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Shale/claystone response to air and liquid exposure, and implications for handling, sampling and testing

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ABSTRACT

This paper documents the results of scientific studies to quantify the impact on shales and claystones of exposure to air, and to different liquids. These studies show that the water content of such rocks is controlled by the relative humidity of the surrounding air. Most shales/claystones will lose water, shrink and desaturate if exposed to air, unless the relative humidity is sufficiently high (usually 80–96%), causing desiccation damage. While exposure to brine will prevent desiccation, it results in water uptake and swelling, and a different type of alteration and damage. Confining stress will limit this swelling. Well-preserved shales are generally fully saturated under zero-stress conditions, but they have a negative pore water pressure. Hydrocarbon will not normally enter a fully saturated shale. Based on these findings, it is recommended that shale water content should not be altered unless it is done so under controlled conditions; to accomplish this one must minimize exposure to air, and avoid contact with water or brine unless the shale is under stress. Specific guidelines are provided for core handling and preservation, sampling, and testing. Gas shales are both similar to, and different than, claystone shales, and this alters slightly the findings and guidelines.

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1. Introduction

Mechanical, coupled poro-mechanical and coupled chemo-mechanical behaviors of shales and claystones are important to the fields of mining, civil engineering, tunneling, oil/gas drilling and production, nuclear waste storage, and injection of CO_2 or other liquids/gasses for storage or sequestration. Shales and claystones are commonly tested in the laboratory to determine these behaviors. Often not much thought is put into the handling history of such samples, and whether or not the samples are representative of the in-situ material. The history of a shale/claystone core or lab sample may have involved exposure to air, or exposure to a liquid such as water, brine or hydrocarbon.

This paper documents the results of scientific studies to quantify the impact on shales and claystones of exposure to air, and to different liquids. The summary conclusion of these studies is that shales/claystones are altered unless they are contacted only by hydrocarbon, or by air of controlled relative humidity. The first part of this paper explains not only the findings but the underlying physical mechanisms. Based on these findings, the second part of the paper provides guidelines for shale handling, sampling and testing. The main focus of the guidelines is to maintain shale native water content, to avoid damage due to drying, to avoid

damage due to uncontrolled swelling or other chemical effects, and to avoid testing artefacts associated with pore water suction and other capillary pressure or osmotic pressure effects.

Clay-supported rocks will be commonly referred to as 'shales' in this paper, even though many of them are more properly defined as claystones (a clay-dominated mudstone lacking fissility). Gas shales, or 'resource shales', are different than both claystones and most of the rocks traditionally referred to as shales, due to different mineralogy and other characteristics. Gas shales are addressed in the final section of the paper.

2. Technical background – key concepts

2.1. Shales used for air and brine exposure tests

Air and brine exposure tests were performed on nine different preserved shales. These shales were obtained from different locations around the world, each one from a section of preserved oilfield core. The shales cover a broad range of depths (and therefore compaction state).

Table 1 lists the bulk density, porosity, native activity and cation exchange capacity (CEC) of each shale. Bulk density and porosity were determined from multiple samples of each shale which had precise dimensions and were fully-preserved and fully-saturated. Porosity is based on the weight loss at 105–110 °C, assuming 1 g/cc

Table 1
Key petrophysical properties of each shale.

Shale	Saturated bulk density (g/cc)	Porosity OD[a] (%)	Native activity	CEC (meq/100 g)
A	2.17	29.6	0.96	33
B	2.19	28.6	0.96	25
C	2.21	27.0	0.96	21
D	2.32	22.2	0.92	25
E	2.34	21.9	0.88	31
F	2.38	21.3	0.91	24
G	2.42	18.4	0.80	32
H	2.54	13.0	0.70	35
I	2.51	11.5	0.86	23

[a] Porosity from weight loss upon oven drying (OD) at 105–110 °C.

for the evaporated water. Native activity is explained in a later section. Cation exchange capacity was determined using the Co(III)-hexamine^{3+} technique of Bardon et al.[1]

Each of the nine shales contains 65–76 wt% of clay minerals, as determined by a quantitative X-ray diffraction technique.[2] The remainder is dominated by quartz silt, with small amounts of feldspar silt and occasional minor amounts of siderite. Clay grains are the load-bearing matrix in these rocks, with silt grains 'floating' in the porous clay and generally not touching each other. Shales A, E and H contain only small amounts of kaolinite and are dominated by mixed-layer illite–smectite clay, while the remainder of the shales contain significant kaolinite as well as significant amounts of illite–smectite.

All samples were cut from downhole shale core that had been cored with oil-base or synthetic-base drilling fluid and kept fully preserved. Exposure to air was minimized, and only decane was brought in contact with the shales during sample preparation. After each sample was cut, it was either wrapped in cellophane and stored in a small sealed vial, or was stored in a sealed jar of decane. These steps were taken to prevent changes in shale water content.

2.2. Shales exposed to air

2.2.1. Effects of air exposure and changing relative humidity

This section documents data obtained on samples that were placed in vacuum desiccators of constant relative humidity. In a vacuum desiccator, water is added to or removed from the shale through vapor-phase movement.[3] This vapor-phase movement of water continues until the shale sample reaches equilibrium with the controlled relative humidity environment. This equilibrium condition determines the shale water content (g water/g dry shale). Crossplots of water content change vs. relative humidity are shown in Fig. 1 for the nine different shales. Each datum point is a separate sample, which was equilibrated to that particular relative humidity value. Dry weight is then obtained by drying the shale for about two weeks at a temperature of 105–110 °C.

Because each data point represents a separate sample, there is expected to be some scatter in the data. In spite of this scatter, however, it is seen that increasing relative humidity results in higher water content. It is also apparent that the higher porosity shales (see Table 1) undergo much larger changes in water content than the lower-porosity shales, for a given change in relative humidity. The relationship between water content and relative humidity for any particular shale represents its adsorption isotherm.[4] In the field of geotechnical engineering and unsaturated geomechanics, it is referred to as the water retention behavior, and suction pressure is used in place of relative humidity (e.g. 5–7).

For each sample we also have precise measurements of its length and diameter, which can be converted to volume. Fig. 2
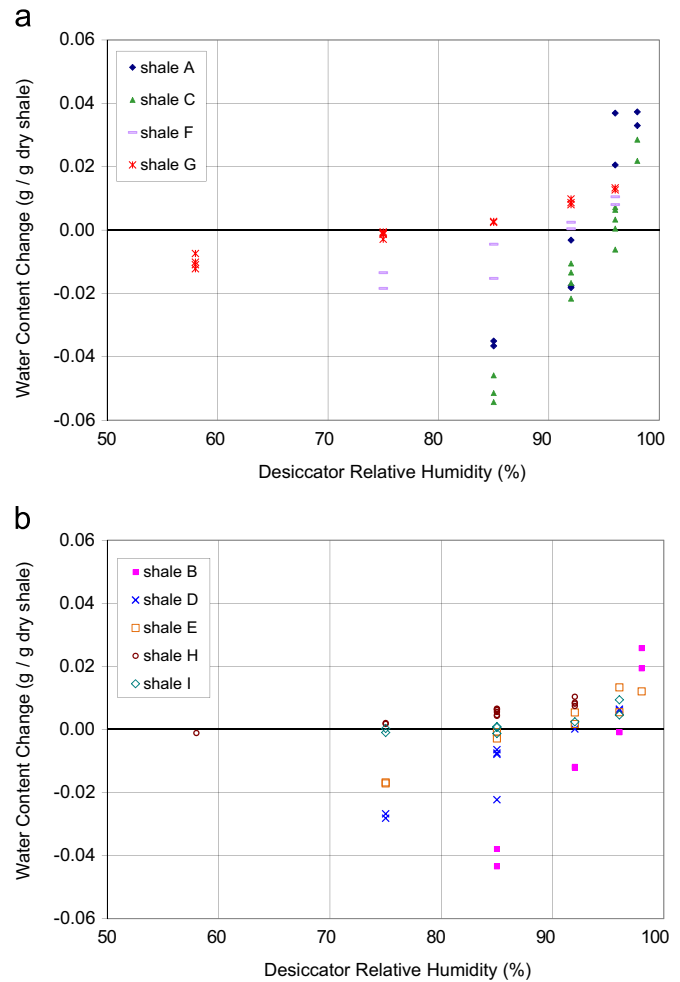

Fig. 1. Water content gain or loss vs. relative humidity; each data point is a separate sample. (a) shales A, C, F and G; (b) shales B, D, E, H and I.
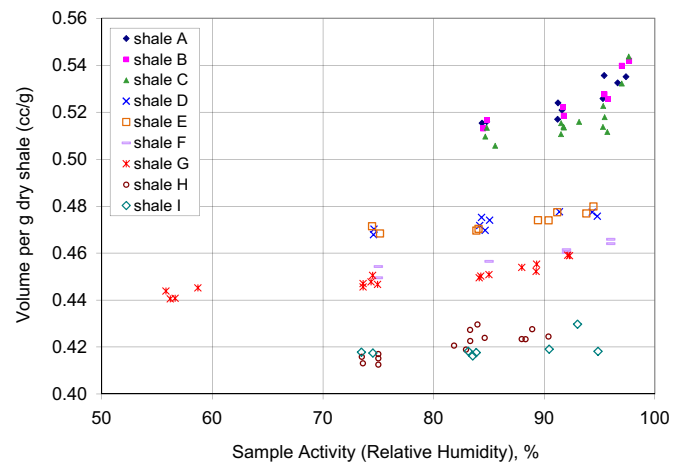

Fig. 2. Sample volume, normalized to dry weight, vs. sample activity (relative humidity).

shows a crossplot of sample volume (per gram of dry shale) against the sample activity, or relative humidity, at the time of volume measurement. It is seen in Fig. 2 that a higher relative humidity corresponds to a larger sample volume. This means that the samples expand as they gain water (higher relative humidity) and shrink as they lose water (lower relative humidity). Note that volume per gram dry shale is directly related to the shale porosity,