Maximizing the core value – joint investigations with special emphasis on complex electrical conductivity give new insights into Fontainebleau Sandstone

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ABSTRACT
The Fontainebleau Sandstone is supposed to be well known amongst the core analysts' community. It has the reputation of being a reference sandstone, i.e. it is widely used to study and to assess petrophysical relationships and pore scale (mostly flow) related processes, due to its straightforward mineral composition (more than 99% quartz) and its ideal and well connected pore system. The results of these analyses are often used to understand and interpret data from other locations that feature clastic sedimentary reservoirs. Nevertheless, the assumption that the Fontainebleau Sandstone is "easy" to understand is not true, if the entire Fontainebleau Formation, and not only the high porosity zones, are taken into account. Within our joint research project, we are investigating Fontainebleau samples from almost every stratigraphic section of the Formation, covering a wide range of petrophysical data (i.e. porosity ranging from 2 % up to 24 %, and permeability covering five orders of magnitude). Besides classic core analysis, including 2D and 3D imaging, special emphasis has been laid upon the determination of the frequency dependent complex electrical conductivity by using the spectral induced polarization (SIP) method. This method has proven to be very sensitive towards the specific surface area and surface topology, as well as towards the type of pore system. As one result, it has been possible to characterize the different Fontainebleau units in detail, and to assess the highly increasing degree of complexity qualitatively and quantitatively, as this sandstone changes its type of pore system from single porosity towards dual porosity. Amongst the different petrophysical methods, only SIP was able to detect this transition reliably.

INTRODUCTION
Spectral Induced Polarization (SIP) measurements (i.e. "low frequency range" impedance spectroscopy) are used in many different ways to characterize natural rocks and soils. Main

foci of interest are the enhanced characterization of the causes of polarization effects in sedimentary rocks. The interactions between the matrix-fluid-system and within the electrical double layer as well as the correlation with petrophysical parameters, such as specific surface area, permeability, and pore radii distribution as derived from mercury intrusion capillary pressure data are considered. A variety of polarization models, either grain based or pore based, have been developed over the past years to describe the polarization effects of sedimentary rocks [e.g.: 1, 2, 3, 4, 5, 6]. The electric resistivity, as well as electric conductivity are both described as complex quantity values. As explained by [7], complex conductivity (σ^*) of a rock sample is determined by measuring the magnitude of conductivity ($|\sigma|$) as well as the phase shift (φ), relative to a reference resistor. The real (σ') and imaginary (σ'') part of σ^*, which represent the ohmic conduction and polarization charge transport mechanisms, respectively, are directly determined from impedance measurements. The phase shift thereby is defined as:

$$\varphi = \arctan(\sigma''/\sigma') \approx \sigma''/\sigma' \text{ (for } \varphi < 100 \text{ mrad)}, \qquad (1)$$

which is by convention defined as a positive value in conductivity space. Generally, complex conductivity models are based on two contributing conductivity terms, as denoted amongst others by [8]:

$$\sigma^* = \sigma_{el} + \sigma^*_{surf}. \qquad (2)$$

The first term (σ_{el}) represents the conductivity of the electrolyte filled interconnected pore space, whereas the second term (σ^*_{surf}) reflects conduction and polarization within the Electrical Double Layer (EDL) of the interconnected pore (matrix) surface, respectively. The SIP method itself has developed from classic frequency domain IP, and takes both, electric resistivity as well as phase shift into account. This method has become a potentially good tool for the enhanced characterization of rocks as well as of soils, due to the dependence of the measurements on the internal surface area (S_{por}) of the investigated materials [9]. Additionally, strong efforts have been made to predict permeability by using SIP method [10; 11]. Hence it seems promising to combine SIP measurements with other petrophysical methods such as mercury injection porosimetry (MIP), steady state permeametry, nitrogen adsorption and buoyancy measurements, as well as state of the art imaging techniques, such as environmental scanning electron microscopy (ESEM) and X-ray micro computed tomography (µ-CT).

SAMPLES & METHODOLOGY

The Fontainebleau (FO) sandstone, is often used as a reference sandstone for systematic laboratory studies, due to its high mineralogical “purity” (> 99.6 vol.-% SiO_2). In fact, the mineralogical main component is quartz is a young tertiary sediment, for which several, controversial depository models exist [12; 13]. Despite that, the Fontainebleau sandstone features a broad variety of porous systems and hence of petrophysical properties [14]. For this study, we showcase first results of the denser part of the formation. Accordingly, the pore networks consist of pore bodies that are primarily connected via intergranular planes in between the idiomorphic quartz crystals (figure 1).

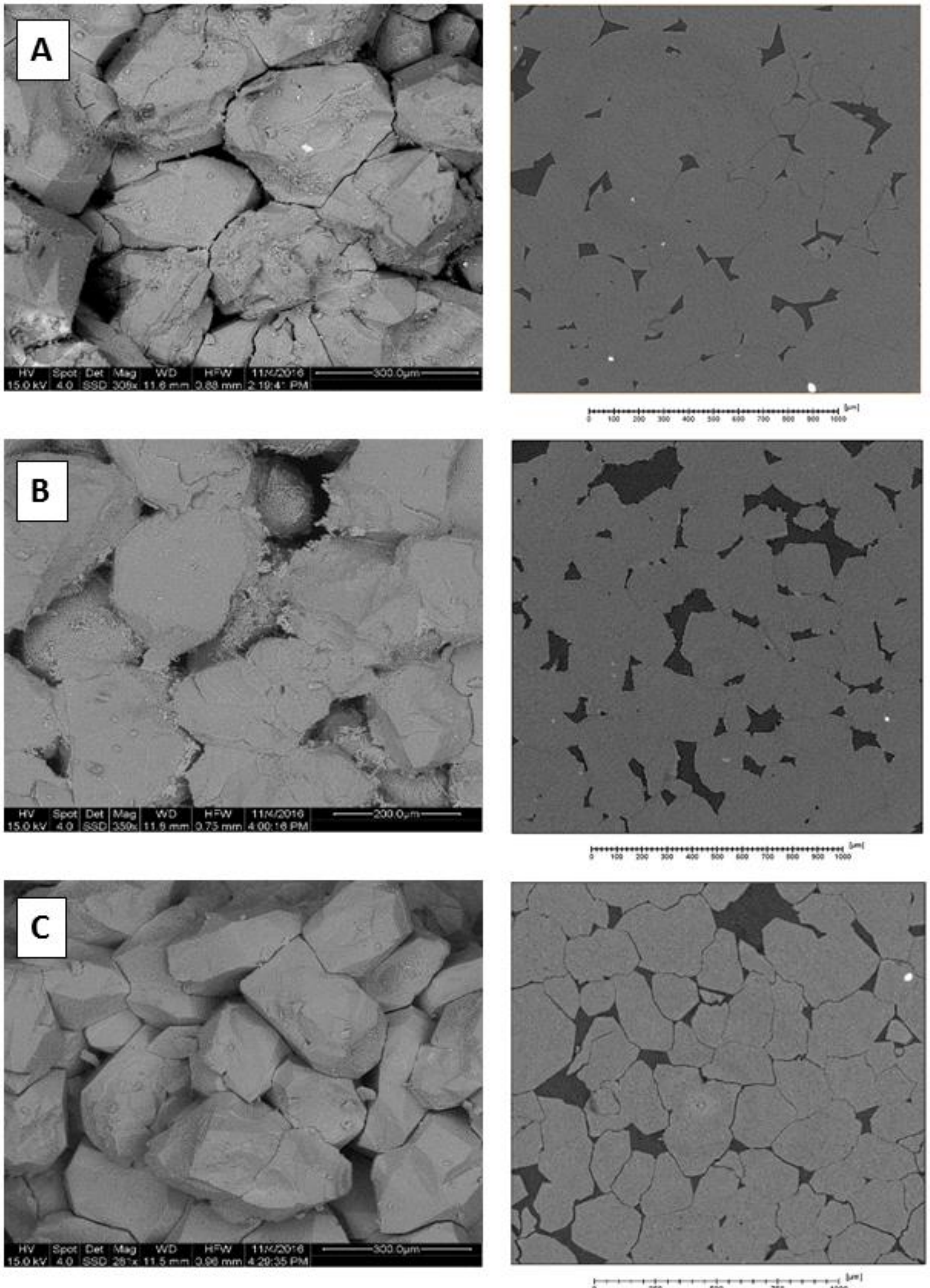

Figure 1: SEM (left hand side) and µ-CT (right hand side) images of representative samples from three different Fontainebleau stratigraphical blocks: A = block 36 (tight strata), B = block 64 (porous strata), C = block 93 (intermediate strata).

The specific surface area was determined by gas adsorption using the Brunauer-Emmet-Teller (BET) method [15]. Measurements were performed with a Quantachrome Autosorb iQ device with approximately 50 g of sample mass. Surface areas have been derived by 7-point BET adsorption measurements for each sample. Additionally, krypton has been used instead of nitrogen, due to the better adsorbent characteristics for the significant lower surface areas of these "clean" rock samples. Measurements were carried out at 77°K in a relative pressure (i.e. measured against ambient atmospheric pressure) range of 0.05 – 0.3. Mercury intrusion porosimetry (MIP) measurements were conducted by using an AutoPore III from Micromeritics within a pressure range from 0.003 MPa to 400 MPa. The calculated differential intrusion versus pore diameter curve has been used to determine the dominant pore throat diameter (D_{dom}). An Environmental Electron Scanning Microscope (ESEM) of the type FEI Sirion D1625 (low vacuum, 0.6 mbar) was used to investigate air-dried sub-samples of the three rock strata. The µ-CT imaging has been performed with a high resolution X-ray CT system (nanotom 180 S, GE Sensing & Inspection Technologies). Effective porosity of these sandstones has been determined by buoyancy method. As saturation fluid, degassed and desalinated tap water was used. The fluid density was presumed as constant (1 g/cm^3). Gas permeability has been determined by a custom built steady-state permeameter under ambient conditions, featuring a special Fancher-type core sleeve for lower overburden pressures (12 bar = 174 psi) and hence less mechanical influence upon the samples. These investigations were conducted following protocols provided by [16]. SIP measurements have been performed with a SIP-ZEL device [17] by using a custom built 4-electrode core holder [18] and within a frequency range from 1 mHz to 20 kHz. Demineralized water has been used for mixing the NaCl-solution with a varying fluid conductivity from 5 mS/m, 10 mS/m, 25 mS/m, 50 mS/m to 100 mS/m at a temperature of 20 °C for sample saturation.

RESULTS

The results from permeability, porosity and surface area measurements are showcased in figure 2. The permeability of the FO sandstone (fig. 2, A) used in this study covers four orders of magnitude (from 0.1 to 300 mD). The results are in good accordance with the data from [14] and follow the known trend. Reference data, provided by Cydarex, over-estimate the permeability for lower porosity samples slightly, due to the decreased accuracy of the used Tinyperm device (personal communication with Roland Lenormand). The different FO strata can be clearly distinguished from each other (block #36 lowest porosity and permeability values, which successively increase from block #93 to block #64) and are coherent with the qualitative results from sample imaging (fig. 1). Figure 2 (B) showcases the correlation between the Klinkenberg corrected gas permeability (k_{kl}) and specific surface area related to the pore volume (S_{por}). Again, the different block strata can be clearly differentiated from each other. Samples from block #36 and block #93 follow a steep trend line, showing a strong dependence of the hydraulic conductivity towards the surface area. Since these samples solely consist of idiomorphic quartz, it can be assumed that the surface area for these strata is dominantly influenced by the accessible pore network and not by the matrix material at all.

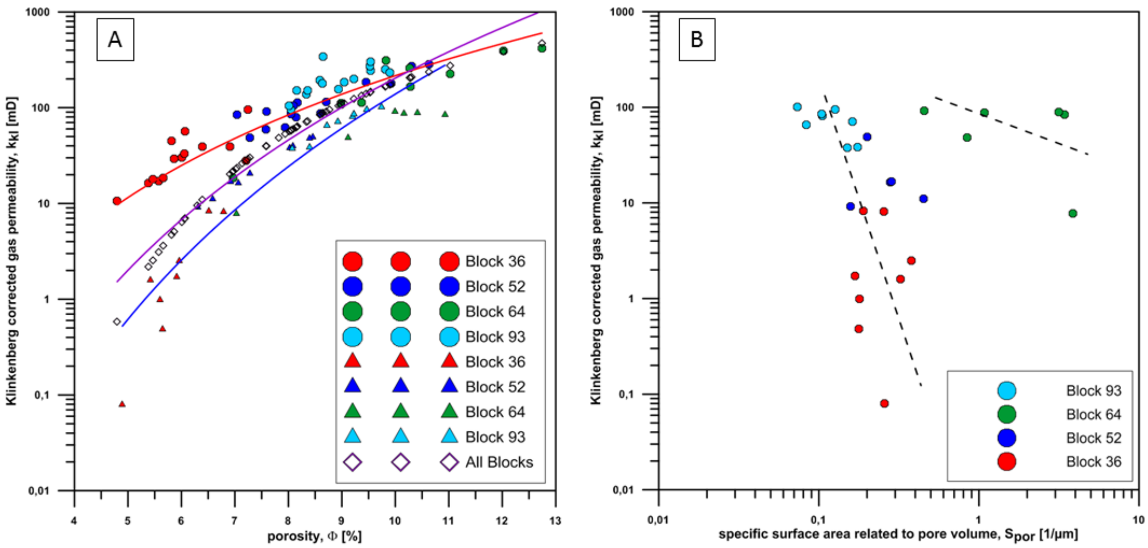

Figure 2: Porosity – permeability (A) and surface area – permeability relationship (B) for the investigated stratigraphical Fontainebleau block units (circles: data from Cydarex, $R^2 = 0.75$; triangles: data from LIAG, $R^2 = 0.88$; diamonds: data from [14] $R^2 = 0.97$).

The samples originating from block #64 follow a significantly different trend than the other strata, showing a less pronounced dependence of the permeability upon the surface area. Here, this effect can be explained by the overgrowth of the idiomorphic crystal surfaces by secondarily precipitated quartz, as shown within figure 1 (B), left hand side. Accordingly, here the influence of the matrix material (or better of the surface topology) dominates over the overall influence of the pore network structure.

Interestingly, SIP measurements on samples of these three different strata show an even higher sensitivity towards the differences in the pore networks and pore surfaces. In figure 3, representative SIP phase shift (left hand side) and resistivity spectra (right hand side) for block #36, #64 and #93 are presented. All investigated samples from block strata #36 and #93 show for low fluid conductivities a pronounced phase shift maximum at low frequencies (approx. at 0.01 Hz for unit #36 and approx. at 0.1 Hz for unit #93). This phase peak vanishes as theoretically expected, for stepwise increasing fluid conductivity according to [7]. Hence, the different relaxation times (i.e. inverse of frequency) can possibly be linked towards the accessible pore pathways, i.e. the lesser connected the pores, i.e. the longer the plane like pores that connect the few pore bodies, the longer the corresponding relaxation time (i.e. the smaller the peak frequency). Contrary to that, the samples from block #64, which feature the distinct secondarily precipitated quartz overgrowth throughout the entire sample set, show a completely different phase shift behavior. For these samples, a double phase peak behavior in the low (peak one around 0.02 Hz) and middle (peak two around 0.35 Hz) frequency range is observed (fig. 3, middle). Furthermore, if the fluid conductivity increases, the middle range phase peak vanishes, whereas the low frequency phase peak can still be assumed, but the peak shifts towards higher relaxation times in the sub-millihertz range.

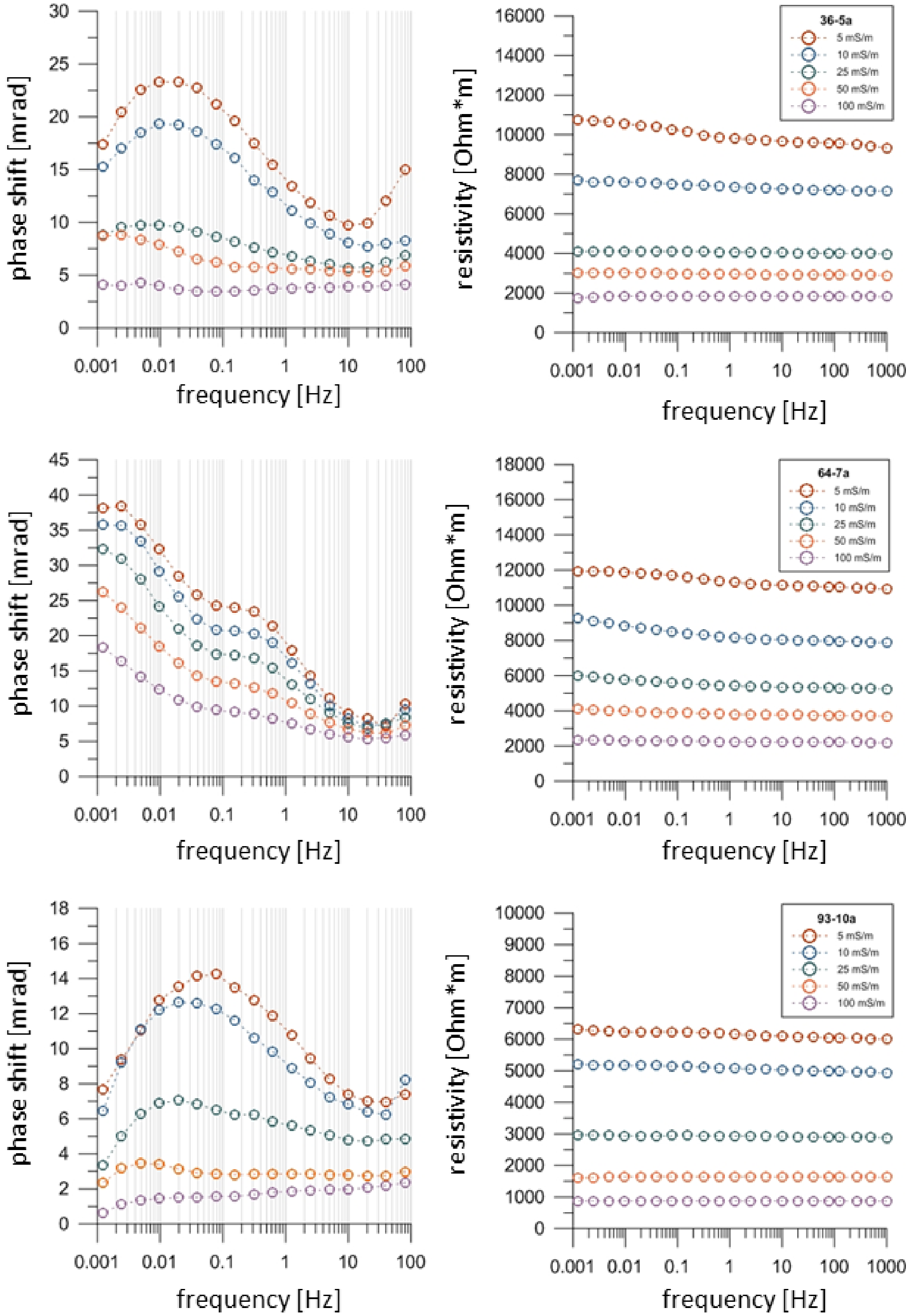

Figure 3: Representative SIP phase spectra (left hand side) and resistivity data (right hand side) for three block units: block #36 (top, sample FO-36-5a), block #64 (middle, sample FO-64-7a), block #93 (bottom, sample FO-93-10a).

Consequently, it seems reasonable to assume that the maximum phase shift peak may be linked with a characteristic (pore or grain) length within the samples (compare [19]). Following this idea, the maximum phase shift has been correlated to the dominant pore throat diameter, which has been measured by MIP. This correlation is presented in figure 4, which also has been observed for other sandstones as reported by [20]. Three possible outliers (marked by dashed circles) for the entire sample set can be identified within the given graph. These measurements either do not indicate a distinct phase shift (i.e. a "true" phase maximum within the measured frequency range), or are influenced by low permeability (< 5 mD), i.e. by a low amount of hydraulically and electrically connected pore space, and hence by an overall low polarization capability. Without these samples, a correlation ($R^2 = 0.76$) between D_{dom} and the related block strata can be detected. By trend, samples from block #93 seem to feature the largest pore throat diameter and samples from block #64 and #36 the lowest. Especially for block #64 this is a surprising result, since these samples are the most porous and permeable of this study.

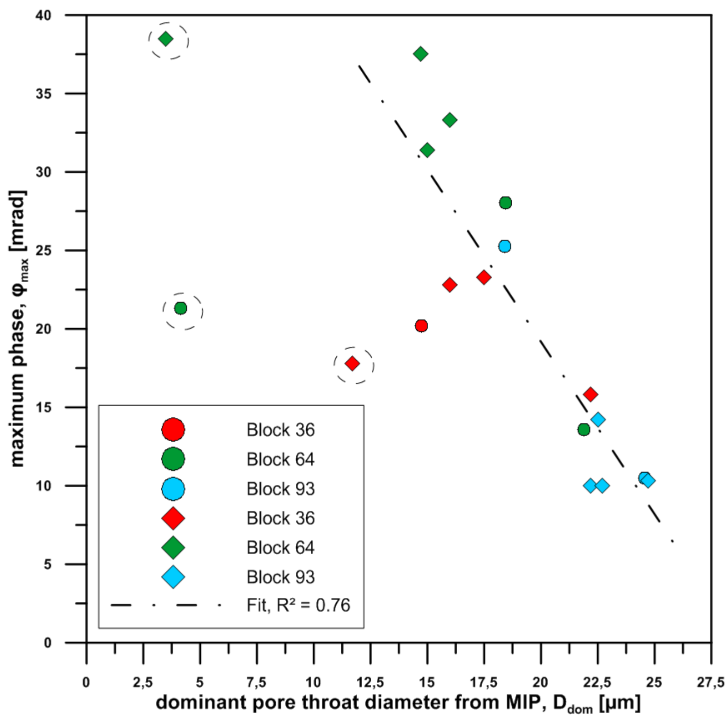

Figure 4: Correlation of the maximum phase shift (measured with SIP) with the dominant pore throat diameter (measured by MIP) for the three investigated Fontainebleau block strata (circles indicate LIAG, diamonds indicate BAM data; $R^2 = 0.76$). Dashed circles mark outliers as mentioned in the text.

SUMMARY & CONCLUSIONS

Within this study we have shown, that spectral induced polarization is a reliable method for the enhanced characterization of the Fontainebleau sandstone in general, and for its related stratigraphical units in particular. Due to its high sensitivity towards pore network and pore surface changes, different stratigraphical units can be clearly differentiated and probably even classified. We have observed a good correlation between the maximum of the SIP phase shift and the dominant pore throat radius for this rock type, as it has been reported for others sandstones before [20].

OUTLOOK

As part of an ongoing project, research upon the Fontainebleau sandstone is not finished yet. Consequently, all stratigraphical units will be investigated by SIP measurements within the near future. In addition, more multi-salinity conductivity, fluid ion-valence variations, zeta-potential, as well as formation factor measurements will be conducted to gain more insights for the correlation with SIP data. Furthermore, the impact of silica leaching upon the SIP relaxation behavior and upon the pore structure related petrophysical properties will be investigated.

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