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Assessing mechanical properties of organic matter in shales: Results from coupled nanoindentation/SEM-EDX and micromechanical modeling

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ABSTRACT

Mechanical properties of organic matter in fine grained source rocks has been the subject of extensive research. Most studies on kerogen have been performed on kerogen samples isolated by dissolving the rock matrix. However, recent studies have shown significant microstructural alteration of kerogen samples after demineralization leading to alteration of their mechanical properties. In this work, in order to study the role of organic matter on mechanical properties of the rock, both organic-rich and organicfree carbonate-rich shale rocks are investigated. A recently developed nano-chemomechanical characterization method is used to directly correlate mechanical properties of both organic-rich and organic-free samples with their chemical composition and mineralogy at micrometer length scales using coupled nanoindentation and Energy Dispersive X-ray Spectroscopy (EDX) technique. The experimental results are used in multiscale structure thought models in which the role of organic matter is modeled as a matrix surrounding inclusions in the composite. Application of these models to the interpretation of nanoindentation results allows us to obtain consistent mechanical properties for the main constituents of these samples, i.e. organic matter and calcite, which are in excellent agreement with the results in the literature. Moreover, adequate agreement is observed between model predictions and experimental measurements of elastic properties of carbonate-rich shale rocks at macroscale.

1. Introduction

One of the most challenging tasks in developing unconventional reservoirs hosted by organic-rich shales is the accurate prediction of mechanical properties of composite shale materials (Sone and Zoback, 2013; Ahmadov, 2011; Vernik and Milovac, 2011). Sufficient knowledge about mechanical properties, such as elasticity and strength of shales is essential to the success in many aspects of drilling, seismic exploration, and production. However, prediction of mechanical properties of organic rich shales is intricate due to their complicated chemistry, extremely heterogeneous microstructure, and multiscale mechanical performances. Such complexity requires advanced and innovative experimental and theoretical tools for a complete understanding of the role played by different constituents (i.e. organic and inorganic components) in the chemo-mechanical properties at multiple scales.

Despite extensive research, mechanical properties of organic-matter are not fully understood due to the small size and delicate nature of kerogen within organic-rich shales. Most studies on kerogen have been performed on kerogen samples isolated by dissolving the rock matrix. However, recent findings have shown significant microstructural alteration of kerogen samples after demineralization (Thomas et al., 2014) which affects their mechanical properties. Recently, however, new advances in in-situ characterization of mechanical properties have provided an opportunity to probe directly physical properties of shales at micrometer and sub-micrometer length scales (Abedi et al., 2016a; Eliyahu et al., 2015; Wang et al., 2017).

In this study, mechanical properties of immature kerogen aggregates are quantified through coupled experimental and micromechanical analysis of kerogen/mineral composite in organic-rich shales. In order to study the role of organic matter, both organic-rich and organic-free Eagle Ford samples are investigated. Employing a recently developed nano-chemomechanical characterization technique (Abedi et al., 2016a), mechanical properties of both organic-rich and organic-free samples are directly correlated with their chemical composition and mineralogy at micrometer length scales using coupled nanoindentation and EDX. The extensive data sets obtained by experiments are inferred using statistical

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tools such as clustering analysis to categorize different chemo-mechanical phases present within the probed region.

Our investigation employs two different multiscale structure thought models for organic-free and organic-rich Eagle Ford samples (Fig. 1), with organic-free samples represented with granular morphology (Self-Consistent scheme) and organic-rich samples modeled with matrix-inclusion morphology (Mori-Tanaka scheme). This morphological attribution is in agreement with recent observations regarding the connected texture of immature organic-rich samples (Prasad et al., 2009, 2011; Prasad and Mukerji, 2003; Zargari et al., 2013). The three-level models extend from the scale of elementary building blocks of organic-free and organic-rich Eagle Ford shales (Level 0) to the scale of macroscopic composite (Level II). The scale separability condition is satisfied within the models for the application of micromechanics tools; that is, the characteristic length scale of each level is much smaller than the characteristic length scale of the next level. In case of organic-free Eagle Ford, Level 0 corresponds to calcite particles at nanometer length scale. In turn, Level I corresponds to the sub-micrometer scale of the porous calcite composite. This scale is the scale of chemo-mechanical characterization technique employed in this study. Finally, Level II represents the characteristic length scale in the millimeter and sub-millimeter ranges. At this scale, the material is a porous calcite composite intermixed with hard/soft inclusions (e.g. quartz, pyrite, etc.). This scale is the scale involved in shale acoustic measurements using compressional and shear wave velocity data. On the other hand, in case of organic-rich Eagle Ford, Level 0 represents kerogen aggregates whereas Level I and Level II correspond to porous calcite/porous kerogen composite and porous calcite/kerogen composite plus hard/soft inclusions respectively.

Experimental results at Level I are fed into micromechanics-based inverse models to infer mechanical properties of the main constituents of these samples, i.e. calcite particles in case of organic-free samples and kerogen aggregates in case of organic-rich samples. The obtained results are in excellent agreement with the reported values in literature which validates the micromechanical approach developed in this study. Moreover, the results are used in a forward modeling approach to predict mechanical properties of carbonate-rich shales at macroscale and the results show adequate agreement between model predictions and experimental values.

Table 1
Mineralogy, porosity and TOC measurements of Eagle Ford samples. The mineralogy and porosity data were obtained by XRD, pycnometer and dry bulk density measurements, respectively.

	Quartz (wt%)	Calcite (wt%)	Clay (wt%)	Other (wt%)	TOC (wt%)	Porosity (%)
Organic-free Eagle Ford-Sample E	3.6	94.4	1.1	0.9	0	5.2
Organic-rich Eagle Ford-Sample B	25.6	65.1	1.5	7.8	3.14	8.99
Organic-rich Eagle Ford-Sample D	10	74.9	2.6	11.5	1.52	10.4

2. Materials and methods

2.1. Samples specifications

Our study was conducted on sets of outcrop organic-rich and organic-free Eagle Ford (located in west Texas) samples. Table 1 compiles relevant experimental information regarding the material composition, TOC, and porosity of the samples. Based on the results of Rock-Eval Pyrolysis, organic-rich Eagle Ford samples are characterized as thermally immature outcrop samples.

The data regarding the composition of the studied samples can be transformed into volume fractions. The volume fraction of the *i*-th mineral in the samples can be obtained from:

$$\eta_k = \left(1 - \phi^{II}\right) \frac{m_k/\rho_k}{\sum_{i=1}^{N} m_i/\rho_i} \tag{1}$$

where N denotes the number of solid constituents in the sample, m_i are the mass fractions provided by e.g. X-Ray Diffraction (XRD), and ρ_i denote the corresponding solid densities (in this study, densities of 2.65, 2.65, 2.71 g/cm^3 are considered for clay (which is represented by illite as the main clay component), quartz and calcite respectively), and ϕ^{II} is the porosity at macroscale, Level II (porosity of the bulk material). The density of kerogen is assumed to be 1.2 g/cc (Abedi et al., 2016a; Vernik and Landis, 1996). Table 2 summarizes the volume fractions of the detected minerals and organic matter in organic-free and organic-rich Eagle Ford samples.

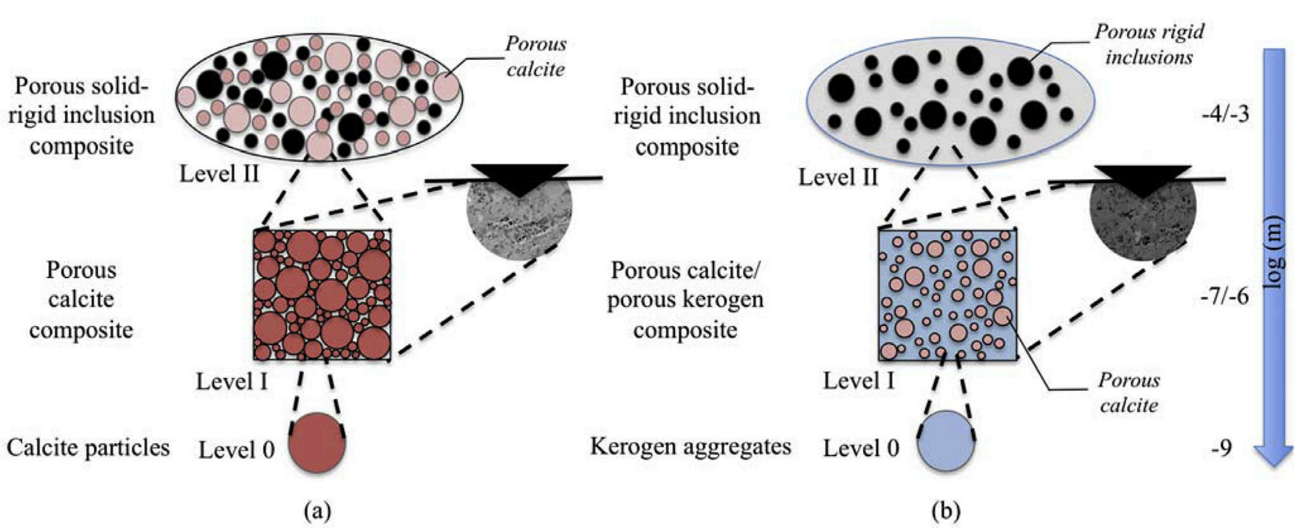

Fig. 1. Multiscale thought model of a) organic-free Eagle Ford: Level 0 corresponds to calcite particles at nanometer length scales. Level I is the sub-micrometer scale of the porous calcite composite. Level II represents the porous calcite composite intermixed with hard/soft inclusions (e.g. quartz, pyrite, etc.) b) organic-rich Eagle Ford: Level 0 represents kerogen aggregates whereas Level I and Level II correspond to porous calcite/kerogen composite and porous calcite/kerogen composite plus hard/soft inclusions respectively.