Measuring Elastic Properties to determine the influence of TOC on Synthetic Shale Samples

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INTRODUCTION

Organic shale is a fine-grained sedimentary rock that is known to have a rich source of natural gas trapped inside (Boyer et al, 2006). Such rocks existing as unconventional natural gas reservoirs are challenging to characterise. The elastic properties of shale rock vary significantly within and across reservoirs due to variable mineral composition and fabric anisotropy exhibited by these organic rich shales (Zhu et al, 2011). Presently our understanding of their dynamic and elastic behaviour is very limited. Experiments involving organic shale core samples are rare but can be found in Dewhurst et al, 1998; Dewhurst and Siggins, 2006; Dewhurst et al, 2011; Delle-Piane et al, 2011. One of the difficulties is that organic rich shales are chemically and mechanically unstable, due to the organised distribution of platy clay minerals (Zhu et al, 2011) and compliant organic materials (Vernik and Nur, 1992; Vernik and Liu, 1997; Sondergeld et al, 2000; Vernik and Milovac, 2011). There are also suggestions in literature that it is not only the amount of clay or organics affecting the anisotropy in organic rich shales, but also the maturity of the shales (Sone, 2012).

It is difficult to account for all the factors affecting the velocity and anisotropy of a real shale sample. In particular, factors including stress state, stress history, clay mineral content, and TOC content are not widely reported (Bohacs et al, 2005; Passey et al, 2010). In previous laboratory experiments we have studied the effects of the stress, lithology and TOC on wave velocities using real shale core samples (Altowairqi et al., 2012). Such experiments are difficult to control and are dependent on the sample quality. In contrast, synthetic shale samples provide us with the opportunity to conduct investigations in a fully-controlled environment where variability of parameters is controlled by the user. We can thus produce core samples with known percentage of clay minerals, non-clay minerals and TOC content under variable and/or in-

SUMMARY

This paper describes the factors that control elastic properties of organic shale, which is crucial for exploration and successful gas production from unconventional reservoirs. Mechanical and dynamic elastic properties are main shale characteristics that are not yet well understood as there have been a limited number of investigations involving organic rich shale samples. Synthetic shale core samples whose clay mineralogy, non-clay mineral content and Total Organic Carbon (TOC) content are known can be used to study variations of elastic parameters in a controlled experimental environment including in-situ stress conditions.

More than 20 synthetic shale samples were created for our investigations under reservoir stress conditions with different mineral composition and TOC percentage. Ultrasonic transducers were used to measure body wave velocities, which were then used to calculate the elastic properties of different shale samples. The results demonstrate that P- and S-wave velocities vary with changing TOC under isotropic stress conditions. It is shown that the velocities of P-and S-waves are inversely proportional to TOC content. In addition, the increase in the TOC produced a decrease in density from approximately 2.4 g/cc to 2.15 g/cc and increase in porosity from approximately 16% to 20%.

Key words: Organic shale; Synthetic shale; TOC; Ultrasonic waves
The primary objective of the research reported here was to investigate the effect of variable TOC on elastic properties of synthetic shale. More than 20 synthetic shale samples were manufactured under reservoir stress conditions with similar mineral compositions but different TOC percentages resembling those found in the field. Specifically, the impact of TOC of gas-shale samples and the resultant impact on elastic wave velocities were evaluated.

MANUFACTURE OF SYNTHETIC SHALE

Synthetic samples were prepared with pre-defined properties to enable systematic investigations of their elastic properties as a function of TOC or some other factors such as mineral composition. Thus such highly controlled “environmental” conditions enable us to investigate in a quantitative sense, the effect of TOC content alone on the shale elasticity under reservoir stress conditions.

As such measurements are rare the impact of the organic matter on the shale elasticity is currently not well understood. Most reports have been about manufacturing synthetic sandstone samples by different methods (Sherlock and Siggins, 2003). In this work, a number of synthetic shale samples were made using clay, quartz and calcite but with different TOC percentages. Zhu et al., (2011) noted that gas-shales usually consisted of different types of minerals such as clay, quartz, calcite, dolomite and pyrite which generally form intricate and perhaps an anisotropic matrix of rocks. More than 70 samples were created using different methods over two years of studies to create representative synthetic shale samples. In this process a method was developed to manufacture synthetic shale samples, having the correct weight to density ratio and the best mix of minerals to produce a homogeneous media. To achieve this, a mix of clay and non-clay minerals was compressed with organic matter under high pressure inside a steel core holder.

The composition of synthetic shale samples used in our experiment had the same composition of a typical gas-shale formation found in the Perth Basin Arrowsmith-2 well and Redback-2 well (Well Completion Report, 2011). This composition combined with a mix of clay minerals and non-clay minerals, and 15% cement plus water was poured into steel cylinder casts designed to be used for high-pressure (8000 PSI) applications. Each synthetic sample was formed under high pressure using a True-Triaxial Loading Frame, to produce the type of simulated core shown in Figure 1. True-Triaxial Loading Frame contains control system and Digital displays, which can help to control a force at constant rate or at constant pressures. Almost 7000 PSI of hydraulic pressure was applied by oil driven hydraulic pump. The first stage of preparing the samples was to mix a fine powder using a mixer machine having 45% clay minerals (kaolinite), 40% non-clay minerals (quartz), and 15% cement with some water. Organic matter was added to the mixer minerals with different percentages for each sample. Petrographic analysis results for the organic material used as TOC indicated a mixture of vitrinite (71%) and Semi-Fusinite (13%) and some other minor components. The of organic material is about 1.4 g/cc

The second stage in the process was to compact the mix of minerals into the steel cylinder and compress it using a solid cylinder at constant rate (3 mL/min) until the pressure rich to 7000 PSI. The equation used to calculate the (total-pressure) which had been applied to the sample is:

\[ \text{Total pressure} = \text{Pc} \times 3.976/\text{Ac} \]

where \( \text{Pc} \) is the hydraulic energy and \( \text{Ac} \) is the area of the cylinder.

The next stage was to keep the constant pressure of 7000 PSI for one day to fully compact each sample. In the last stage, the sample would be removed from the cylinder, and the weight and volume were measured. The samples’ bulk density and porosity for both compositions were in the range of 2.15 g/cc to 2.4 g/cc and 16% to 20%, respectively.

In addition test we have created another group of synthetic samples using the same preparation, but with different composition combined with a mix of 40 % of clay minerals and 30 % of non-clay minerals, and 30% of cement plus water. Organic matter was added to the mixer minerals with different percentages for each sample. All the samples were tested ultrasonically to obtain the elastic properties.

ELASTIC PROPERTIES OF SYNTHETIC SHALES

Organic shales are characterised by low velocity and low density (Vernik and Nur 1992; Vernik and Liu 1997). Many shale-gas formations share properties, including a low matrix porosity and low permeability (Vernik et al, 2010). This experimental technique was chosen since it allowed a fast ultrasonic measurement to meet our objectives. The objective
was to investigate the variation of P- and S-wave velocities as a function of TOC percentage. Initially we measured ultrasonic velocities at atmospheric pressure without confining pressure as first stage.

Figure 1. (A) Steel cylinder casts designed to for high pressure use, (B) the mineral mix as a fine powder, (C) True-Triaxial Loading Frame used to create synthetic shale, and (D) Synthetic shale core samples with different percentages of TOC.

We measured P-wave, S_H and S_V for more 20 samples for two different minerals composition. While P-wave arrivals were clean the S-waves were less easy to pick. In the first composition that is mixed with 45% clay minerals (kaolinite), 40% non-clay minerals (quartz), and 15% cement. The velocities of the P- and S-waves show a smooth decrease as the amount of TOC weight percentage of the synthetic shale was increased in Figure 2. P-wave decreased from 2520 m/s in sample having 0.0 %wt of TOC to 1800 m/s in sample having 20%wt of TOC. The S-wave decreased from 1650 m/s to 1165 m/s as the TOC percentages increased.

Figure 2. Vp and Vs versus TOC% for a number of synthetic shale cores as the TOC percentage increased, the velocities decreased.

In the second composition that is mixed with 40% clay minerals (kaolinite), 30% non-clay minerals (quartz), and 30% cement. The velocities of the P- and S-waves show a smooth decrease as the amount of TOC weight percentage of the synthetic shale was increased in Figure 3. P-wave decreased from 2020 m/s in sample having 0.0 %wt of TOC to 1800 m/s in sample having 20%wt of TOC. The S-wave decreased from 1100 m/s to 850 m/s as the TOC percentages increased.

Figure 3. Vp and Vs versus TOC% for a number of synthetic shale cores as the TOC percentage increased, the velocities decreased.

A comparison between the ultrasonic waves and the total organic matter percentages suggested that at high TOC, all synthetic shale samples showed a decrease in their P- and S-wave velocities. In the both compositions, the increase in the TOC corresponds to a polynomial decrease in density from 2.32 g/cc to 2.05 g/cc, and form 2.4 to 2.15 as shown in Figure 4. Vernik and Milvaco (2011) showed a similar relationship between bulk density versus TOC of core sampled from organic shale formations worldwide superimposed by different models for 5% porosity and 10% porosity. Also, the porosity of the synthetic shale samples was in the range of 16% to 20 % as the TOC percentage changed.

Figure 4. Density reduced with an increase in TOC%. The first composition (red) reduced from 2.3 (g/cc) to
2.05 (g/cc) and the second composition (blue) reduced from 2.4 to 2.15.

The ratio of Vp/Vs decreased for both compositions, from approximately 1.6 to 1.5 over the 0% to 20% of the TOC percentages used in first composition, and decreased from 1.8 to 1.5 over the 0% to 20% of the TOC percentages in second composition (Figure 6). The dynamic Poisson’s Ratios calculated from these synthetic shale samples decreased also from approximately 0.2 to 0.1 as the TOC percentage increased from 0% to 20% in first composition, and decreased from 0.29 to 0.24 as the TOC percentage in second composition (Vernik and Nur, 1992; Vernik and Liu, 1997; Sondergeld et al, 2000; Vernik and Milovac, 2011).

![Vp/Vs & TOC graph](image)

**Figure 6.** Vp/Vs ratio reduced with increasing TOC%.

**CONCLUSIONS**

The discussion here has been limited to the impact on geophysical properties of TOC content when all other factors are constant. Because organic matter has lower velocities and gas-shale densities are expected to be lower than shales containing other percentages of minerals like quartz and calcite, the acoustic impedance trend tends to reduce. Our studies show that organic matter has a significant impact on the elastic properties of synthetic shale samples. Organic matter as well as other mineralogy (e.g., clay, quartz and silica) strongly affects the strength of the rock for hydraulic fracturing applications, and this is reflected in the fact that Young’s Modulus, Poisson’s Ratio and Vp/Vs are strongly affected by in-situ rock parameters including the amount of TOC content and mineral compaction. These results demonstrate that synthetic shale rocks having controlled levels of TOC, clay minerals and non-clay minerals are suitable for testing some of the fundamental geophysical relationships between physical properties in a controlled and systematic manner without the scatter that is inherent in measurements with natural gas-shale rock.

**REFERENCES**


16th Geophysical Conference and Exhibition, Adelaide, Australia, February 2003.

Sone, H., 2012, Mechanical properties of shale-gas reservoir rocks and its relation to the in situ stress variation observed in shale-gas reservoirs, Stanford University, SRB volume 128.


