A Seismic-Frequency Laboratory Study of Solid Substitution in Bentheim Sandstone

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Abstract

Studying the effects of the solid pore fills on the elastic properties of rocks is of significant importance for a range of applications, such as, for example, monitoring of heavy oil reservoirs or modeling the transport properties of the rocks affected by salt precipitation or cementation. We present the results of the laboratory solid-substitution experiments carried out on Bentheim sandstone at a seismic frequency of 2 Hz. The experiments were performed when the pore space of the tested sample was filled sequentially with solid (23°C) and liquid (40°C) octadecane. Previously, ultrasonic measurements on the same octadecane-filled sandstone showed significant discrepancy from the predictions of Ciz-Shapiro theory, which was explained by the effect of stiffening of compliant pores at the grain contacts. However, the present results show good agreement with the predictions of the Ciz-Shapiro theory and suggest that the effect of stiffening of the compliant pores in presence of solid is negligible at low frequency.

1. Introduction

The evaluation of the impact of solid pore fills on the elastic characteristics of rocks is important for seismic monitoring during the development of heavy oil reservoirs (Han et al., 2008) as well as for characterizing the efficiency of CO2 storage affected by salt precipitation and rock matrix dissolution (Vanorio et al., 2011). Since a solid pore fill exhibits finite rigidity preventing the pore pressure equilibrium throughout the pore space of the rock, which is a key requirement of the Gassmann theory, the Gassmann substitution model to estimate the elastic properties of the rock with the solid pore fill may become inapplicable.

Several theoretical approaches were developed recently to model the solid-filled rocks. Ciz and Shapiro (2007) proposed exact solid-substitution equations containing some effective elastic moduli of the pore space material, which are hard to determine independently. When these unknown parameters are taken to be equal to the moduli of the pore fill, these equations reduce to Gassmann’s relation for bulk modulus and a very similar equation for shear modulus. These simplified solid-substitution equations are usually called Ciz-Shapiro equations. Saxena and Mavko (2014) used the embedded-bound method to obtain the generalized “Gassmann-like” solutions for the elastic moduli of a rock filled with fluid or solid when the stress is nonuniformly distributed in the pore space. Saxena and Mavko (2015), Gurevich and Saxena (2015), and Glubokovskikh et al. (2016) extended this approach to include effects of shear resistance of a solid-like high-viscosity fluid saturating stiff and compliant pores of a rock.

The laboratory studies of solid-filled rocks presented in literature are rather numerous (see, e.g., Batzle et al., 2006; Behura et al., 2007; Chopra et al., 2010; Han et al., 2008; Kato et al., 2012; Spencer, 2013; Sun & Han, 2017; and Rabbani et al., 2017) but are mainly limited to experiments at ultrasonic frequencies. One main implication of a number of experimental investigations (Batzle et al., 2006; Behura et al., 2007; Das & Batzle, 2008; Hinkle et al., 2008) is that the elastic parameters of heavy oil in a quasi-solid state are strongly frequency-dependent. Therefore, the data obtained at low frequencies can significantly differ from the data measured by ultrasonic methods, which makes it rather challenging to use laboratory ultrasonic results to interpret field data collected at seismic frequencies. Literature on the seismic-frequency studies of the effect of solid fill on the elastic properties of rocks is very sparse. Behura et al. (2007) studied shear moduli of Uvalde bitumen and Uvalde bitumen-saturated carbonate in the frequency range of 0.01 to 80 Hz and revealed that heavy oil in rocks can be considered as a solid at room temperature and becomes a fluid at higher temperatures. Batzle et al. (2006) showed that the shear modulus of Uvalde bitumen measured at ultrasonic and seismic frequencies can differ by more than factor of 2. Das and Batzle (2008) investigated...
the applicability of the Ciz-Shapiro equations for predicting the elastic moduli of Uvalde bitumen-saturated carbonate and found a reasonable match with experimental data. The laboratory tests conducted by Spencer (2013) on McMurray bitumen sand at low frequencies (0.2–205 Hz) showed that the frequency-dependent elastic moduli of the sand, as well as the attenuation peaks, shift in frequency consistently with the decrease of bitumen viscosity as temperature is increased. Spencer also found that the P wave and bulk moduli of the bitumen sand are significantly more susceptible to the increase of temperature than the shear modulus.

The purpose of this study is, first, to estimate the effects of solid pore fill on elastic properties of Bentheim sandstone at seismic frequency using the standard forced oscillations method, and, second, to explore the applicability of the Ciz and Shapiro (2007) and Saxena and Mavko (2014) models for predictions of the elastic moduli of Bentheim sandstone with solid pore fill.

2. Sample Description

The Bentheim sandstone studied in this research is homogeneous quartz-rich (95%) sandstone with insignificant clay content (<1%). The physical characteristics of the sample tested in our experiments are: porosity ~23.6%, length ~7.9 cm, diameter ~3.9 cm. The density of the sample in dry state is 1,974 kg/m³. The densities of the liquid- and solid-filled sample are almost identical and equal to 2,160 kg/m³. The permeability of the sample was quantified with regard to water at a flow rate of 1 ml/min as a function of the effective pressure, which is defined as the difference between the confining and pore pressures. The measured permeability is shown in Figure 1.

The homogeneity of the sample, which presents one of the key requirements of the forced-oscillation method, was verified using an X-ray micro-computed tomography system VersaXRM-500 (XRadia-Zeiss). The cross-section images scanned in X-Y plane are presented in Figure 2. Based on these X-ray images, we assume that the sandstone tested in this study is homogeneous on a macroscopic (sample-size) scale.

3. Experiment

The low-frequency laboratory apparatus used in our study is developed for measurements of the complex Young’s modulus and Poisson’s ratio of a rock at seismic frequencies and at strains of $10^{-7}$-$10^{-6}$ (Mikhaltsevitch et al., 2014).

![Figure 1. The pressure dependence of the water permeability of Bentheim sandstone.](image1)

![Figure 2. Electron microscopy images of the sandstone sample scanned at two resolutions: (a) the scale bar is 1 cm; (b) the scale bar is 250 µm.](image2)
In our experiment, we used octadecane as a pore fill for the tested sandstone sample. Octadecane CH₃(CH₂)₁₆CH₃ is a hydrocarbon with a melting point of 28°C, which gives us the opportunity to use this substance for two aggregate states of the pore fill: solid (at 23°C) and liquid (at 40°C).

The densities of octadecane at temperatures of 23 and 40°C are close and equal to 780 and 770 kg/m³, respectively. The viscosity of octadecane at 40°C is 3.1 mPa·s (Wohlfarth & Wohlfarth, 2002) and >10⁴ Pa·s at 20°C (Delgado et al., 2012). The bulk and shear moduli of octadecane are measured at a frequency of 0.5 MHz and shown in Figure 3.

The experiment was organized as follows. First, the sample was saturated for 24 hrs with liquid octadecane under vacuum at 50°C. After the saturation, the temperature was decreased to the point of solidification of octadecane (28°C). Then, the solid-filled sample was jacketed, placed inside a sleeve within the test cell, and measured at a frequency of 2 Hz under confining pressure incrementally shifting from 3.5 to 30 MPa. At the second stage, the test cell was heated for a few hours at a temperature of 40°C and the sample within the cell was resaturated with liquid octadecane at a confining pressure of 6 MPa with the use of a back pressure of 2 MPa. After that, the measurements were repeated at 40°C under the effective pressure used for the solid-filled sample, with the difference that the pore pressure was now equal to 2 MPa. At the third stage, octadecane in the pore space of the sample was removed by pumping n-decane, a strong solvent for octadecane, through the rock at a rate of 2 ml/min, and then the line for the pore fluid was opened. Afterward, the drained bulk and shear moduli were measured under the confining pressures applied in the first stage.

4. Results and Discussion

The pressure dependences of the Young’s modulus and Poisson ratio obtained at 2 Hz for liquid and solid pore fills are presented in Figures 4a and 4b, respectively. As one can see, when solid fill is replaced by liquid, the Poisson ratio decreases insignificantly, but the Young’s modulus is reduced by at least 15%. Let us also note that the accuracy of the Young’s modulus measurements was estimated in accordance with the uncertainty analysis procedure elaborated for the forced-oscillation measurements in Adam et al. (2006) and Adam et al. (2009) and is equal to 3%.

The bulk $K$ and shear $\mu$ moduli of the sample were found using relations

$$K = \frac{E}{3(1-\nu)}, \quad \mu = \frac{E}{2(1+\nu)},$$

where $\nu$ and $E$ are the Poisson ratio and Young’s modulus of the rock, respectively, and are given in Figure 5. These results show that the change of the sample state from solid-filled to fully liquid-saturated and from the latter to drained decreases the bulk modulus of the sandstone by ~33% and 13%, respectively (Figure 5a),

![Figure 3](image-url)  
**Figure 3.** The melting temperature dependences of the bulk and shear moduli of octadecane measured at ambient pressure.

![Figure 4](image-url)  
**Figure 4.** The Young’s modulus (a) and Poisson ratio (b) dependences on the effective pressure obtained for the solid-, liquid-filled, and drained Bentheim sandstone. The measurements are conducted at a frequency of 2 Hz. The pore pressure in the case of the liquid-filled sandstone was 2 MPa.
whereas the shear modulus of the sandstone with solid filling is 17% larger than in case of liquid saturation. The drainage of the pore fluid from the liquid-saturated sandstone leaves the shear modulus virtually unchanged (Figure 5b). Note that the sharp rise of the bulk moduli with pressure, observed at low confining pressures (Figure 5a), can be explained by the effect of squeezing of the compliant pores in the sample. In more detail, the effect of the compliant pores on the pressure dependence of the bulk modulus of Bentheim sandstone is discussed in Sun, Gurevich, Lebedev, Glubokovskikh, Mikhaltsevitch, et al. (2018).

To verify the accuracy of the experimentally found values of drained moduli, we use the Gassmann (1951) equation

$$K_{\text{sat}} = K_{\text{dry}} + \frac{1}{\phi} \left( \frac{K_{\text{dry}} - K_{\text{m}}}{K_{\text{m}} - K_{\text{dry}}} \right)^2 \cdot \frac{\phi K_{\text{fluid}} + (1-\phi) K_{\text{m}} - K_{\text{dry}} K_{\text{m}}}{C_16/C_17},$$

(2)

where $K_{\text{sat}}$ is the bulk modulus of the fluid-saturated rock, $K_{\text{m}}$ is the bulk modulus of the grain mineral, $K_{\text{dry}}$ is the drained bulk modulus, and $\phi$ is the porosity of the rock.

It is usually assumed that the Gassmann theory is applicable at frequencies less than $0.1f_B$, where $f_B$ is the Biot characteristic frequency

$$f_B = \frac{\eta \phi}{2\pi \rho \kappa},$$

(3)

where $\eta$ is the viscosity of pore fluid, $\rho$ is the fluid density, and $\kappa$ is the permeability of the rock. For the Bentheim sandstone filled with liquid octadecane ($k = 2D$, $\eta = 3.1 \text{mPa} \cdot \text{s}$, $\rho = 770 \text{ kg/m}^3$, $\phi = 0.236$), the Biot characteristic frequency is $f_B = 75.5$ kHz. This result confirms that Gassmann’s equation is applicable for prediction of the bulk modulus of the tested sandstone saturated with liquid octadecane at a seismic frequency. The good agreement between the Gassmann predictions and experimental data (Figure 5) confirms the accuracy of our measurements of the drained bulk and shear moduli.

The predictions of the Ciz-Shapiro theory for the shear and bulk moduli of a homogeneous solid-filled rock are based on generalization of Brown-Korringa’s and Gassmann equations and can be found as follows (Ciz & Shapiro, 2007):

$$\left( K_{\text{solid}}^{\text{CS}} \right)^{-1} = K_{\text{dry}}^{-1} - \frac{\left( K_{\text{dry}}^{-1} - K_{\text{m}}^{-1} \right)^2}{\phi \left( K_{\text{dry}}^{-1} - K_{\text{m}}^{-1} \right) + \left( K_{\text{dry}}^{-1} - K_{\text{m}}^{-1} \right)^2 / C_16/C_17},$$

(4)

$$\left( \mu_{\text{solid}}^{\text{CS}} \right)^{-1} = \mu_{\text{dry}}^{-1} - \frac{\left( \mu_{\text{dry}}^{-1} - \mu_{\text{m}}^{-1} \right)^2}{\phi \left( \mu_{\text{dry}}^{-1} - \mu_{\text{m}}^{-1} \right) + \left( \mu_{\text{dry}}^{-1} - \mu_{\text{m}}^{-1} \right)^2 / C_16/C_17},$$

(5)

where $\mu_{\text{dry}}$ and $K_{\text{dry}}$ are the shear and bulk moduli of the solid-filled rock, $\mu_{\text{dry}}$ is the drained shear modulus, $\mu_{\text{m}}$ and $K_{\text{m}}$ are the shear and bulk moduli of the pore fill, and $\mu_{\text{m}}$ is the shear modulus of the grain mineral.
We also compared our results with the solid-substitution model, which was developed by Saxena and Mavko using the embedded-bound method (Mavko & Saxena, 2013; Saxena & Mavko, 2014). The Saxena-Mavko equations for the effective bulk $K_{\text{solid}}^{SM}$ and shear $\mu_{\text{solid}}^{SM}$ moduli of rocks containing only stiff pores are given by

$$K_{\text{solid}}^{SM} = K_{bc} + \left(1 - \frac{\mu_{\text{dry}}}{\mu_{\text{if}}}\right)^2 \frac{2^2 \phi}{\frac{1}{K_{\text{dry}}} + \frac{1}{K_{\text{dry}}} - \frac{\mu_{\text{dry}}}{\mu_{\text{if}}}},$$

$$\mu_{\text{solid}}^{SM} = \mu_{bc} + \left(1 - \frac{\mu_{\text{dry}}}{\mu_{\text{if}}}\right)^2 \frac{2^2 \phi}{\frac{1}{\mu_{\text{dry}}} + \frac{1}{\mu_{\text{dry}}} - \frac{\mu_{\text{dry}}}{\mu_{\text{if}}}},$$

where

$$K_{bc} = \frac{(1 - \phi) \left(\frac{1}{K_{\text{dry}}} - \frac{1}{K_{\text{m}}\phi}\right)}{\frac{1}{K_{\text{dry}}} + \frac{1}{K_{\text{m}}\phi}\left(\frac{1}{K_{\text{dry}}} - \frac{1}{K_{\text{m}}\phi}\right)},$$

$$\mu_{bc} = \frac{(1 - \phi) \left(\frac{1}{\mu_{\text{dry}}} - \frac{1}{\mu_{\text{m}}\phi}\right)}{\frac{1}{\mu_{\text{dry}}} + \frac{1}{\mu_{\text{m}}\phi}\left(\frac{1}{\mu_{\text{dry}}} - \frac{1}{\mu_{\text{m}}\phi}\right)},$$

$$\chi = \mu \frac{9K + 8\mu}{8K + 2\mu}. \quad (10)$$

As can be seen from equations (4) and (5) and (6)-(10), the Saxena-Mavko equations can be obtained from equations (4) to (5) by replacing the drained moduli $\mu_{\text{dry}}$ and $K_{\text{dry}}$ with the effective moduli $\mu_{bc}$ and $K_{bc}$, which are dependent not only on the drained moduli and moduli of the grain mineral, but also on the moduli of the solid pore fill $\mu_{\text{if}}$ and $K_{\text{if}}$. The latter dependence disappears when the moduli of the rock fall on the Hashin-Shtrikman (1963) bounds.

For our calculations, we used the following parameters. The bulk modulus of liquid octadecane $K_{\text{fluid}}$ at 40°C is 1.42 GPa; the bulk $K_{\text{if}}$ and shear $\mu_{\text{if}}$ moduli of solid octadecane at 23°C are 3.8 and 1.13 GPa, respectively; the bulk $K_{\text{m}}$ and shear $\mu_{\text{m}}$ moduli of the grain mineral are 37 and 45 GPa, respectively. The obtained results are shown in Figures 5a and 5b. For comparison in Figure 5, we also present the Hashin-Shtrikman bounds for the bulk (Figure 5a) and shear (Figure 5b) moduli found using the following equations:

$$K_{H^{\text{HS}}} = K_1 + \frac{f_2}{K_1 - K_1}, \quad (11)$$

$$\mu_{H^{\text{HS}}} = \mu_1 + \frac{f_2}{\mu_1 - \mu_1}, \quad (12)$$

where $K_{H^{\text{HS}}}$ and $\mu_{H^{\text{HS}}}$ are upper/lower bounds for the bulk and shear moduli of the rock containing two phases presented by the grain material and pore fill; $K_i$, $\mu_i$, and $f_i$ are the bulk modulus, shear modulus, and the volume fraction of the $i$th phase ($i=1$ and 2). For the upper bound, phases 1 and 2 are assigned to the grain material and pore fill, respectively, for the lower bound; the phases are assigned in the inversed order.

As one can see in Figures 5a and 5b, the predictions of the Ciz-Shapiro model are in a better agreement with the results of the measurements than the estimates based on the Saxena-Mavko model. The match between the experimental data with the bulk modulus computed using the Saxena-Mavko model is reasonably good, whilst the discrepancy in the measured and computed shear moduli is rather significant and exceeds 10 % of the measured values.

It should also be pointed out that the observed good correlation of the Ciz-Shapiro approximation with the experimental results indicates a high homogeneity of the deviatoric strains induced within the solid-filled pore space in response to the periodic stress applied to the sample.
In Figure 6, we present the extensional attenuation obtained for solid-, liquid-filled, and drained states of the sample. The attenuation is derived from the phase delay between the applied stress and the strain in the rock (O’Connell & Budiansky, 1977). The error of the phase measurements in our experiments is ± 0.003 rad. As can be seen from Figure 6, for all states of the sample, the attenuation is within the measurement error at effective pressures exceeding 3 MPa, when the distribution of stress inside the rock becomes uniform. Taking into account the connection between modulus dispersion and attenuation due to the causality principle (Mavko et al., 2009), we can expect that the moduli change at frequencies adjacent to 2 Hz is negligible.

Finally, let us note, that the results of this study are at variance with ultrasonic data on the same rock (Sun, Gurevich, Lebedev, Glubokovskikh, Mikhaltsevitch, et al., 2018; Sun, Gurevich, Lebedev, Glubokovskikh, Squelch, et al., 2018). In particular, Sun, Gurevich, Lebedev, Glubokovskikh, Mikhaltsevitch, et al. (2018) found that the Ciz-Shapiro model underestimated both bulk and shear moduli of the rock saturated with octadecane and explained this discrepancy by the effect of stiffening of compliant pores (grain-to-grain contacts). In contrast, the low-frequency measurements for both bulk and shear moduli in the present study are consistent with the Ciz-Shapiro model, which suggests that the effect of stiffening of the compliant pores in presence of solid is negligible. Careful examination of both data sets shows that values of the bulk modulus of the solid-filled rock at seismic and ultrasonic frequencies are very close, but the Ciz-Shapiro predictions are different due to substantial difference between the seismic and ultrasonic drained moduli. We believe that the drained bulk modulus measured at low frequencies is more reliable, since it is the true drained modulus of a fluid-saturated rock, whereas the ultrasonic modulus was measured on the dry rock, and hence could be altered by the drying process and depends on relative humidity (Rasolofosaon & Zinszner, 2012; Yurikov et al., 2018). Conversely, the shear moduli of the dry and drained rock are very close, but for solid-filled rock the ultrasonic shear modulus is higher than low-frequency modulus (by about 4 GPa). This discrepancy may be due to the difference in the effect of solid-filled compliant porosity at ultrasonic and seismic frequencies, which will be explored in future work.

5. Conclusions

We present the results of the laboratory tests designed to study the behavior of the elastic moduli of the Bentheim sandstone sample, when the state of the pore fill of the rock was altered from solid to liquid, and then from liquid to drained. For both liquid and solid pore fills, we used octadecane CH₁₈(CH₂)₁₆CH₃, a substance with a melting point of 28 °C. The measurements on the solid-filled sample were conducted at a temperature of 23 °C, and on the liquid-filled and drained sample at 40 °C. After the measurements on the octadecane-filled sample, the sample was flushed and saturated with n-decane, a solvent for octadecane, the fluid line was opened and the drained moduli were measured at the confining pressure used for the solid-filled state. Thus, the measured drained moduli were not affected by the absorption of water vapor on the grain contacts, which can occur when measuring dry moduli in an atmosphere with ambient humidity. All measurements were performed at a frequency of 2 Hz. For verification of the measured drained moduli, the Gassmann substitution theory was used. The extensional attenuation in the sample measured for all states of the pore material was within the measurement error at effective pressures above 3 MPa.

The experimental data obtained for the solid-filled sample were compared with the predictions of the Ciz-Shapiro and Saxena-Mavko theoretical models developed for isotropic rocks with homogeneous pore distribution. The Saxena-Mavko theory assumes heterogeneity of the stress in the pore space caused by rock stiffening due to nonzero shear stresses in pores, while the Ciz-Shapiro theory suggests that in the case of a homogenous pore material, the stress induced in the pores is uniform and the stiffness of the pore space can be approximated by the stiffness of the pore material. The advantage of choosing the Ciz-Shapiro and Saxena-Mavko models is that they both can be directly verified experimentally.
The good agreement between the Ciz-Shapiro model and the experimental results, taken in conjunction with the attenuation data, suggests a high uniformity of the stress generated within the solid-filled pore space in response to the dynamic force applied to the rock.

The bulk modulus obtained using the Saxena-Mavko model is in a reasonably good agreement with the experimental results, whereas the measured shear modulus of the solid-filled sandstone is at least 10% lower the predictions of the Saxena-Mavko equations.

The good match between the models computed using the Ciz-Shapiro equations and the moduli obtained in our experiments on the solid-filled Bentheim sandstone demonstrates the applicability of the Ciz-Shapiro model for predictions of the elastic moduli of a solid-filled rock at seismic frequencies.

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The experimental data presented in the paper are available in the repository Zenodo (https://doi.org/10.5281/zenodo.2613217).

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