A systematic workflow in quantifying surface area and pore-size distribution using the nitrogen adsorption experiment focused on clay rich mudrocks

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Introduction

With increasing interest in shale reservoirs, the need for understanding pore structures of shales becomes crucial. Pore structures can be studied using many established techniques, such as NMR methods, mercury intrusion porosimetry, electron microscopy, and x-ray tomography. However, shale formations are shown to have pore sizes in nanometer scales, which are below the resolutions of most conventional pore characterization methods. A quantitative method proven to be useful in characterizing nanopores in terms of their specific surface area (SSA) and pore-size distribution (PSD) is nitrogen adsorption. The theories behind this method have been extensively studied for common adsorbents since the 19th century, and the applicability of it on rocks is shown in the literature, but not without some challenges.

Methods

Nitrogen adsorption is conducted in two different instruments, namely Quadrasorb EVO/SI and Micromeritics ASAP 2020^{TM}. The experimental method consists of two parts: degassing the material and gas adsorption at a subcritical temperature. Outgassing ensures the removal of physically adsorbed unwanted vapors and gasses from sample's internal and external surface. Sample material can either be prepared in powder or pellets. It will be outgassed at a temperature of 200°C for a minimum of 12 hours under a vacuum of <0.005 Torr/min. This is a standard procedure, but the length of time and temperature can vary depending on the sample. Too high of a temperature may do irreversible damage to the samples. After degassing, the tubes are transferred to the instruments, where adsorption takes place. Several techniques of data inversion process will be discussed as a method of analysing the data.

Results

BJH model is a widely used standard model and so the results are easy to compare. However, it does not account for micropores and for pore geometry. On the other hand, the family of DFT-models are used to combine all pore sizes.

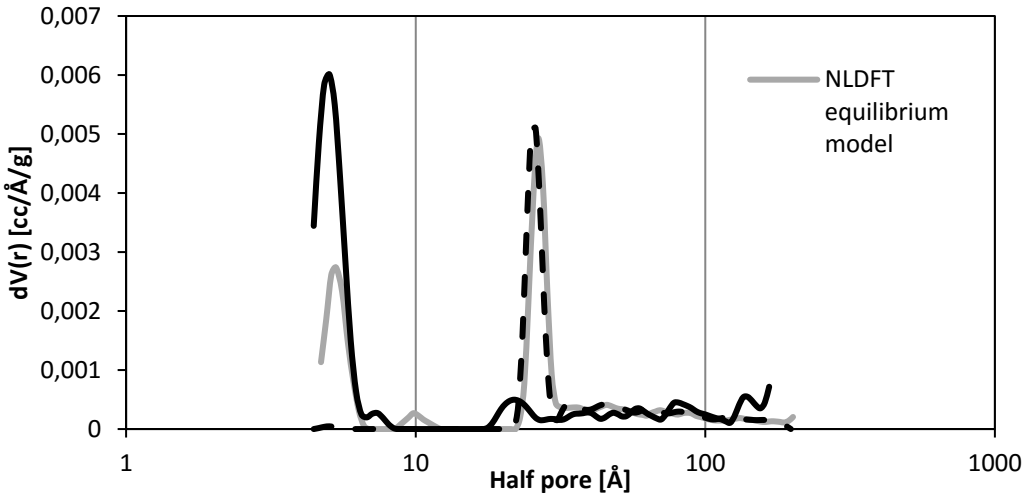

Figure 1: Example of for the Na-rich Montmorillonite shown above illustrates the variations in PSD that can arise from each model.

Discussion

The standard procedures used to inverse SSA and PSD obtained from the nitrogen adsorption on clay rich mudrocks are based on the classical thermodynamics models, a near idealization of rock-fluid interactions. Inherently, this idealization creates quantifiable errors. Recent advancements in adsorption techniques described by the 2015 IUPAC publication can be integrated into rock physics to address these challenges: (i) Nitrogen gas has been widely used as the adsorptive for pore characterization, however, the fluid – rock interaction due to quadrupole moment of nitrogen molecules is not accounted in the BET method for SSA inversion; (ii)the preferred method to inverse PSD, BJH technique, ignores pore geometry and its effect on pore confined thermodynamics; (iii)the inability of the classical methods to encompass the whole range of micro-mesopore scales.

Conclusions

The main focus of this paper is to present a systematic workflow, which integrates both classical and statistical thermodynamics based methods, to guide users in obtaining repeatable and justifiable results.

References

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