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Original and pyrometamorphical altered Bentheimer sandstone; petrophysical properties, surface and dielectric behavior

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ABSTRACT

Bentheimer sandstone is a quartz-rich permeable hard sedimentary rock used for core flooding experiments. When fired to stabilize clays (subjected to high temperatures), pyrometamorphical phase changes induce texture and pore framework alteration. As a consequence the new dielectric response may influence wettability. The literature regarding pyrometamorphical behavior during and after thermal treatment is ambiguous, so we evaluate desirable effects (fixation of clay minerals) and undesirable effects (dielectric surface changes) in the matrix. Porosity, permeability, surface charge, specific surface area and dielectric respond were measured before and after firing of samples up to ~1000 °C under oxidizing and non-oxidizing conditions. The matrix properties were determined using X-ray diffraction and X-ray fluorescence, scanning electron microscope imaging, and thermomechanical-, and thermogravimetric analysis with differential scanning calorimetry.

Firing causes dehydration, dehydroxylation and irreversible transformation of original clays, organic matter, and carbonates to glass, oxides and feldspars. During heating quartz transfers from α - to β -quartz and back during cooling. This changes the grain volumes and consequently reduces the matrix integrity. The sandstone has a slight porosity and permeability increase (~5%). Further, a shift in the point of zero charge toward a higher pH may result in wettability alteration from strongly water-wet to oil-wet. Additionally, a decrease in the permittivity value and marginal dispersion of the dielectric constant (~5%) between the high and the low frequencies was observed. Due to firing and related dispersion of the iron oxides within the matrix framework, Bentheimer sandstone becomes a weaker insulator.

1. Introduction

Bentheimer sandstone (BS) is widely used as a model rock for studying fluid flow in porous media and preparing 3D pore network models for theoretical studies (De Boever et al., 2013; Klein and Reuschlé, 2003; Ruedrich and Siegesmund, 2007). It is an easy to obtain, inexpensive sample material that has a matrix composed of well-sorted, mostly rounded to sub-rounded grains with a regular distribution of pore bodies and pore throats diameter (Peksa et al., 2015). The presence of accessory clay, organic matter and oxides or hydroxides is often ignored. Nevertheless, they influence petrophysical behavior such as permeability and electrical conductivity behavior.

1.1. Motivations for firing

Sandstone samples are heated to temperatures exceeding 500 °C as a common step in core preparations to limit the risk of permeability

reduction during displacement experiments (Potts and Kuehne, 1988; Sydansk). The objective is to remove the chemically bounded water and to transform and stabilize clays. A strong influence of clays in contact with water on sandstone porosity/permeability, pore size geometry and electrical behavior was reported in the literature (Al-Mjeni et al.; Civan and Knapp, 1987; Gabriel and Inamdar, 1983; Gray and Rex, 1966; Huntley, 1986; Klimentos and McCann, 1990; Neasham, 1977; Sharma and Yortsos, 1987; Wildenschild et al., 1999). The high cation exchange capacity of clays, the large surface area and strong interaction with interstitial fluids result in swelling and/or migration. In addition, permeability reduction can be expected because of pore clogging downstream, which causes serious injectivity problems (Gray and Rex, 1966; Jones; Mohan and Fogler, 1997; Mohan et al., 1993; Norrish, 1954). Clay migration was even found to clog high permeable sandstones (>0.5Darcy) (Gray and Rex, 1966). Note that besides clay fines, other components such as organic matter, very fine quartz and feldspars particles, are considered as mobile fines.

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Nomenclature

| | |
|-----------------------|---|
| α | Average coefficient of expansion, [-] |
| A | Effective area of the electrode, mm ² |
| $\Delta\epsilon_r$ | Dielectric dispersion magnitude |
| Δn | Dissolution rate, mmol |
| C_0 | Capacitance of space in a parallel-plate capacitor, pF |
| C_s | Capacitance of dielectric in a parallel-plate capacitor, pF |
| DAR | Dielectric Absorption Ratio, [-] |
| ϵ^* | Complex electric permittivity in the frequency domain, F/m |
| ϵ_0 | Permittivity of space, $\epsilon_0 = 8.85 \times 10^{-12}$ F/m |
| ϵ' | Real part of the electric permittivity, F/m |
| ϵ'' | Imaginary part of the electric permittivity, F/m |
| ϵ_r | Real value of the relative permittivity, [-] |
| $\epsilon_{r,i}$ | Imaginary value of the relative permittivity, [-] |
| \mathbf{E} | Electric field vector, V·m ⁻¹ |
| f | Frequency, Hz |
| F | Faraday constant, C/mol |
| G | Conduction, S |
| G_{AC} | AC conduction, S |
| G_{DC} | DC conduction, S |
| $g(t)$ | Dielectric response function of Bentheimer sandstone sample |
| \mathbf{I} | Total current density vector, A/m ² |
| I | Electric current, A |
| \mathbf{I}_C | Capacitive part of the current density vector, A/m ² |
| I_C | Capacitive part of the electric current, A |
| \mathbf{I}_L | Leakage part of the current density vector, A/m ² |
| I_L | Leakage part of the electric current, A |
| I_P | Polarization current through the sample, A |
| I_r | Insulation resistance, Ω |
| L | Sample length, mm |
| L_0 | Initial sample length, mm |
| $(\delta L/\delta T)$ | Slope of the expansion curve at the temperature, mm/°C |
| M | Mass, g |
| P | Polarization, C/m ² |
| PI | Polarization index, [dimensionless] |
| φ | Effective porosity, [dimensionless] |
| ρ | Bulk density, kg/m ³ |
| R | Lossy part of the dielectric, Ω |
| τ_r | Relaxation time, s |

| | |
|---------------------|--|
| S_s | Specific surface, m/m ² |
| σ_0 | Charge, C/m ² |
| σ | Electrical conductivity, S/m |
| T | Temperature, °C |
| t_p | Polarization duration, min |
| $\tan\delta$ | Dissipation factor, [dimensionless] |
| $\tan\delta_{pot}$ | Polarization losses on the dielectric, [dimensionless] |
| $\tan\delta_{cond}$ | Conduction losses on the dielectric, [dimensionless] |
| U | Voltage, V |
| V_m | Matrix volume, m ³ |
| ω | Angular frequency, Hz |
| | Abbreviations |
| AC | Alternating current |
| Ant-Rt | Rutile formed as a result of the polymorphic transformation of anatase |
| BET | Brunauer-Emmett-Teller |
| BS | Bentheimer sandstone |
| CTE | Coefficient of thermal expansion |
| CT | Computed tomography |
| D | Dimension |
| DC | Direct current |
| DSC | Differential Scanning Calorimetry |
| F | Fired samples |
| Fsp | Feldspar |
| G | Glass |
| H^+ | Hydrogen ion |
| Kln | Kaolinite |
| M | Molar concentration |
| Mul | Mulite |
| PZC | Point of zero charge |
| Qtz | Quartz |
| Rt | Rutile |
| SEM | Scanning Electron Microscope |
| SOL | Dissolution |
| TGA | Thermogravimetric analysis |
| UF | Unfired samples |
| WL | Wave length |
| wt% | Weight percent |
| XRD | X-ray diffraction |
| XRF | X-ray fluorescence |

1.2. Firing controls on wettability

Firing of the sandstone caused disintegration of clay minerals, a partial transformation of feldspar and quartz into glass and iron oxides. Moreover, according to [Barclay and Worden \(2009\)](#), if the specific surface of the iron oxides increases, a positive correlation of oil-wetting iron mineral content in the sandstones is observed. [González and Moreira \(1991\)](#) reported iron oxides to be strongly oil-wet due to substantial adsorption of asphaltenes. They observed a significantly higher maximum of asphaltenes adsorption for hematite than for clay minerals (up to 1.5 times) ([González and Moreira, 1991](#)). According to [Wang and Guidry \(1994\)](#), the mechanisms of the strong oil-wetness of iron oxides can be explained by (1) ferric ions, allocated on the mineral surface. They represent possible adsorption sites for carboxylic acid anions which can change the wettability of the mineral surface; (2) ferric ions, mainly on the surfaces. They can cause oxidation of the petroleum components using catalysts and produce polar wettability-altering compounds; (3) ferric ions in solution. They may behave as bridges between petroleum and a silica surface. In addition, it must be noted that the wettability state of the reservoirs containing ferric ions (Fe^{3+}) is indirectly controlled by the chemistry i.e. redox state, of the

reservoir fluids ([Barclay and Worden, 2009](#)).

1.3. Firing characterization

Following previous studies ([Ma and Morrow, Shaw et al.; Wu and Firoozabadi, 2010](#)) that high temperatures cause changes in mineralogical composition of sandstones and in physical and electrical properties, we decided to fire the Bentheimer samples to temperatures where all silica and carbonate minerals but quartz are dehydrated, dehydroxylized, decarbonized and disintegrated. For Bentheimer sandstone, the information on pyrometamorphical and electrical properties has been found available in the literature is rather limited ([Loahardjo et al., 2012; Maloney et al., 1990; Olafuyi et al., 2010](#)). In particular we are not aware of published works on the role of high-temperature minerals, resistive to chemical weathering, within its framework after firing and their effect on wettability. To fill the gap in the understanding of these systems, we decided to perform a series of thermal treatment at temperatures ≥ 600 °C and petrophysical, petrological and dielectric experiments.

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