

Interactive comment on “Porosity and permeability determinations of organic rich Posidonia shales based on 3D analyses by FIB-SEM microscopy” by Georg H. Grathoff et al.

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We thank Lionel Esteban for his very detailed and constructive criticism. He states that he is unsure of the “usability of the scientific data”. We know that there are issues concerning the representative volume using FIB-SEM, that the data collection was not gathered under insitu conditions and that the sample have undergone some alteration between being in place and analyzed in the FIB-SEM. However we argue, below in detail, that these alterations are minor and that the calculations compare well with other published data and can be used as one piece of puzzle to understand the big picture of porosity and permeability developments in the Posidonia shales. In general we have significantly added content to the introduction as well as the discussion to address the

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points that the reviewer made. I. Regarding the unpreserved shale issue The reviewer argues that working with unpreserved shales leads to porosities that are too high because clay minerals and organic matter are strongly influenced by degassing, unloading and dehydration. We agree that these problems exist and are clarified in the edited manuscript, however the problem is not as significant to our samples as the reviewer thinks. The problem of unloading could be solved digitally by calculating the deformation of the microstructures after simulating in situ conditions, which we currently can't do. As far as we know such a module is being implemented in GeoDict in the future. For our samples, WIC and HAD, unloading is a problem we can ignore because the samples come from shallow depths of only down to 50 m below ground level (Littke, 1988). Regarding the drying of the material we assume that dehydration of the shale only poses a minor problem. Mineralogical investigations by Kaufhold et al. (2015) on the samples we used have shown that all shale samples are predominantly composed of carbonates (62 % WIC, 42 % HAD). The illite/smectite mixed layer mineral content ranges from 12 % (WIC) to 21 % (HAD). IS is the only swelling clay mineral. Based on Srodon (1984) the smectite content is below 20 % in these mixed layer minerals. The shrinkage caused by dehydration of the swelling clay minerals has a rather insignificant effect on the determined pore space morphology. Slit like pores are often a result of dehydration. We have only observed them in a very small amount. We added that point to the paper. II. Regarding the total porosity determination It was criticized that the true total porosity derived from density logs and grain density calculations was missing. To address that we added a Table with porosity and permeability data of other studies that worked with the samples of the same formations (Ghanizadeh et al. (2014), Klaver et al. (2012, 2016), Mathia et al. (2016), Mohnhoff et al. (2015), Kaufhold et al. (2015), Gasparik et al. (2013), Rexer et al. (2014)). However, the work of Kaufhold et al. (2015), working on the same sample set compared several direct and indirect methods (He/dryflow, N₂-absorption, Hg-porosimetry, CO₂-adsorption, FIB-SEM, μ -CT) to determine the porosity. Therefore we see no need to add new measurements. We are aware that we can only determine a small fraction of the total porosity with our methods.

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III. Regarding the permeability calculations The usability of permeability calculations was criticized. In the newly submitted manuscript we added a section to the discussion, where we discuss modelled and measured permeability coefficients (Ghanizadeh et al. (2014) and Mohnhoff et al. (2015)). The results of the GeoDict calculations depend strongly on the assumed permeability of the organic matter. As shown in our work and by others (e.g. Curtis et al. (2012), Rexer et al. (2014), Klaver et al. (2016), Mathia (2016)) the organic matter porosity depends strongly on the maturity of the shale and on the type of the organic matter. Therefore, organic matter permeability should differ with increasing maturity. We did not use different permeability coefficients for the two samples in order to be able to compare the differences in the calculated permeability with changes in the pore space. We are aware that the assumed $K = 1e-21 \text{ m}^2$ is very likely different from the true K-value. A next step would be to perform ultra-high resolution ($\leq 5 \text{ nm}$ pixel size) FIB-SEM on OM particles in order to determine 3D pore network structures in the smallest a transport relevant regime, which we mention as a next step in our paper. This data can then could be used to calculate permeability coefficients for the OM in order to improve the initial model presented in this study. The results in this work represent a first step in the modelling of permeability coefficients for shales from microscopic data. K-values shown reflect the current state of our research and shall not be taken as final true values. We clarify this in the new version of the manuscript. Regarding intra-/interparticle pores The terminology of interparticle versus intra-particle pores were corrected. Further, we corrected the $v_{\text{water-x}}$ for HAD from $5,16e-7$ to the correct value of $5,16e-9 \text{ m}^2$. Additionally, a permeability of $1e-21 \text{ m}^2$ was used for the OM, not $1e-22 \text{ m}^2$ as described before.

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