If pyrite content results are incorporated as well as a suggestion by Zijp et al. (2014) that an increase in pyrite content corresponds with higher TOC, section 2 and the upper part of section 3 might be more favorable, especially since they also exhibit lower clay content. Paragraph 3.3 also mentions a deviation in the data points close to the WS. But Zijp et al. (2014) mention that the results they present are anticipated results and that samples are currently being analyzed further. Furthermore, TOC content was not measured for the purpose of this thesis, thus this thesis cannot confirm that pyrite content correlates with TOC. This is just based on the suggestion by the mentioned paper. For these 2 reasons, this conclusion is not chosen as the decisive one to base the final favorable section choice upon. But this should certainly be kept in mind.

Comparison of the 4 obtained subsections to the stratigraphic column by Linde van Laerhoven shows that the subsections more or less coincide. The first boundary is set at the Curling Stones, the second at the Whale Stones and the third slightly below the Canon Ball Doggers. Figure 39 depicts the same graphs as in Figure 38, but this time compared to Linde Laerhoven’s stratigraphic column.
Figure 39: Subsections compared to Linde van Laerhoven’s (TNO) stratigraphic column of the WMF\textsuperscript{21}

\textsuperscript{21} Modified from Linde van Laerhoven (TNO)
Conclusions

- Obtained helium pycnometer porosities of the WMF vary from 0.26 to 5.82%. There is no correlation between porosity and height in the WMF, the data is scattered. The measured range is low compared to porosities from other shales, which is not favorable for gas exploration.

- Matrix densities measured on cores vary between 2.25 and 2.55 g/cc while matrix densities measured on powdered samples vary between 2.21 and 2.8 g/cc. Matrix densities above the WS seems to be more clustered than below the WS indicating that the upper part of the section might be more homogeneous than the lower part. But this trend is not visible in other characteristics.

- Mineral composition extracted from XRF results indicate high clay content and low quartz content. Dr. Maartje Houben (UU) confirmed the high clay content using a SEM. Another conclusion from these results is that pyrite content increases close to the WS. Zijp et al. (2014) suggest that this correlates with an increase in TOC content. Comparison of the quartz and clay content to other developed shales, indicates that the WMF has high clay content and low quartz content, which is not favorable for the fraccability of the rock.

- Seismic anisotropy analysis shows that the WMF is highly anisotropic with values up to 36%.

- As small amounts of stress are applied, certain properties such as velocity, velocity anisotropy and attenuation anisotropy show enormous changes (in the beginning). This indicates that the WMF contains many microcracks and fractures which are closing as stress is applied. Because the WMF samples are taken from an exhumed outcrop which is subjected to weathering and samples were detached from the formation using a hammer and a chisel, it is possible that some of the fractures are artificially created. This might create a distorted view on the WMF.

- Compressional attenuation exceeds shear attenuation in most of the measurements. According to Toksöz et al. (1979) this is expected in dry rocks.

- At maximum applied stresses most samples display higher shear attenuation measured normal to the bedding compared to values parallel to the bedding. According to literature, this is caused by scattering attenuation due to the presence of heterogeneities (layering) which are of the same order of dimension as the wavelength of the S-wave.

- Compressional attenuation anisotropy is lower than shear attenuation anisotropy. Lucet & Zinszner (1992) explained that shear wave splitting may cause the S-wave to lose more energy because it splits into 2 polarizations. This increases S-wave anisotropy with respect to P-wave anisotropy.

- Because of the extremely friable nature of shale and consequently the difficulty to prepare suitable samples (Jones & Wang, 1981; Alimzhan Zhubayev, personal communication, October 2014), there are very few laboratory measurements of attenuation on shale. This complicates the comparison of the WMF data to other shales.

- The Thomsen’s parameters indicate that the WMF is highly anisotropic. Other WMF data (Zhubayev & Barnhoorn, 2013) confirms this. As expected, $\varepsilon$ and $\gamma$ decrease with increased applied stress (anisotropy decreases), but the WMF is still highly anisotropic at maximum applied stress. Compared to other shale samples, the WMF samples are in the highest anisotropy regime. This is not favorable for gas exploration, but if the values are known beforehand, they can be incorporated in calculations.
Compared to other shales, the results of the Young’s modulus turn out to be in the low range while Poisson’s ratios turn out to be in the low to intermediate range. According to Rickman (2008), the low E values are not favorable for the rock’s ability to maintain the created fractures, but the low to intermediate ν’s are favorable for the rock’s ability to fail under stress (brittle behavior).

Integration of all analyzed data leads to the conclusion that the WMF is highly anisotropic and that no horizon proves to be obviously favorable for gas exploration. The WMF can be divided into 4 subsections based on changes in the obtained characteristics. But since none of the 4 subsections exhibits obviously favorable characteristics for gas exploration, the choice is based on BI and porosity, which prove to be the most decisive. The upper and lower most part of the section turn out to be the most promising. Because of the high clay content in the lowest section (section 4), the upper section (section 1) is preferred out of those two. Thomas Ravestein also confirmed that the upper and lower part of the section are the most fraccable. But this is relative, because overall, the fraccability of the most optimistic value of the dynamic to static E ratio, correlates with the lowest possible prospective shales (Britt & Schoeffler, 2009). Petrophysical properties show heterogeneity, which is not promising either. These pessimistic results might be a distorted view caused by the many induced fractures and weathering. But one thing is clear, this time analogue of the Posidonia Shale Formation is no Barnett equivalent.

Low clay content and high pyrite content (presumably corresponding with high TOC (Zijp et al. 2014)) are detected in section 2 and the upper part of section 3 (close to the WS). This might indicate that these 2 sections are more favorable based on composition. But this conclusion is not chosen as the decisive one because it is only based on anticipated results from Zijp et al. (2014) and this thesis does not contain TOC values to confirm it. But it should certainly be kept in mind.
Recommendations

In this chapter, recommendations based on the progress and results of this thesis will be presented.

- If extra samples will be collected in the future, it may be worth to try to use other methods to get the samples out of the outcrop instead of using a chisel and a hammer, which may cause extra fractures, creating a distorted view.

- Despite efforts made to preserve the cores in their original state, they still dried out. In the future, maybe cores should be stored in climate rooms, for better preservation. Dried cores may influence the number of microcracks which again influences the anisotropy of the material, creating a distorted view.

- The number of measurements is limited because of the time consuming process of data gathering. More measurements would definitely enhance the quality of the dataset and could yield better trends. An example from this thesis is the estimation of Thomsen’s parameters from porosity. Due to the limited amount of data points, it is not very clear. If this dataset could be amplified, seismic anisotropy might be estimated from porosity. Such equations can be of great use when there is no actual data available, like in the Dutch Posidonia shales. If porosity is available from logs through the shales, seismic anisotropy can be estimated using the time analogue WMF data.

- To mimic downhole conditions even better, a set-up with P- and S-wave transducers that applies confining stress, should be considered. Moreover, if both the P- and S-wave transducers could be mounted on the sample at the same time, conducting experiments would be twice as fast.

- To determine whether anisotropy of the WMF is the result of preferred orientation of clays (illite, at least 20% in all the samples as confirmed by the XRF analysis), the degree of clay mineral alignment in shales can be constrained by orientation indices produced using XRD analysis (Johnston & Christensen, 1995). According to this paper, there should be a strong positive correlation between the degree of preferred orientation (orientation indices) and seismic anisotropy. If this can be confirmed for the Whitby shales, the cause of the anisotropy can be determined more accurately.
References


APPENDIX

APPENDIX A: Porosity and matrix density measurements

Figure 40: Samples used for porosity and matrix density measurements on cylindrical samples
Figure 41: Samples used for matrix density measurements on powdered samples and XRF analysis
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Table 8: Porosity and grain density (powdered and cylindrical specimens) with height in the Whitby section. Grey part below the WS.
Figure 41 also shows the depths of the samples XRF analysis was conducted on.

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Table 10: XRF results of samples 95, 70, 80, 29
### Table 11: XRF results of samples 3, 22 and 45

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<th>wt%</th>
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### Table 12: Mineral composition obtained using the XRF results in software Basica

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<th>MONTMORILLON.</th>
<th>QUARTZ</th>
<th>CALCITE</th>
<th>PYRITE</th>
<th>GOETHITE</th>
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APPENDIX C: Velocity and seismic anisotropy measurements

Figure 42: Velocity and seismic anisotropy measurements conducted on samples taken from various heights in the section.
Figure 43: Samples used for anisotropy measurements with corresponding colors as used in the results section
Figure 44: S- and P-wave attenuation measured // to the bedding

Figure 45: S- and P-wave attenuation measured at a 45° angle to the bedding
APPENDIX E: Thomsen’s anisotropy parameters and elastic coefficients

This information is taken from Wikipedia:
In reflection seismology, normal moveout (NMO) describes the effect that the distance between a seismic source and a receiver (the offset) has on the arrival time of a reflection in the form of an increase of time with offset. From an equation, it is possible to calculate the velocity when the offset and two-way times at zero and non-zero offset are known. This velocity is the NMO velocity and can be used to remove the effect of offset on the travel times (as shown in the diagram below). For further explanation: http://en.wikipedia.org/wiki/Normal_moveout

Figure 46: NMO correction

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22 From Wikipedia