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Research paper

Using micro-computed tomography and scanning electron microscopy to assess the morphological evolution and fractal dimension of a salt-gypsum rock subjected to a coupled thermal-hydrological-chemical environment

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ABSTRACT

It is well known that salt-gypsum evaporites play a significant role in controlling the physio-mechanical performance of the earth's upper mantle as well as forming extensive "halokinetic" structures that are directly linked to lithologic hydrocarbon reservoirs and petroleum accumulation. Under many geological conditions, hot saline groundwater frequently intrudes into salt-gypsum evaporite sequences. However, relatively little is known regarding the micromechanical behaviour of salt-gypsum in these saline environments, and this behaviour governs the macro-mechanical behaviour and the overall deformation. In this study, we examine the microstructural evolution of salt-gypsum and its weakening mechanisms under varied brine conditions (or coupled thermal-hydrological-chemical environments). A series of laboratory tests, including scanning electron microscopy and micro-computed tomography (MCT), were conducted to study how the petrographic characteristics, including porosity, pore size distribution and fractal dimension, evolved. In total, 81 specimens were prepared and then soaked in brines of 3 different concentrations and at 3 different temperatures. The results showed the following: 1) after brine treatment, the MCT slices of the specimens generally contained four areas: a residual porous skeleton area, an undissolved area, a cracked area and an interface area. 2) For a given concentration, the porosity and fractal dimensions of the specimens gradually increased with temperature, while for a given temperature, the porosity and fractal dimension tended to decrease as the brine concentration increased. 3) Because the nucleation or initiation rate of new voids was slower than the growth and coalescence rate of the original voids, the proportion of 0–1 μm pores gradually decreased over time. However, the proportion of 5 to μm pores gradually increased over time. To study the effects of each factor and the interactions between them on the response variables (porosity and fractal dimension), 2×2 and 2×3 factorial designs were employed to assess the brine concentrations and temperatures. The results verify that the water temperature significantly weakened the salt-gypsum, while the chlorine ions had a much weaker effect.

1. Introduction

Salt-gypsum is an evaporite mineral commonly found in layered sedimentary deposits in association with halite, anhydrite, sulphur, calcite and dolomite. It is a soft sulphate mineral composed of sodium chloride and calcium sulphate dihydrate (see Fig. 1). Salt-gypsum sequences act as weak decollement horizons and thus play a significant role in controlling the physio-mechanical performance of the earth's upper mantle, foreland fold-thrust belts and nappes. Examples include the margin of Meso-Cenozoic sedimentary basins in north-western China, the southern French Alps (Beach, 1981), and the Greek Hellenides (Underhill, 1988). Additionally, studies of tectonic-geologic frameworks and petroleum reservoir geology show that halokinetic structures, such as pillows, domes and diapirs, are generally observed in gypsum sequences. These special structures are directly linked to lithologic hydrocarbon reservoirs and petroleum accumulation, as seen in the Sverdrup Basin of northern Canada (Balkwill, 1978). Notably, in deep geological settings, salt-gypsum is almost always associated with high temperatures and other mineral substances such as sodium chloride. There is frequently evidence for the intrusion of hot saline groundwater during salt-gypsum evaporite sequences (Jordan, 1988, 1991; Wang et al., 2015; Meng et al., 2015, 2016; Shi et al., 2018).

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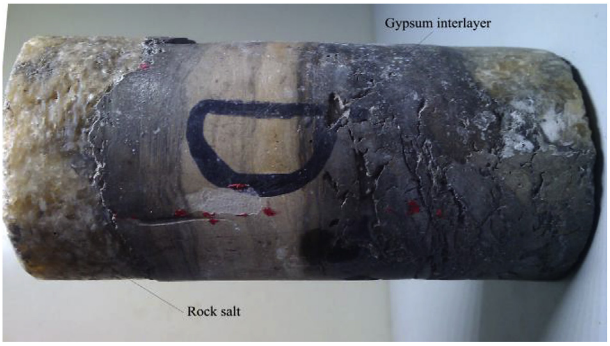

Fig. 1. Salt-gypsum in Yunying salt deposits (Meng et al., 2015).

Accordingly, salt-gypsum will be exposed to saline brines at different temperatures and concentrations. These saline brines greatly affect the strength and deformability of salt-gypsum by influencing its petrographic characteristics, including microstructure, granular minerals, composition and morphology, which will ultimately affect hydrocarbon migration and accumulation. However, relatively little is known regarding the micromechanical behaviour of salt-gypsum in these saline environments, even though this behaviour governs the macro-mechanical behaviour and overall deformation.

For these reasons, there is considerable interest in the microstructural response of salt-gypsum to various saline brine concentrations and temperatures—that is, subjected to a coupled thermal-hydrological-chemical (THC) environment—and the corresponding microstructural evolution and weakening mechanisms (Meng et al., 2017). Understanding these factors will assist in modelling fractures in various strata and hydrocarbon migration. Therefore, it is important to investigate the relationship between the coupled THC environment and the petrographic characteristics of salt-gypsum rocks, such as microstructure, texture, and morphology, and assess how these variables influence the macro-mechanical properties.

Previously, several researchers have studied the mechanical behaviours and weakening mechanisms of gypsum in a THC environment. For example, in the mining and civil engineering fields, studies have examined the long-term mechanical behaviours and aging processes of gypsum pillars under varying relative humidity. It was concluded that, due to the hydro-sensitive nature of the gypsum, its pore structures and crystal morphology were closely related to the relative humidity and the intensity of the dissolution traces (Auvray et al., 2004, 2008; Hoxha et al., 2005, 2006). However, the effects of saline brines on the mechanical properties of the gypsum were not considered. Siese et al., 1999 investigated the creep behaviour of a wet granular gypsum based on the saline brine concentration. They showed that the NaCl content depended on the mechanical response of the gypsum and concluded that the creep rate increased with brine concentration. However, the study used crushed and artificially synthesized gypsum, which may not reflect the mechanical responses of natural gypsum. Additionally, the test gypsum was crushed, sieved, washed, and then compacted to form samples with a fixed particle size. The gypsum samples made in this manner had a porosity of approximately 43%, while natural intact gypsum has a porosity of approximately 2%. According to Hoxha et al. (2006), the difference in porosity between synthetic gypsum and natural gypsum may lead to different weakening mechanisms. In addition, the effect of brine temperature on the weakening mechanism of the gypsum was neglected. Wang et al., 2018a,b,c,d first proposed a mathematical model to predicate the collapse process of salt cavern roof. His model is very original, advanced and useful, which can provide the theoretical direction for the stability and safety of salt cavern in china (Wang et al., 2018a, 2018b; 2018c, 2018d). Liang et al., 2012 examined the macro-mechanical properties of natural gypsum soaked in varying saline brines. They suggested that the saline brine concentration strongly affects the geotechnical parameters of the gypsum, and the corrosion damage to the gypsum was greatest when soaked in a saturated saline brine. Conversely, using scanning electron microscopy (SEM), Yu et al., 2016 argued that a semi-saturated brine had the strongest corroding effect on gypsum. The causes for this discrepancy could be related to the following factors: 1) the experimental results discussed above were limited to the variation in surface topography, while the evolution of the internal pore structures remained unclear. Additionally, SEM tests can only provide qualitative rather than quantitative evidence. 2) Only three samples were used for each test condition. Given the strong heterogeneity of gypsum, the experimental data may thus have yielded inconsistent results. Gill et al. (2005) showed that the number of test trials should depend on the degree of importance of the study or engineering project; the precision index p should be less than or equal to 1.20 (i.e., the number should be at least 9) for underground construction studies, based on the small sampling theory. Therefore, the influence of saline brine concentration and temperature on the mechanical behaviour of gypsum and the corresponding weakening mechanisms have not been unambiguously identified. Given these factors, questions remain about how the pore microstructure of the gypsum changes in a coupled THC environment. Does the saline brine temperature and concentration strongly affect the pore microstructure, and eventually the mechanical properties? What are the key factors associated with coupled THC effects? To address these questions, it is necessary to study how the pore microstructure of salt-gypsum soaked in a saline brine evolves with varying concentration and temperature.

Based on literature regarding microstructures and fractal theory, it is well known that the porosity and fractal dimension can reflect the macroscopic mechanical properties, the degree of damage, and the weakening mechanisms of rock materials (Xie et al., 1997, 1999; 2001; Bao et al., 2018; Meng et al., 2018a, 2018b). Although natural gypsum has strong heterogeneity and low porosity, it has been shown that the porosity and fractal dimension can quantitatively describe the complexity and irregularity of the pore structure of a tight rock (Liu et al., 2016b). Additionally, many mechanical models incorporate the porosity and fractal dimension, including the Costa, Schueter and Xu-Yu models. Including these factors can greatly improve the estimation of mechanical properties (Costa, 2006; Schlueter et al., 1997; Xu and Yu, 2008).

In summary, the aqueous environment can greatly affect the physical properties of a rock, and the porosity, pore size distribution and fractal dimension are important microstructural indicators (Mata et al., 2005; Frydman et al., 2014; Liu et al. 2015, 2016a; Menéndez and Petráňová, 2016). To the best of our knowledge, few studies have addressed the microstructural evolution and weakening mechanisms of salt-gypsum under a coupled THC environment. The response of minerals to any chemical depends on their crystal structure and chemical composition. Thus, in this study, a series of laboratory tests, including micro-computed tomography (MCT) scanning and SEM, were conducted to determine the porosity, pore size distribution, and fractal dimension of salt-gypsum exposed to hot saline brine and to discuss the corresponding weakening mechanisms.

2. Methods and materials

2.1. Test equipment

The main equipment used in this study was the μCT225kVFCB high-accuracy (μm grade) computed tomography (CT) test analysis system (see Fig. 2), consisting of a digital flat detector, a micro-focus X-ray machine, a high-precision work turntable and fixture, an acquisition and analysis system, and other structural components, as shown in Fig. 2 (Yu et al., 2012; Kang et al., 2017). The micro-focus X-ray machine in the micro-CT test system allowed weak currents (only 0.01–3.00 mA), issuing a small, conical X-ray beam and then projecting the scanned image to the digital flat-panel detector for display. The minimum focus size of