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## Porosity effects of electrostatic forces in chalk powder

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### Introduction

We present a novel method aiming at directly observing electrostatic double layer related forces at the grain scale by using NMR spectrometry to accurately measure changes in porosity of sediment columns saturated with different ionic aqueous solutions. Results from experiments executed on calcite, quartz and kaolinite are compared to and corroborated by Zeta Potential data found in the literature, validating the method. Results from calcite sedimentation experiments are then correlated to results from rock mechanical testing done in outcrop chalk specimens by different authors. We find a link between the disjoining pressure induced by the Electrical Double Layer (EDL) related forces and water weakening observed in outcrop chalk.

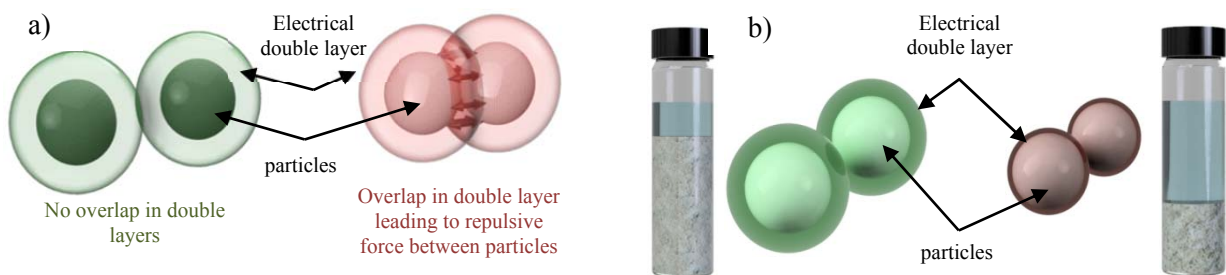
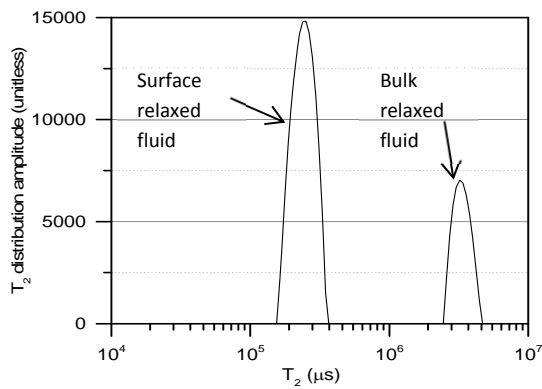


Figure 1: (a) Repulsion between solid particles caused by the overlap of double layers. (b) Effect of disjoining pressure on porosity of powders

### Methodology

**Samples.** Crushed Stevns chalk powder was saturated with brines containing ions found in seawater ( $\text{Na}^+$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Cl}^-$  and  $\text{SO}_4^{2-}$ ) at varying ionic strengths or, as a non-polar reference, with ethylene glycol. Solids were slowly added to the liquid phase and the samples agitated to wet all the surfaces and avoid the entrapment of air bubbles within the powder. Each sample was then left to rest for a day. For each of the following days, porosity of the sediment column was measured by NMR spectrometry. We found no significant difference between two subsequent porosity measurements after four days. This porosity defined the natural settlement porosity. Following measurements done under natural settlement, each sample was placed in a centrifuge to simulate overburden stress; and the porosity was measured immediately after.

**Porosity measurement.** In a  $T_2$  relaxation distribution derived from a NMR acquisition, the area below the curve represents the volume of fluid relaxed at different relaxation rates. Relaxation rate of hydrogen nuclei in molecules adsorbed to a solid surface is higher than the rate of nuclei in molecules of the bulk solution. This means that we can calculate the fraction of the total sample volume relaxed within the sediment column, which is equal to the pore volume.



The grain volume is determined by dividing the weight of the powder by its density. Porosity is then given by:

$$\phi = \frac{V_{pore}}{V_{pore} + V_{solid}} \quad (1)$$

Figure 2 - Surface and bulk relaxed fluids in NMR  $T_2$  distribution for powder column covered by and saturated with aqueous  $\text{CaCl}_2$  solution.

## Results

Porosity of the brine saturated calcite powder samples is relatively high for the brines containing  $\text{Ca}^{2+}$ , and relatively low for brines containing  $\text{SO}_4^{2-}$ ; the effect increases with ionic strength. For the samples representing high overburden, the pattern is roughly shifted to lower porosity.

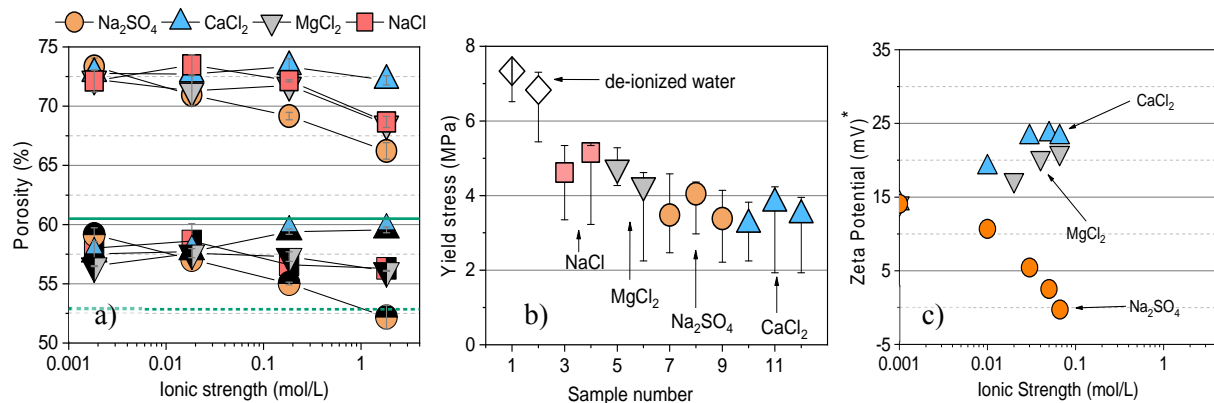


Figure 2: (a) Critical porosity of calcite powder column at initial state and final state. Green lines show results for ethylene glycol samples (b): Peak strength of Stevns samples saturated with aqueous solutions of  $\text{CaCl}_2$ ,  $\text{MgCl}_2$ ,  $\text{Na}_2\text{SO}_4$ , or  $\text{NaCl}$ , as well as de-ionized water. Samples were loaded at near-Oedometer conditions, with  $\sigma_r \approx 0.3 \sigma_a$ . Adapted from Katika et al. (2015). (c) Zeta potential measurements from Song et al (2017).

## Conclusions

We developed a method to assess electrostatic forces acting at grain scale. Correlation with yield strength measurements performed on outcrop chalk indicates that electrostatic forces might be an important mechanism in water weakening of chalk. Results obtained with this technique correlate with Zeta potential measurements of Song et al. (2017). Weakening observed by Katika et al., (2015) on samples saturated with the  $\text{Na}_2\text{SO}_4$  brine could not be explained in terms of EDL-related forces.

## Acknowledgements

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## References

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