

Impact of mineral composition and distribution on the mechanical properties of porous media

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Abstract. Geological sequestration of CO₂ in deep saline formations is a promising means of reducing atmospheric CO₂ emissions. Once injected, CO₂ dissolves into formation brine, lowering pH and creating conditions favorable for mineral dissolution. Cations released from dissolving minerals may create conditions favorable for secondary mineral precipitation, which can result in the long-term mineralogical trapping of injected CO₂. These reactions may alter the natural rock mechanical properties, which can affect the safety and efficiency of geological sequestration. This work aims to investigate the impact of mineral composition and distribution on the mechanical properties of porous media. In this study, the mineralogy, mineral distribution, and mechanical properties of samples from Escambia County, AL, are evaluated. The mechanical properties of the rock samples are evaluated using the unconfined compression and indirect tensile tests in the combination with digital image correlation. The mineral composition and distribution are determined through the analysis of scanning electron microscopy backscattered electron and energy dispersive X-ray spectroscopy images of thin sections. These analyses showed that the mechanical properties vary with composition, which may have significant practical consequences for geological sequestration of CO₂.

1 Introduction

Rapid industrialization and human activities, such as excessive use of automobiles and burning of coal, gases, and oils, have caused an unprecedented increase in the emission of greenhouse gases, especially carbon dioxide (CO₂), to the atmosphere causing significant challenges such as climate change and global warming. Scientists and researchers around the globe have developed various methods to tackle this situation [1]. To date, one of the suitable and economically feasible solutions is known as carbon capture and storage in underground rock formations [1, 2]. Nevertheless, in spite of its excellent environmental benefits, CO₂ capture and storage in underground geological formations implementation is limited. This is in part due to a lack of understanding of fundamental water-rock-CO₂ interactions, including, for example, the effect of clay mineralogy on the coupled hydro-mechanical phenomena [3-5]. Injection of CO₂ in saline aquifers results in the formation of carbonic acid and subsequent dissolution of primary minerals and alterations of the natural rock mechanical properties. These processes may affect the safety and efficiency of the geological sequestration process.

The strength of reservoir rocks after CO₂ injection was found to decrease with an increase in the porosity of the sample [6, 7]. This was a result of the dissolution of the rock minerals when the rock samples were treated with a CO₂ saturated brine solution. It should be noted,

however, that the short-term effect of CO₂ on rock cores is anticipated to be negligible compared to the long-term effect [6, 8]. As time progresses, secondary minerals may precipitate from released ions, again altering the properties of the formation. The impact of the mineral dissolution and precipitation reactions on formation properties, including strength, however, is not well understood [9].

In recent years, several scholars have worked on this topic and researched the relationship between mineralogy, microstructure, and rock mechanics properties [10-12]. Lindqvist et al. studied the relationship between rock intrinsic properties, such as mineralogy and microstructure, and functional and durability properties of stone and rocks [10]. Johansson showed that mineralogy, micropore porosity, grain size, and as well as shape are the most important factors influencing rock mechanics properties [11]. The study by Tuğrul et al. revealed that the textural characteristics of the rock have a higher effect on the rock engineering properties than the mineralogy. They have also found that the types of contacts, mineral shape and size significantly influence the engineering properties of the granitic rocks [12]. Although many significant results have been obtained by numerous researchers in the past, there are still many unknowns in the fundamental theoretical research and production process. Therefore, the research on rock's mineralogy, structure, porosity,

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and rock mechanics, especially the relationships among them, are needed.

The main objective of this research is to investigate the effect of mineralogy on the mechanical properties of geologic porous media to enhance predictions of the impact of mineral transformation on strength. In this study, the mineralogy, mineral distribution and mechanical properties of three samples from Escambia county, AL, are evaluated. The mechanical properties of the rock samples are evaluated using the unconfined compression and indirect tensile tests. The mineral composition and distribution are determined through analysis of scanning electron microscopy (SEM) backscattered electron (BSE) and energy dispersive X-ray spectroscopy (EDS) images of thin sections. These analyses show that the mechanical properties vary with composition, which may have important practical consequences for geological sequestration of CO₂. The results from these analyses will be integrated into a constitutive model based on the chemo-plasticity framework to consider how the composition and mechanical properties will evolve after reaction with CO₂ acidified brine.

2 Materials and methods

2.1 Rock samples

In this work, two different rock formations were investigated. Samples have been collected from a depth of 1.3 km in the north of the Escambia County, AL.

2.2 Scanning electron microscope imaging

Core samples from each formation were examined using scanning electron microscopy to determine the general composition and mineral distribution of each formation. For this, unfractured cores were sent to Applied Petrographic Services Inc. where they were cut into thin sections and polished. Once prepared, BSE and EDS images were taken using the SEM. The BSE images provide detailed grayscale images from which porosity can be readily determined and the EDS images provide elemental maps that can aid in mineral classification. Here, Matlab and ImageJ were used for image segmentation. In the BSE images, most mineral phases were easily distinguishable by grayscale and images of each individual phase/value were first created from manually thresholded BSE images using ImageJ. Background noise was then removed using Matlab image processing codes. Images of each major mineral phase were then compiled into a single image where unique colors were assigned to each mineral phase using Matlab. Mineral volume fractions were then determined by pixel counting.

2.3 Mechanical testing

To determine mechanical properties of samples, unconfined compression and splitting tensile tests were conducted as detailed below.

2.3.1 Unconfined compression test

The uniaxial or unconfined compression test is a simple laboratory testing method to assess the mechanical properties of rocks. Here, cylinder specimens having a 25 mm diameter and a 50 mm height were tested. For determination of compressive strength, the axial load was applied with a strain rate of 1.5%/min, as per American Society for Testing and Materials Standard (ASTM) ASTM D2166/D2166M36. A 100 kN Instron servo-hydraulic universal testing machine was used. The applied load for each increment of strain was recorded using an automatic data acquisition system. The compressive strength of the specimen was computed by dividing the maximum load attained during the test by the cross-sectional area of the specimen.

2.3.2 Splitting tensile test

To determine the tensile properties of the rock samples, splitting tensile tests were performed following general procedures described in the ASTM D3967-1637. The specimens for the splitting tensile strength test had a diameter and height of 25 mm and 12.5 mm, respectively. The same 100 kN Instron servo-hydraulic universal testing machine was used. The load was applied at the constant strain rate of 1.5%/min. It must be noted that a splitting test does not give direct rock tensile properties under tensile loading. Thus, the tensile strength needs to be back-calculated from the maximum compressive load and the dimension of the specimens using the following formula:

$$\sigma_T = 2P/\pi LD \quad (1)$$

where σ_T is tensile strength; P is a compressive force at failure; D is the diameter of the specimen; L is specimen thickness.

2.4 Digital image correlation

Digital Image Correlation (DIC) is a precise, non-contact, full-field image analysis method, based on grey value digital images that can determine the contour and the displacements of an object under external load. The splitting tensile test was combined with the DIC to accurately observe the onset of strain localization and propagation based on the measurements of the surface displacement field.

Prior subjecting specimens to the external load, a unique pattern of random speckles was applied to the specimen surface. The speckle pattern was artificially made by spraying white paint on the natural specimen surface. The speckle pattern enables the observation of the changes in the position of the speckles during the deformation period.

For measuring the deformation, a high-speed video camera (The Shimadzu Hyper Vision HPV) with up to one million frames per second, and 312x260 pixel resolution was used to acquire digital images. In addition, to ensure the optimal image processing, it is

necessary to have a uniform and constant light source in time. Thus, the REL SURE-bright lighting system was used. The images were processed using a commercially available software VIC – Correlated Solutions. The localized deformation zone was identified from the detailed measurements of the displacements. The reader is referred to the VIC – Correlated Solutions manual [13] for further details about the strain computations.

3 Results

3.1 Scanning electron microscope imaging

SEM BSE and EDS images and the final processed images are shown for samples from the two formations Figures 1-3. In the processed images (Fig. 1a and 2a), each color corresponds to a different mineral phase. The corresponding volume fractions determined from the images are given in Table 1. Formation 1 is predominantly quartz (80.3 a%), with 6.3 a% a variety of minor phases, and 13.4 a% pore space. Formation 2 is predominantly dolomite (78.1 a%) with minor anhydrite (4.0 a%), with 17.3 a% pore space, and 0.6 a% various minor phases.

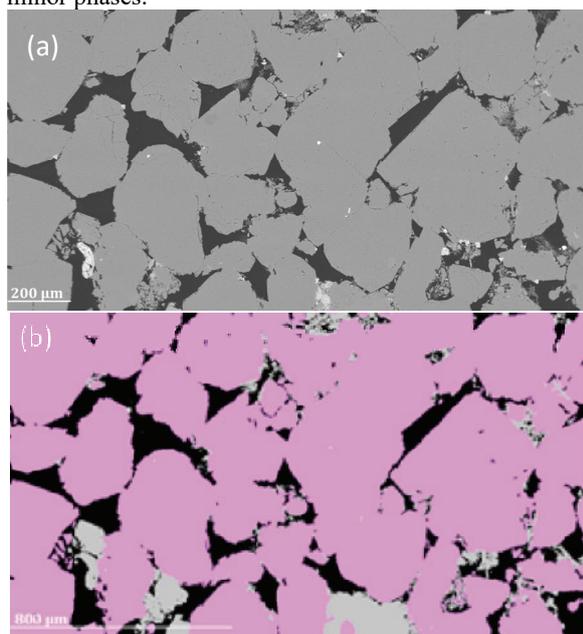


Fig. 1. Unaltered BSE image (a) of Formation 1, and final processed image (b), where pink is quartz, black is pore space, and grey represents various minor phases.

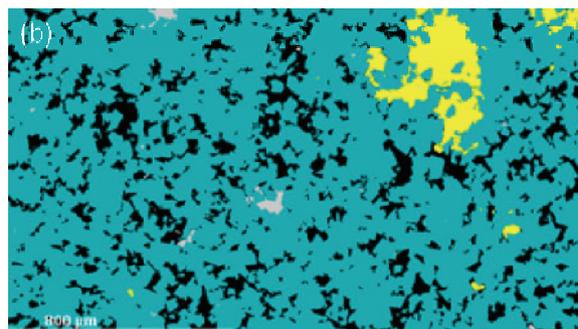
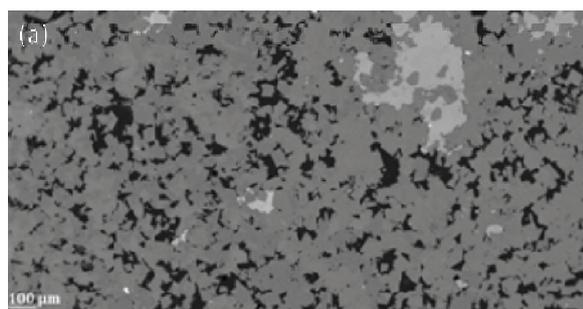


Fig. 2. Unaltered BSE image (a) of Formation 2, and final processed image (b), where blue represents dolomite, yellow represents anhydrite, black represents pore space, and grey represents various minor phases.

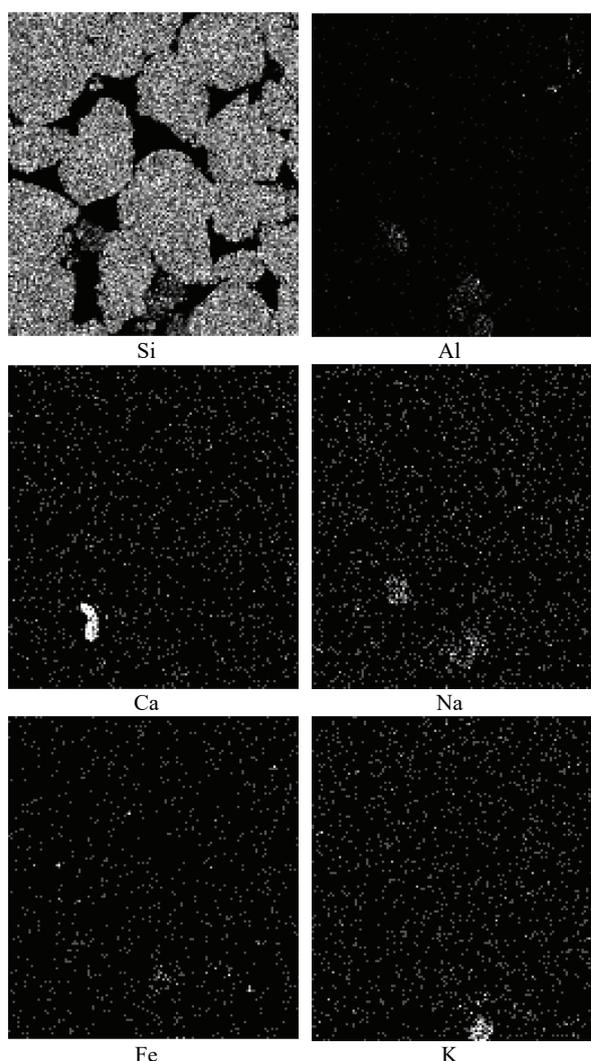


Fig. 3. Example EDS elemental maps from Formation 1 used to determine mineralogy.

Table 1. Different mineral and pore distribution obtained from image processing.

Formation	Abundance (a%)				
	Quartz	Dolomite	Anhydrite	Pore	Other
1	80.3	-	-	13.4	6.3
2	-	78.1	4	17.3	0.6

3.2 Mechanical testing

3.2.1 Unconfined Compression Test

Rock mechanics properties are affected by many external and internal factors. In addition to stress and natural environments, the internal factors such as mineral composition, microstructure, and porosity have a significant effect on it. Figure 4 shows the average uniaxial compressive stress-strain responses of rock cores from two different formations. The peak strength and its corresponding strain depend on the mineral composition and pore size distribution. The average compressive strength was reported as 60 MPa for Formation 1 sample that had a higher abundance of quartz and lower porosity. The average peak compressive strength of the Formation 2 samples was around 25 MPa. The increase in the porosity and the presence of dolomite caused a reduction in strength.

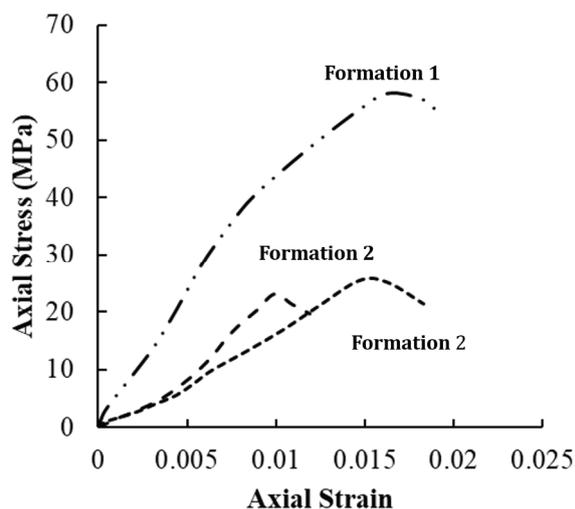


Fig. 4. Effect of mineralogy on the uniaxial compressive behavior of rock.

3.2.2 Splitting Tensile Test

Similar behavior was observed in the tensile stress-strain results. Figure 5 depicts the average tensile stress-strain responses of rock cores from two different formations. The average peak tensile strength of the Formation 2 is

approximately 40% lower than the Formation 1 tensile strength. This finding further confirms that the mineral composition and pore size distribution play a significant role in the mechanical properties of the porous geologic media. As the porosity decreases and the presence of quartz increases, the tensile strength increases.

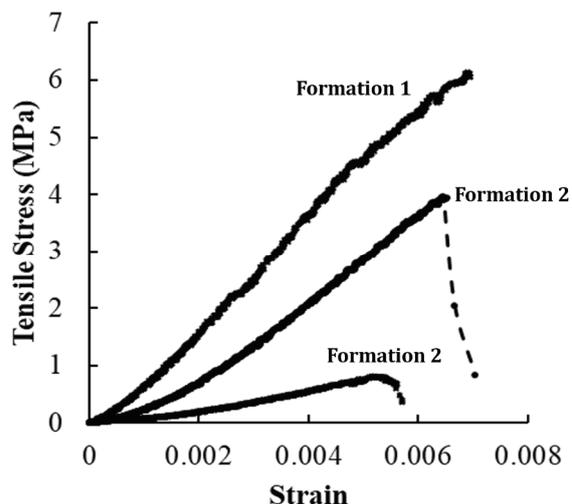


Fig. 5. Effect of mineralogy on the tensile behavior of porous geologic media.

3.3 Digital image correlation analysis

Figure 6 depicts the horizontal strain fields (i.e., strain component perpendicular to the loading direction, which is vertical in the figure) of indirect tensile tests on Formation 1 specimen. The strain initiation is obtained with DIC analysis before the visible crack appears. The strain initiates at the edges and propagates almost through the center of the sample forming a clear fracturing pattern. The horizontal strain field inside fractured surface was noted as 0.57% just before crack formation. A sudden failure or fracture was observed immediately after the initiation of crack.

Horizontal strain fields of indirect tensile tests on Formation 2 specimen can be seen in Figure 7. The strain is initiated at the edge and propagates in more branches, and a spreading pattern for the strain field is visible during the fracture process. The horizontal strain field inside the fractured surface was noted as 0.1% just before crack formation. Lower horizontal strain field inside of the fractured surface in Formation 2 specimen can be contributed to the higher porosity and mineralogy (i.e., no quartz) of the Formation 2. In addition, similarly, as in Formation 1 specimen, a sudden failure or fracture was observed immediately after the initiation of crack.

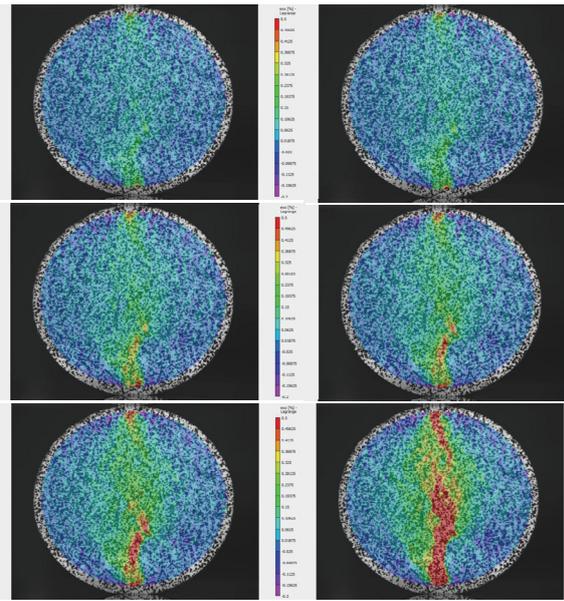


Fig. 6. Horizontal strain fields of indirect tensile tests on Formation 1 specimens.

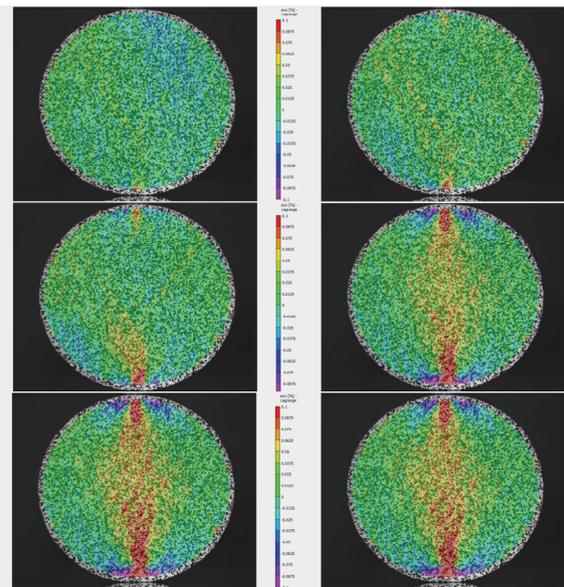


Fig. 7. Horizontal strain fields of indirect tensile tests on Formation 2 specimens.

4 Conclusions

The main aim of this research was to study the effect of mineral composition, microstructure, and porosity on the rock mechanical properties. Two different mineral compositions of the rock were analyzed by using SEM-EDS images, while the microstructure and porosity were obtained by using SEM BSE images. Finally, the uniaxial compression and splitting tensile tests, in combination with the digital image correlation, were carried out to investigate the mechanical characteristics. The uniaxial compressive strength, tensile strength, and

fracture properties were obtained from these experiments.

The results of this study showed that the mineralogy, microstructure, porosity, and mechanical properties of the rock are closely related. In particular, quartz is an essential factor affecting rock mechanics properties. Formation with the closely 80a% of quartz had approximately three times higher compressive and tensile strengths than the formation made out of the 80a% of dolomite. Moreover, porosity is related to the rock mechanics properties. It was found that with the decrease of porosity, the compressive and tensile strengths increased. These findings may have significant practical consequences for many applications, such as geological sequestration of CO₂, mining, rock slope stability, nuclear waste disposal, and many more.

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References

- [1] S. J. Baines, R. H. Worden, Geological Storage of Carbon Dioxide. Geological Society, **233** (1) (2004).
- [2] S. Bachu, Sequestration of CO₂ in geological media: criteria and approach for site selection in response to climate change. *Eng. Convers. Manage.*, 41, 953-970, (2000).
- [3] A. Settari, R. B. Sullivan, The Modeling of the Effect of Water Blockage and Geomechanics in Waterfracs. SPE Annual Technical Conference and Exhibition, (2002).
- [4] M. Sharma, S. Agrawal, Impact of Liquid Loading in Hydraulic Fractures on Well Productivity. SPE Hydraulic Fracturing Technology Conference, (2013).
- [5] F. Cherblanc, J. Berthouneau, P. Bromblet, V. Huon, Influence of Water Content on the Mechanical Behaviour of Limestone: Role of the Clay Minerals Content. *Rock Mechanics and Rock Engineering*, 49 (6), (2016).
- [6] I. O. Ojala, The effect of CO₂ on the mechanical properties of reservoir and cap Rock. *Energy Procedia*, 4, 5392-5397 (2011).
- [7] B. Lamy-Chappuis, D. Angus, QJ Fisher, BWD Yardley. The effect of CO₂-enriched brine injection on the mechanical properties of calcite-bearing sandstone. *Int. J. Greenh. Gas Con.*, 52, 84-95, 2016.
- [8] J. Rohmer, A. Pluymakers, F. Renard, Mechano-chemical interactions in sedimentary rocks in the context of CO₂ storage: Weak acid, weak effects? *Earth-Science Reviews*, 157, 86/110, (2016).
- [9] AT Akono, JL Druhan, G Davila, T Tsotsis, K Jessen, S Fuchs, D Crandall, Z Shi, L Dalton, MK Tkach, AL Goodman, S Frailey, CJ Werth. A review of geochemical-mechanical impacts in geological carbon storage reservoirs. *Greenhouse Gas Sci. Technol.* 9, 474-504, 2019.

- [10] J.E. Lindqvist, U. Åkesson, K. Malaga, Microstructure and functional properties of rock materials. *Mater Charact*, 58 (11–12) (2007).
- [11] E. Johansson, Technological properties of rock aggregates. Luleå University of Technology, Luleå (2011).
- [12] A. Tugrul, I.H. Zarif, Correlation of mineralogical and textural characteristics with engineering properties of selected granitic rocks from Turkey. *Eng Geol*, 51 (4) (1999).
- [13] VIC – Correlated Solutions manual - <http://www.correlatedsolutions.com/installs/Vic-2D-2009-Manual.pdf> (2009)