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Microstructural characteristics of wellbore cement and formation rocks under sequestration conditions

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ABSTRACT

One of the most important aspects of the interaction between well-bore cement and the formation rock are the porosimetric parameters of both materials. Porosity and pore-size distribution affect the behaviour of the porous solid material, including the fluid movement and flow. In order to analyse the porosity of rock and cement material in the presented investigation the following procedures were applied: mercury intrusion porosimetry (MIP), gas adsorption technique and X-ray microtomography (XMT). The investigation proved the usefulness of XMT method, which enables the microstructural characterisation of the composed (cement–rock) samples during the subsequent stages of the experiment. Imaging the cement degradation in the rock–cement contact zone is possible, thanks to the differentiated densities of the altered zones, resulting from the rock or cement dissolution, and new phases precipitation.

Five rock samples of different lithology (quartzitic sandstone, eolian sandstone, shale, limestone and anhydrite) were selected for the examination. The prepared samples composed of well bore cement and rock were exposed to CO_2-saturated brine, under static conditions. Basing on the shape of the gas adsorption isotherms the classification of the materials (cement and rocks) was performed. In the case of the rocks classified as macroporous, because of the porosity characteristics close to those of cement, the CO_2 saturated brine flow is uniform in both materials (cement and rock), and the dissolution and precipitation processes are less intense. The obtained results seem to be very useful for predicting the integrity of wellbore, in case of different caprock and reservoir rock.

1. Introduction

Subsurface carbon sequestration in abandoned gas and oil fields is applied to reduce the atmospheric CO_2 concentration. The gas is injected into deep geologic formations, including abandoned petroleum reservoirs, deep saline aquifers and coal seems. The injected CO_2 tends to escape upwards into the overlying formations and back to the atmosphere. Wellbores are the most likely pathways of CO_2 leakage in the process of sequestration, as the probability of CO_2-saturated brine to interact with wells is high (Zhang and Bachu, 2011). The rate of CO_2-saturated brine to interact with wells is dependent on dissolved CO_2 concentration, temperature, existence of impurities (O_2 and SO_2/H_2S), and the composition of the cement (Kutchko et al., 2008, 2009; Jacquemet et al., 2012; Zhang et al., 2014). The permeability of the cement, as well as the integrity of the cement-casing or cement-host rock system will determine the leakage possibility. The original cement permeability is extremely low; hence, the flow through the fractures is the dominant pathway of possible leakage. Some authors (Duguid and Scherer, 2010; Kutchko et al., 2008) suggest that, though carbonic acid does react with the cement, a low permeability is limiting degradation on the time scales relevant to CCS for a properly completed well annulus. However, if there is no full adhesion in the contact zone between cement and formation rock, the fracture can occur. This fracture permeability alters as a consequence of chemical and mechanical processes in the contact zone. Experimental data of Huerta et al. (2013, 2015), Carey et al. (2009), Cao et al. (2015) and Newell and Carey (2012) suggest that leakage of CO_2-rich fluids along a wellbore may seal the leakage pathway, if the initial fissure is small and the residence time allows the mobilization and precipitation of minerals along the fracture.

One of the most important aspects of the interaction between well-bore cement and the formation rock are the porosimetric parameters of both materials. Porosity and pore-size distribution affect the behaviour of porous solid material, including the fluid movement. The performance of wellbore cement is significantly

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controlled by its microstructure, in particular the pore network plays a critical role in determining the mechanical properties and interactions with the fluids (Gallucci et al., 2007; Jung et al., 2014).

The goal of the presented study is to determine the influence of the formation rocks' microstructure on wellbore cement–rock integrity under sequestration conditions. In order to analyse the porosity of rock and cement material the following methods were applied: mercury intrusion porosimetry (MIP), gas adsorption technique and X-ray microtomography (XMT). MIP is the widely used method for pore-size analysis of macroporous rocks (e.g. sandstones, limestones) and cement stone. The mercury porosimeters can theoretically generate the pressure of 414 MPa (60,000 psi), covering the pore diameter range from approximately 0.003 to 360 μm. The intrusion of mercury in the pore structure is controlled by pore throats (the smallest dimension in an irregular pore) (Washburn, 1921), while the adsorption phenomenon during the subcritical N_2 gas-adsorption technique is controlled by the pore body (Kuila and Prasad, 2013). The limitation of the gas-adsorption technique is the inability to measure the large pores (over 200 nm). A combination of the mercury intrusion and gas-adsorption gives information on the entire pore structure, especially on microporous rocks, such as clays and shales (Darłak et al., 2012). It is worth mentioning, however, that the direct comparison of the pore volumes from these two measurements is not possible, as a consequence a different record technique (Kuila and Prasad, 2013).

In order to identify the changes in the structure of the cement–rock contact zone, the μ-CT method was used. The studies of Cao et al. (2013), Jung et al. (2013), Mason et al. (2014) and Yalcinkaya et al. (2011) have demonstrated that XMT is capable of non-destructively visualizing the spatially heterogeneous degradation of wellbore cement material, including the density and pore structure changes, due to CO_2 attack under geologic sequestration conditions. This method enables the visualization of potential leakage pathways, which can be formed at the cement–rock interface, as a consequence of degradation processes. Mason et al. (2014) performed the flow-through experiments, showing that the distinct alteration is visible after 8-days experiment. They used the sophisticated XMT image segmentation method combined with models of cement alteration, which is in contact with caprock (calcite-cemented sandstone). Their observations show that the uniform models, such as those proposed by Walsh et al. (2014), appropriately describe the reactions observed in the cement samples. Mason et al. (2014) used the values of "effective" linear activity coefficient (ELAC) to estimate material proportions from the XMT images. These authors (Mason et al., 2014) focused their studies on wellbore cement, because the caprock remained nearly unaltered, and was not discussed; while we were encouraged to study also the alteration of rock which is in contact to cement (Fig. 1).

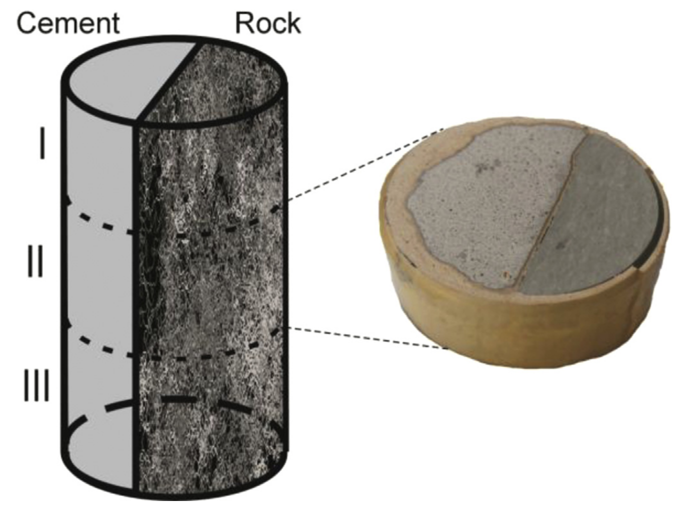

Fig. 1. Example of cement–rock cylindrical sample with a diameter of 2.54 cm and a height of 4 cm. The sample is divided into three parts for each stage of the experiment.

2. Materials and methods

2.1. Formation rocks and wellbore cement material

Five rock samples of different lithology (quartzitic sandstone, eolian sandstone, shale rock, micritic limestone and anhydrite) were selected for the examination. The samples represent reservoir and sealing rocks for hydrocarbons deposits in Poland. The stratigraphic position and geological situations are presented in Fig. 2.

The rock samples were prepared in the form of cylinders with a height of approximately 4 cm and a diameter of 2.54 cm. Rock cores were cut lengthwise in half and supplemented with cement grout (Fig. 1). The cut surface of the rock was cleaned of dust, but not polished, so as to provide better adhesion for added cement. The cement grout was composed of the following materials: tap water (w/c ratio 0.52), defoamer, fluid loss additive, plasticizer, latex, latex stabilizer, retarder, microcement, swelling additive and the Portland cement CEM I 32.5. The grout was prepared in accordance with the procedure laid down in the relevant standards (DIN EN ISO 10426-2). The samples were cured in an autoclave at 50 °C, at a pressure of 17 MPa. Curing time under these conditions was 48 h.

From the prepared samples thin cuts were made, observable under polarizing microscope Axioskop by Zeiss. The aim of the observation was to determine the mineral composition of the rock and to observe the zone of rock–cement contact. The rest of the sample was sliced in the form of about 1 cm thick plates, and placed in the reactor, according to the schedule listed below.

The composed samples were also tested on each stage of the experiment with the electron microscope. SEM analysis were performed with Scanning Electron Microscope FEI Quanta-650 FEG, equipped with the analyzers: energy dispersive analyzer (EDX)-EDAX Galaxy.

2.2. Experiment conditions

The prepared samples composed of wellbore cement and rock were exposed to CO_2-saturated brine, under static conditions. The experiment was performed in an autoclave reactor, which was constructed in the laboratories of VSB Ostrava. After initial petrographic and petrophysical analyses the samples were placed in the reactor with brine (NaCl concentration of 105 g/l) saturated with CO_2 (0.8 mol CO_2/kg of solution). The initial pH of the solution was 3.4. The conditions of the experiment were: 50 °C and 10 MPa. Provided the average increase of the temperature in the well of about 3 °C for each 100 m, it can be assumed that the temperature of 50 °C is corresponding to the depth of 1000 m below the ground level in static conditions. The assumed conditions (50 °C and 10 MPa) can be present in the wellbore in the case of deep carbon injection and storage depth (Jung and Um, 2013). The selection of static conditions for the experiment is justified by the fact that in deep wellbores for CO_2 storage, the flow velocity of formation water is typically in the order of millimeters to centimeters per year, due to the low permeability of rocks (Bachu et al., 1994). For such low flow rates, the reactions are regarded as similar to those