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1. Introduction

Guggenheim and Martin (1995), define the clay as a naturally occurring aluminum silicate composed dominantly of fine-grained minerals. Several other definitions and classifications based on the grain size, pore size, sedimentation, lattice, and other properties can be found in the literature. The attention here is, however, not related to these definitions instead it is about “how to calculate reservoir clay volume using Scanning Electron Microscope (SEM) data”.

The rock that predominantly contains clay minerals is by definition called Shale. Shales are mostly fine-grained clastic sedimentary rocks composed of clays and fragments of other minerals i.e., carbonates and siliciclastics. Most of the clay minerals contain variable amounts of water trapped in the mineral structure (Wiki definition). Clay volume is one of the key parameters used to correct porosity and water saturation for the effects of clay bound water in petrophysical evaluation (Crain, 2000).

A problematic aspect of evaluating hydrocarbon bearing reservoirs is to accurately model the effects of clay and shale components on reservoir properties. Clay minerals and other sedimentary materials as detrital shale components in the form of shale lamina, structural clasts and dispersed shale matrix can be deposited in the sedimentary rocks. Clay minerals are also commonly present as diagenetic clays, including pore-filling clays, pore-lining clays and pore-bridging clays (Wilson and Pittman 1977).

Accurate quantification of the clay content, distribution and clay type in the reservoir rock using core and wireline log data is rather complicated by a number of factors including: rock heterogeneity, mud filtrate invasion and fluid contamination, alteration of clay microstructure and wettability by mud invasion. On the other hand, a number of geological and petrophysical methods have been developed to run the clay volume calculation.

Traditionally, a log interpreter i.e., petrophysicist, uses wireline log data including clay indicator logs of gamma rays and spectral gamma rays in combination with other porosity and resistivity logs to estimate reservoir clay volume. Integrated analyses of these logs together with geological information are generally used for clay volume calculation. Clay volume in homogeneous reservoir rocks can be estimated by conventional methods however, a mixture of clay minerals, quartz, and calcite notably complicates the interpretation of wireline log data. Rock heterogeneity influences the resistivity logs and
thereby water saturation as well. The focus here, however, is to discuss the use of SEM data in combination with wireline logs to calculate reservoir clay volume.

2. Conventional shale volume calculation

Generally, porosity logging tools (neutron, density and sonic) display higher porosity measurements in clay-rich rocks, whereas much of the porosity is neither effective for hydrocarbon accumulation nor for hydrocarbons transmission through the rock. The shale porosity has to be removed from the total porosity to obtain effective reservoir porosity. The clay correction is therefore an essential step to carry out before estimating reservoir porosity and saturation. Figure 1 displays the gamma-ray, spectral gamma-ray, and calculated clay volume from these logs for a reservoir in the offshore Norwegian Barents Sea. Presence of siliciclastic fines and diagenetic minerals (e.g. dolomite) within carbonate breccias has resulted in a heterogeneous carbonate reservoir in the prospect area. Interactive Petrophysics (IP) software was used for wireline log interpretation and petrophysical analysis of the well.

Carbonate rocks, depending on the diagenesis and sedimentary environment, are generally uranium-rich compared to siliciclastic rocks (Luczaj and Goldstein, 2000). A large amount of uranium may precipitate during and after carbonate rock deposition. The process of uranium precipitation is more abundant in the dolomitic sediments. The easy solution to distinguish the uranium-rich from non-uranium rocks is to compare the two most well known logs of natural gamma ray spectrometry (NGS) against natural gamma ray log (GR). Natural gamma-ray (NGS) spectrometry allows estimation of the elemental concentrations of potassium (K), uranium (U), and thorium (Th), which can be used to interpret sediment composition, clay volume, and diagenesis.

Comparison of these logs in Figure 1 shows uranium-rich carbonate rocks in zones 2 and 3 compared to the more silica-rich intervals of zone 5. The higher uranium content originates from uranium-rich organic matter in the reservoir rock. To calculate the shale volume of the reservoir, the spectral gamma-ray log is used. It is also used to verify the calculated shale volume from the gamma-ray log. In Figure 2, track numbers 4, 5, and 6 show the results of the clay volumes for all reservoir sections in this well. The results are shown for potassium, thorium and corrected gamma-ray logs respectively. The shale and clean (sand) lines are drawn on the potassium, thorium, and corrected gamma-ray logs to estimate the bounding values for zero and 100% shale volume. The bounding values to the corrected gamma-ray response are assumed to be 25 API for zero shale volume and 85 API for 100% shale volume. (Crain Petrophysical Handbook). According to the results in figure 2, discrepancy of the results is considerable. The higher uncertainty in clay volume calculation impacts more effectively the reservoir porosity and saturation estimation afterward.

3. NMR log application for shale volume calculation

The NMR log T2 distribution and its characteristics are dependent on the fluid content and pore properties of the formation, and independent of mineral composition. It is by default used for pore size distribution, capillary bound water and free fluid estimation. Basically, modern NMR tools determine the fluid content in the pore space near the wellbore in the order of few inches into the formation. They measure the total rock porosity regardless of the pore fluid type (Dunn et al., 1998). The two T2 distributions, one from the fully polarized
echo train and one from the partially polarized echo train, are merged together to build a T2 distribution from 0.3 ms to over 3000 ms for estimating the total porosity. In other words, the sum of the volume fractions occupied by water, oil, and gas will be the total NMR porosity. Traditional NMR tools miss the very fast T2 decay times; however, in modern tools the T2 spectra include all types and sizes of pores and cracks (Coates et al., 1999).

Fig. 1. Well log data, reservoir zonation and lithology.

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Fig. 2. Shale volume calculation from different methods: track-4 shale volume from potassium log; track-5 shale volume from thorium log; track-6 shale volume from uranium-corrected gamma-ray; track-7 shale volume from NMR log.

The NMR T2 distribution displays a distinct peak at small relaxation times, proving the existence of clay minerals in the logged intervals. To categorize the contribution to porosity from clay, a T2 cut-off value for clay-bound water is applied to the T2 distribution over the reservoir section. Morriss et al. (1997) proposed a T2 clay-bound water cut-off of 3 ms by studying 45 siliciclastic reservoirs. In a supplementary study by Prammer et al. (1996) different T2 cut-off values were found for particular clay types at shorter decay times. Knowing the clay type and implementing an appropriate T2 cut-off value, more accurate
determination of clay-bound water volume is possible. The clay-bound water T2 cut-off and clay type/shale volume estimation are important parameters in reservoir characterization. The shale volume estimation is more complicated when the rock is composed of a mixture of clay minerals and tiny fragments of other minerals. Greater amounts of fragments of non-clay minerals increase the uncertainty of obtaining an accurate clay bound T2 cut-off value for clay-bound water (Chitale et al., 1999). Although estimation of the T2 cut-off has been discussed by many authors, there is still uncertainty in the optimum choice for the value. With reference to the work of Matteson et al. (2000), the standard relaxation time of 3.3 ms is used, which is appropriate where the clay type is not kaolinite.

The calculated shale volumes from gamma-ray, spectral gamma-ray (thorium and potassium only), and NMR logs (Figure 7, tracks 4, 5, 6 and 7) show that the calculated volumes are not identical from these methods. Depending upon the chosen gamma-ray and spectral gamma-ray values for the clean and shaly rock samples, the results for the shale volume can differ significantly with different methods. The results from the spectral gamma-ray log in tracks 4 and 5 are in good agreement within the whole reservoir section except in zone 5. The uranium-corrected gamma-ray log, however, shows shale-free reservoir except in zone 5 and parts of zones 2 and 4. To estimate shale volume from the NMR log, representative clayey intervals (100% clay volume) and the amount of clay-bound water in those intervals estimated from the NMR log are classified as references for particular clay types in the well. The amount of clay-bound water estimated from the NMR log response at any other point in the well is compared to that in the selected reference clay in order to estimate the shale volume at that point. There may be errors in this estimate if the selected reference clay was inappropriate. The estimated shale volume from the NMR log is significantly less than that from the gamma-ray log indicators (track 6, Figure 2), presumably due to the errors in conversion of clay-bound water to the shale volume.

A clear response to the presence of clay in the T2 distribution is seen right at the top of Figure 2 in zone 1, the top seal of marine claystones. However, assuming that the top shale seal is totally composed of clay minerals, the NMR estimated shale volume is also not accurate within this interval. Selection of too low a T2 cut-off for clay-bound water or too rapid a decay of the clay-bound water could explain the errors. In addition to the clays, viscous hydrocarbons (tar) relax at much shorter relaxation times than the clay-bound water T2 cut-offs. The NMR log interpretation can be more complicated where both clay and tar-mats are present in the reservoir rock. Retrieved fluid samples from the wireline formation tester and recovered hydrocarbons from core material obtained from the studied well have not shown any tar in this reservoir. Overall, zones 4 and 5 of the reservoir show uncorrelated results compared to the rest of the well. Assuming a formation rich in potassium minerals coupled with high gamma-ray response, the rock type could be either shale, a mixture of shale and other potassium-bearing rocks, or other rock types rich in radioactive minerals.

To identify the clay minerals and potassium sources of zones 1 and 5, the thorium-potassium reference cross-plot of Schlumberger (1985) and corresponding cross-plot of the well data are shown in Figure 3. According to these plots, potassium sources in zone 5 are glauconitic or feldspathic sandstone together with illite and mica, whereas the clay minerals of zone 1 are mixed-layer clay and/or illite. The result gives valuable information about the source of the clays but it could not provide a volumetric response for the shale volume of
the rock. Further calibration of the logs and petrographical analysis of the rock samples are necessary to quantify the reservoir shale volume.

Fig. 3. (a) Schlumberger reference crossplot of thorium/potassium; and (b) corresponding crossplots for zones 1 and 5 to identify the clay types.

4. Scanning electron microscope (SEM) analyses
To identify the rock mineralogy and quantify the mineral volume fractions, SEM analysis was carried out on the selected reservoir samples. Because of inconsistencies in the amount
of calculated shale volume in zone 5 (Figure 7), most of the samples are chosen from this zone. Backscattered electron imaging of polished thin sections is used to map mineral distributions in a grey scale with intensity related to the average atomic number of the minerals (Reed, 2005). Mineral identification was supplemented by energy dispersive spectroscopy analysis, and X-ray mapping of element distributions was done to further identify and quantify the mineral distributions. A backscattered electron image of a sample of sandy carbonate rock in zone 5 is shown in Figure 4a. To estimate the proportion of one particular mineral, the image is processed and one component, e.g., potassium feldspar, is characterized (Figure 4b). The energy dispersive spectrum of potassium feldspars (black in Figure 4b) is shown in Figure 4c. The proportion of elements (K, Si, Al, and O) in this figure verify that the selected grains are potassium feldspar. Energy dispersive spectroscopy is used to recognize every selected mineral in a thin section sample by its elemental composition, but X-ray mapping of the section provides a map of each element.

Fig. 4. Details of scanning electron microscope imaging of one sample from zone 5. (a) Backscattered electron image. (b) Processed image for the characterization of potassium feldspar, showing up as black. (c) Energy dispersive spectrum of a selected clay mineral to identify it.
The X-ray elemental mapping of the selected thin section in Figure 4 also confirms the abundance of potassium feldspar in this section, but not clays. Figure 5 shows the elemental map of Na, O, K, C, S, Si, Mg, Ca, Ti, Al, and Fe for the selected sample in Figure 4. The volume fraction of potassium feldspar from this analysis is calculated as 16–23%. The map of feldspar content in other thin sections in zone 5 shows that the potassium volume increases toward the bottom of the formation. A lesser amount of illite/mica is estimated in comparison with the estimated volume from the conventional method. The clay types and volume estimations from SEM analysis are also consistent with the Schlumberger crossplot. The results show that the clay minerals of the sample from zone 5 are illite and mica with a volume fraction of 15–21% (Figure 6).

A few thin sections cannot be representative of the whole reservoir section because they have been selected from a heterogeneous interval. However, SEM analysis together with the NMR and spectral gamma-ray logs facilitates shale volume calculation. SEM data provide shale volume and clay types at the selected depths which can be correlated with the spectral gamma-ray log to extract the shale and potassium feldspar volume at those depths. Comparison of these results with the NMR-estimated shale volumes shows that the NMR log underestimates the shale volume. In contrast, the spectral gamma-ray log overestimates the shale volume.

The potassium readings on the spectral gamma-ray log in zone 5 and part of zone 2 come from a mixture of clay minerals and potassium feldspar. As discussed by Ellis and Singer (2008) with reference to the gamma-ray response in shaly rocks, the correlation between clay minerals and thorium is largest, because of the potassium association with other components of the shale, such as feldspars. They also established an empirical equation for...
the total gamma-ray response based on the three radioactive components of the spectral gamma-ray signal, however potassium-rich shaly sand could be inaccurately interpreted as shale by this equation. Therefore, the contribution of potassium feldspar has to be removed from the spectral gamma-ray log to estimate an accurate shale volume in the selected samples. Using Ellis and Singer’s (2008) approximation and estimated potassium values from the SEM samples, the corrected gamma-ray log values were calibrated for selected samples and subsequent shale volumes. The new potassium-corrected log was then used with the thorium log to estimate a revised shale volume in zone 5. Figure 7 shows the calculated shale volumes from all the above-mentioned methods. The calculated clay volumes using potassium, thorium, and uranium-corrected gamma-ray (CGR) and the integration of SEM, CGR and NMR logs are shown in Figure 7 from tracks 4 to 7, respectively. In track 7, the final corrected shale volume has been plotted for the whole interval.

Fig. 6. Backscattered electron image of the sample section in zone 5 showing illite, mica, and feldspar.
Fig. 7. Shale volume calculation by using HCGR, thorium-potassium ratio, thorium, and integration of SEM, thorium, and potassium.

5. Summary

Although the gamma-ray log has traditionally been used for the analysis of shaly formations, the shale volume estimation from this measurement is, to a greater or lesser extent, inaccurate. The GR log responds to the natural-gamma radiation from the formation.
The spectral gamma-ray log works on the same principle, except that the gamma rays are assigned to three different energy bins, showing the concentrations of K, U, and Th in the formation. Generally, carbonate rocks show higher uranium measurements on the spectral gamma-ray log because of the presence of organic matter. Uranium is, therefore, removed prior to the use in clay volume calculation. The sources of potassium also have to be distinguished because potassium is present in shaly and non-shaly rocks. In this study, the NMR log is used as an easy tool to check the reliability of the shale volume calculation from gamma-ray and spectral gamma-ray logs. Inconsistency between the shale volumes estimated from these methods is significant in the lowermost reservoir section. To solve the problem, SEM analysis was done to identify the mineralogy and mineral volume fractions. Backscattered electron images and X-ray mapping of selected samples show a noticeable contribution of potassium feldspar in zone 5. The potassium feldspar content of the rock influences both the gamma-ray and spectral gamma-ray logs and thus the shale volume calculation. The spectral gamma-ray log of potassium was therefore corrected on the basis of SEM information. Subsequently, the SEM information was used as a reference point for the calibration of the spectral gamma-ray log to estimate the shale volume. The uniformity of the spectral gamma-ray logs within zone 5 used for calibration over the entire zone 5. The NMR log was also used to verify the calculation to some extent. The volume of potassium feldspar was then removed from the total potassium reading in the spectral gamma-ray log to obtain the actual shale volume of the rock. The results show a considerable reduction in the estimated shale volume of the reservoir rock. Overestimation of shale volume has a direct impact on the reservoir evaluation by causing underestimation of the effective porosity and, consequently, hydrocarbon volume.

6. Acknowledgements
Authors thank Norwegian University of Science and Technology and Statoil for support and access to petrophysical data, and Stephen Lippard for his advice on the manuscript.

7. Nomenclature

<table>
<thead>
<tr>
<th>Name</th>
<th>Description (unit)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BS</td>
<td>Bit size (inch)</td>
</tr>
<tr>
<td>CBW</td>
<td>Clay-bound water (decimal fraction)</td>
</tr>
<tr>
<td>Depth</td>
<td>Depth from rotary table (m)</td>
</tr>
<tr>
<td>Dt</td>
<td>Sonic transit time (μs/ft-1)</td>
</tr>
<tr>
<td>HCAL</td>
<td>Caliper log (inch)</td>
</tr>
<tr>
<td>HCGR</td>
<td>Compensated gamma-ray (API)</td>
</tr>
<tr>
<td>HCGR_SHALE</td>
<td>Shale volume from HCGR log (decimal fraction)</td>
</tr>
<tr>
<td>HFK</td>
<td>Spectral gamma-ray log potassium (%)</td>
</tr>
<tr>
<td>HTHO</td>
<td>Spectral gamma-ray log thorium (ppm)</td>
</tr>
<tr>
<td>HTHO_SHALE</td>
<td>Shale volume from thorium log (decimal fraction)</td>
</tr>
<tr>
<td>HURA</td>
<td>Spectral gamma-ray log uranium (ppm)</td>
</tr>
<tr>
<td>NMR_SHALE</td>
<td>Shale volume from NMR log (decimal fraction)</td>
</tr>
<tr>
<td>nmrT2cutoff</td>
<td>T2 cut-off (ms)</td>
</tr>
<tr>
<td>NPHIC</td>
<td>Neutron log (corrected) (decimal fraction)</td>
</tr>
</tbody>
</table>

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PHIE Effective porosity (decimal fraction)
PHIT Total porosity (decimal fraction)
RHO Density log (corrected) (g cm-3)
Vanhydrite Anhydrite volume (decimal fraction)
VDol Dolomite volume (decimal fraction)
VLime Limestone volume (decimal fraction)
VSand Sandstone volume (decimal fraction)
VWL Wey clay volume (decimal fraction)

8. References


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Today, an individual would be hard-pressed to find any science field that does not employ methods and instruments based on the use of fine focused electron and ion beams. Well instrumented and supplemented with advanced methods and techniques, SEMs provide possibilities not only of surface imaging but quantitative measurement of object topologies, local electrophysical characteristics of semiconductor structures and performing elemental analysis. Moreover, a fine focused e-beam is widely used for the creation of micro and nanostructures. The book’s approach covers both theoretical and practical issues related to scanning electron microscopy. The book has 41 chapters, divided into six sections: Instrumentation, Methodology, Biology, Medicine, Material Science, Nanostructured Materials for Electronic Industry, Thin Films, Membranes, Ceramic, Geoscience, and Mineralogy. Each chapter, written by different authors, is a complete work which presupposes that readers have some background knowledge on the subject.

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