



## Laboratory Method to Evaluate Fracture Development in Hard Shale Formations Exposed to Drilling Fluids

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### Abstract

A systematic laboratory technique has been developed to evaluate the stability of hard shale formations when exposed to drilling fluids. Wellbore stability problems found in these types of formations have created difficult and costly drilling operations in many areas (e.g. Middle East-Nahr Umr formation, USA-Atoka, and South America-Los Monos).

To date, testing drilling fluids for inhibition generally has focused on the chemical effects of fluids in soft/reactive shale formations. Dispersion and swelling tests have been used to evaluate hydration of clays, dispersion, swelling, high ion exchange capacity, etc. These traditional techniques, however, do not integrate important elements intrinsic to the failure mechanics of hard shales. Rock structure, fabric, texture, and mineral distribution establish conditions that can directly affect fracture development in hard shale formations. A recently developed laboratory method focuses on the analysis of fracture development using shale samples that preserve the natural structure of the rock. The technique combines photographic documentation of visible changes in the shale during fluid exposure and examination of petrographic sections of the shale samples after fluid exposure. The method allows observation, description, and comparison of the effects of different drilling fluids on the shale structure.

This paper presents a detailed description of how this new method is used to evaluate fracture development in hard shale formations. Analyses of a series of laboratory experiments are presented along with qualitative and quantitative data to show how these kinds of formations respond differently to different fluid environments.

### Introduction

The term "shale" is commonly used in the drilling industry to describe a broad group of rocks from highly reactive formations to completely lithified materials. It is important to recognize that shales behave quite differently when they are exposed to fluids.<sup>1</sup> The mechanisms of instability in shale formations vary greatly with the change in mineralogy and structure of

the rock. Identifying the type of shale formation and its characteristics is the a basic step in designing and selecting drilling fluids for shale stability applications. Numerous laboratory techniques are employed for rock/fluid interaction studies, but the less time-intensive tests which involve relatively simple methodologies seem to be the most practical and useful in the industry.

The selection of laboratory experiments and their conditions depend on the basic rock properties. Generally, soft shale formations soften, disperse, and swell rapidly when exposed to water-based fluids. Hard shales have gone through diagenetic process (alteration of clay minerals, secondary cementation, etc.) and tend to remain stable in fluids.<sup>1</sup> Soft shale formations tend to interact readily with fluids. In this case, tests such as dispersion, bulk hardness and swelling are very useful and the results usually show clear differences among fluids.<sup>2</sup> Hard shales interact differently with fluids; their mechanisms of instability are more associated with fracture development and slippage along beddings. This type of failure seems to be caused by fluid penetration along pre-existing fractures and laminated surfaces. The different shale responses to different chemical fluids indicate that the failure mechanisms involve both physical and chemical processes.

Instability mechanisms of hard shales are closely related to the geologic characteristics and properties of the rock such as structure, grain distribution, texture, and presence of fractures. The samples to be tested for fracture development must include these characteristics. Generally, shale cuttings are routinely available and these samples can be used to obtain important information in shale stability studies such as mineralogical composition, cation exchange capacity (CEC), scanning electron microscope (SEM) images, etc.

Shale cuttings are useful in fluid evaluations and, in some cases, also pulverized and reconstituted into pellets for other testing.<sup>3</sup> However the network of fractures or cleavage planes, mineral geometric distribution, texture (laminations, lenses, etc.), and fabric of the shale are partially lost when the shale is collected

as cuttings or reconstituted into pellets.

The method presented in this paper establishes a procedure that uses shale pieces to evaluate the development of fractures in hard shale formations exposed to base fluid and other drilling fluid additives while maintaining the original structure of the rock. This procedure tracks visible changes in the shale pieces during the fluid exposure and recognizes the changes in the microstructure of the rock. In this way, geologic properties and the fluid effects are included and evaluated directly.

## Method

The method of shale study combines two techniques: Time-Lapse Photography (TLP) and Microscopic Analysis of thin sections.

1. The TLP technique is used to record photographically the visible changes in the rock during the fluid exposure over a period of time (usually 24 hours).

2. The microscopic analysis technique is used to observe changes in the microstructure of the rock after fluid exposure through standard petrographic thin sections.

## Materials

### *Shale samples*

Core samples or pieces of shale formation are used for testing. The rock samples are cut into similar sized pieces for testing.

The Los Monos shale sample consisted of irregular shaped and sized pieces. The largest pieces were selected for testing. Pieces of the original sample are shown in **Figure 1**. The mineral composition and CEC of the Los Monos shale are in **Table 1**. Thin-section observations (**Figure 2**) of the sample show that the rock is composed basically of fine silt- and clay-sized minerals grains. Quartz and clay are the main components; organic material and pyrite are present. Moderate orientation of clay minerals was observed. Siltstone lenses and layers are distributed mostly parallel to the bedding planes; most of the fractures were extended along the clay-rich areas. Some pre-existing fractures were observed.

Nahr Umr sample was taken from a formation core in Qatar. **Table 1** shows the mineral composition and CEC of the Nahr Umr shale sample. The thin section (**Figure 3**) shows that the shale sample is composed mainly by clay-size mineral grains. Clay and quartz are the main components with pyrite and insoluble organic material present. The rock shows good lamination and pre-existing fractures extending along the bedding plane.

Other shale formations reported in this paper are mentioned in the results and discussion section to illustrate and explain important findings.

### *Fluids*

Clear/solids-free fluids are used to allow clear

visualization of the samples. Fluids containing base fluids and inhibitor products are prepared for testing. Freshwater, seawater, brines, mineral oil, diesel and synthetic fluids are used as base fluids. The inhibitor products include polyglycols, amine compounds, potassium silicate, etc.

## Procedure

The procedure for handling a shale sample is as follows:

### *Pre-Test Procedure*

1. Identify the basic petrologic features of the samples such as angle and spacing of lamination, direction of crossbedding, presence of pre-existing fractures, change in grain, etc.

2. Saw the rock sample into pieces of approximately equal size (cubic shape) using a saw with diamond blade for dry cutting applications. The maximum sample dimension (length) varies depending of the size of the core and the condition of the rock, but in general terms the maximum dimension ranges between 1 and 2 inch.

### *Test Procedure*

1. Place each piece of rock in an optically clear square container. Keep one piece of sample for comparison purposes.

2. Add the fluids into the containers. Ambient conditions (temperature and pressure) are maintained throughout the test.

3. Take photos during the established time (generally 24 hours). Carefully observe and record photographically the changes in the rock integrity such as fracture development, cracking, crumbling, precipitation of material on the surface of the sample or any visible change.

4. At the end of the test, remove the samples from the containers and dry them at room temperature overnight.

### *Post Test Procedure*

1. Prepare thin sections of the samples (including the unexposed sample). This procedure includes impregnation of the samples with blue dye epoxy, cutting and grinding process in oil.

2. Carefully examine the thin sections with a petrographic microscope. Record and document the main features of the fracture network for each sample. These features include number of fractures, maximum fracture extension, maximum fracture width, typical fracture width, presence of intersecting fractures, principal fracture direction, and any significant characteristics in the rock structure.

3. Compare the original sample and the sample exposed to identify the changes in the rock structure occurred after exposure to fluids.

The analyses of all the information allow the classification of the fluid performance and selection of

the base fluid and other drilling fluid additives which cause the least change in the shale.

## Results and Discussion

The principal results of the test conducted with the Nahr Umr shale are presented. The results of the test conducted with Los Monos shale are presented in more detail to show more clearly how the Time-Lapse Photography and Microscopic Analysis techniques are used and combined to evaluate fracture development in hard shales and how these results are related with the performance of the fluids. Other tests conducted with shale samples from different sources are referenced to explain important findings.

### Observations During Fluid Exposure

Recording photographically the shale samples in the fluids permits visual comparison of how the different chemical compositions of fluids change the samples. The principal changes observed and tracked during the experiments are:

- Development of fractures
- Enlargement of pre-existing fractures or new fractures
- Fracture development direction
- Shale instability behavior (crumbling, spalling, etc.)
- Physical indicators of chemical rock-fluid interaction (material precipitated in the sample)

Close observation of the shale can reveal development of fractures, enlargement of pre-existing fractures, or new fractures. **Figure 4** shows a piece of Nahr Umr shale exposed to seawater containing an amine inhibitor product. The sample exhibited some fractures after 10 minutes of fluid exposure. After 6 hours, the enlargement of the fractures and appearance of new fractures was clearly evident.

The main direction of the fracture development in the shale samples was identified using the photos and thin section analyses. In most of the cases, the fractures tended to extend parallel to the bedding plane, but in some tests cross-bedding fractures were observed. In these cases, the samples became very fragile and easily broke off. Fractures extending in different directions are shown in **Figure 5** where a piece of Nahr Umr shale was exposed to KCl brine with a polyglycol additive. This shale exhibited multiple fractures parallel to the bedding plane and some fractures perpendicular to the bedding plane.

Other shale instability behaviors were documented including spalling and crumbling of fine particles or pieces of shale. **Figure 6** shows a sample from Fortune Bay, Canada exposed to a potassium silicate fluid. In addition to the fracture development, spalling of pieces of rock was observed in this test.

Physical indicators of chemical rock-fluid interaction,

such as material precipitated on the surface of the rock, in the fluid near the rock, or along the fractures, are noted in **Figure 7** which shows the precipitation of white material in a piece of Nahr Umr shale exposed to a potassium silicate fluid.

Other experiments showed a marked difference in the stability of the shale sample using different formate brines (**Figure 8**). The sample from the Mexico Iride field exposed to 10.5-lb/gal potassium formate remained stable in the fluid and few fractures were observed. The piece of shale exposed to 10.5-lb/gal sodium formate brine exhibited more fractures and the presence of intersected fractures was evident. Another sample showed a strong interaction with 16-lb/gal cesium formate brine with multiple fractures and spalling behavior being observed.

Experiments comparing shale samples from different locations with similar CEC values showed that the development of fractures can vary greatly. **Figure 9** shows shale samples from three areas: Nigeria (CEC = 14 meq/100 g), New Zealand (CEC = 15 meq/100 g) and Qatar (CEC = 15 meq/100 g) exposed to freshwater for 24 hours. The fracture development for each sample was completely different. The sample from Nigeria exhibited multiple intersected fractures and the integrity of the rock was greatly affected. The sample from New Zealand remained stable in the fluid and no apparent fractures were observed. The third sample from Qatar (Nahr Umr shale) exhibited several fractures and most of them extended along the bedding plane. This sample became very unstable during the test.

### Thin-Section Analysis (Post Fluid Exposure)

The thin-section analyses of the shale samples reveal important information concerning the fracture network or microstructure of the rock. The comparison of the reference sample (no fluid exposure) to samples exposed to fluids shows changes in the microstructure of the rock caused by the fluid. The principal observations in this analysis are:

- Presence of pre-existing fractures or new fractures
- Main direction of fractures
- Presence of intersecting fractures
- Dimensions and number of fractures (typical fracture width, maximum fracture width, number of fractures, etc.)
- Identification of remarkable changes that describe the fracture network

The original Nahr Umr shale (**Figure 3**) contained micro-fractures extending mainly along the bedding plane. Intersected fractures were not typically found. Micro-fractures of 7-micron width were observed in the section. The maximum fracture width found was ~87 microns.

Examination of the rock sections after fluid exposure

and comparison with the original thin section revealed significant changes had occurred in the structure of the shale. Multiple intersected fractures and broken pieces of rock along the fractures were observed in a thin section of the Nahr Umr shale exposed to a potassium silicate fluid (**Figure 10**). A piece of shale from the same sample exposed to an amine-based fluid developed a complex network of fractures including a big fracture (~500-micron width) and smaller fractures intersecting in different directions (**Figure 11**).

Thin-section observations are very useful where the visual observation and the time-lapse photos do not show significant changes in the rock or when the effects of the fluids are similar. In most of the cases, samples exposed to oil-based fluids remain very stable, but slight changes can be detected using thin sections. **Figure 12** shows a section of the Nahr Umr shale exposed to mineral oil. The fracture network remained almost unaffected by the fluid but the measurement of the maximum fracture widths indicated that some fractures became wider during the fluid exposure. The reference sample contained fractures as wide as ~87 microns while the sample exposed to mineral oil contained some fractures wider than 250 microns.

#### *Los Monos Shale*

In order to more fully illustrate the analytical process, a detailed study of the Los Monos Shale is presented using the basic techniques discussed above - Time-Lapse Photography and Microscopic Analysis

Pieces of Los Monos shale formation were exposed to five fluids: freshwater, potassium silicate base fluid, amine-based fluid (A), amine-based fluid (B), and mineral oil.

The results of dispersion and bulk hardness tests, other type of tests used to measure shale/fluid reaction or inhibition using shale cuttings, are shown in the **Figure 13**. The results showed similar values for the water-based fluids. The recoveries from the dispersion test were over 90% which indicated that the sample had a low dispersive tendency. The bulk hardness test showed similar results for the fluids tested. The graph indicates the hardness of the sample after fluid exposure is similar – well-consolidated pellets indicating the cuttings did not soften in the fluids.

Time-lapse photography of the Los Monos samples indicated different behaviors in the five different fluids tested. After immersing the sample in freshwater, a slight alteration of the sample was observed. Small particles of the rock fell and air bubbles escaped from the sample. After two hours, a gradual development of cracks was evident. After 6 hours, these cracks became wider. Although the cracking occurred, the sample maintained its shape. The sample exposed to the potassium silicate fluid showed good stability during the two first hours; development of cracks was not evident. After two hours, the growth of a material was noted on the surface of the

rock and some cracks were observed. The samples exposed to the amine-based fluid (A) and amine-based fluid (B) had a similar behavior. No evident changes were observed when the samples were immersed in the fluid. Development of fractures was noted after two hours; however the samples maintained their shape. The sample exposed to the mineral oil did not exhibit visual changes during the test. **Figure 14** shows photos of the shale samples before fluid exposure and after 6 hours of fluid exposure.

Thin-section analysis of the Los Monos shale indicated that the reference sample (no fluid exposure) contained original micro-fractures as wide as 9 microns approximately (**Figure 15**). Intersecting fractures were not observed in this sample. Most of the fractures extended along the clay-rich beddings. After exposure to water-based fluids, the thin section exhibited large fractures extending along the bedding plane. Additionally, intersected fractures extending in different directions were observed in some samples. The significance of these large and intersected fractures is that they allow the sample of shale to fall apart. Detailed photomicrographs of intersected fractures can be readily seen in **Figure 16** which shows the shale samples after exposure to water-based fluids.

**Table 2** summarizes some measurements and observations of the thin sections of the Los Monos Shale. The number of fractures recorded suggests that new fractures developed in the samples exposed to the water-based fluids. Based on the number of fractures, there was not a significant difference between the water-based fluids, but measurement of the maximum fracture width indicates that the fracture width varies between drilling fluids. The smaller maximum fracture width suggested that the amine-based drilling fluid (B) exhibited the best performance of those water-based drilling fluids tested. The sample exposed to the mineral oil-based fluid remained stable. Comparing the number of fractures and fracture width with the reference sample, there were no significant changes caused by the mineral oil-based fluid.

#### **Conclusions**

The interactions between hard shale formations and drilling fluids involve mechanisms of rock failure directly related with the structure and composition of the rock. The method presented in this paper can directly track the development of fractures in the shale samples during the fluid exposure and identify the structural changes occurring in the rock. The tests results indicate that the chemical compositions of the fluids have important effects on the stability of the hard shale formations. This technique allows the evaluation of fluid inhibition and the identification of fluids which are most suitable in reducing potential fracture development and slippage along bedding in hard shale formations during drilling operations.

### Acknowledgments

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### References

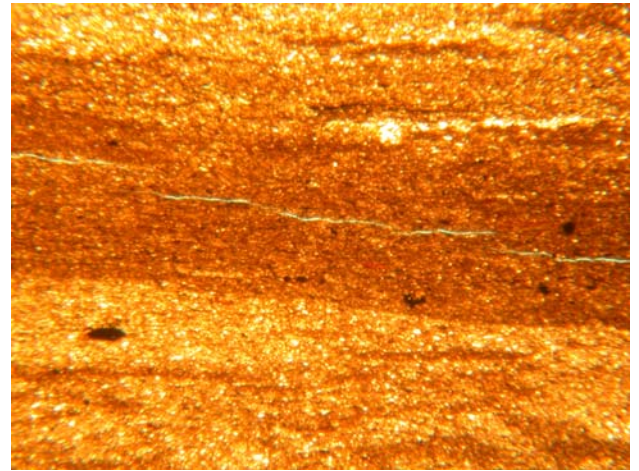
1. Darley, H.C.H.: "A Laboratory Investigation of Borehole Stability," *Journal of Petroleum Technology* (July 1969) 883.
2. Patel, A., Stamatakis, E., Young, S. and Cliffe, S.: "Designing for the Future – A Review of the Design, Development and Testing of a Novel, Inhibitive Water-Based Drilling Fluid," AADE-02-DFWM-HO-33, AADE Drilling Conference, Houston, April 2-3 2002.
3. Lee, John.: "Overview of Shale Stability Testing," *Drilling and Completion Fluids* (May/June 1994) 15.

Mineral Composition	Los Monos Formation	Nahr Umr Formation
Quartz	36%	16%
Feldspar	4%	1%
Calcite	1%	1%
Dolomite	1%	
Siderite	1%	1%
Hematite		2%
Pyrite	1%	
Chlorite	8%	
Kaolinite	15%	50%
Illite	20%	10%
Smectite and Mixed Layer Clays	13%	19%
CEC (meq/100 g)	10	15

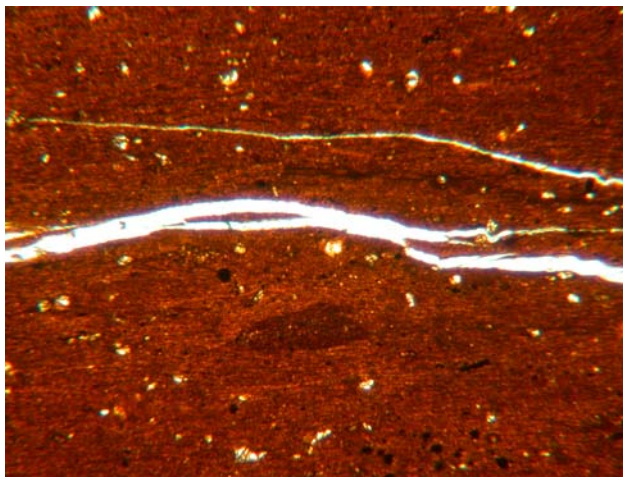
Fluid	Number of Fractures	Intersecting Fractures	Maximum Fracture Width (microns)
No fluid exposure	13	No	22
Freshwater	19	Yes	174
Potassium silicate fluid	18	Yes	312
Amine-based fluid (A)	22	Yes	174
Amine-based fluid (B)	18	Yes	75
Mineral Oil	13	No	15



Figure 1. Original sample of Los Monos shale.



400 microns



200 microns

Figure 3. Thin section of Nahr Umr shale

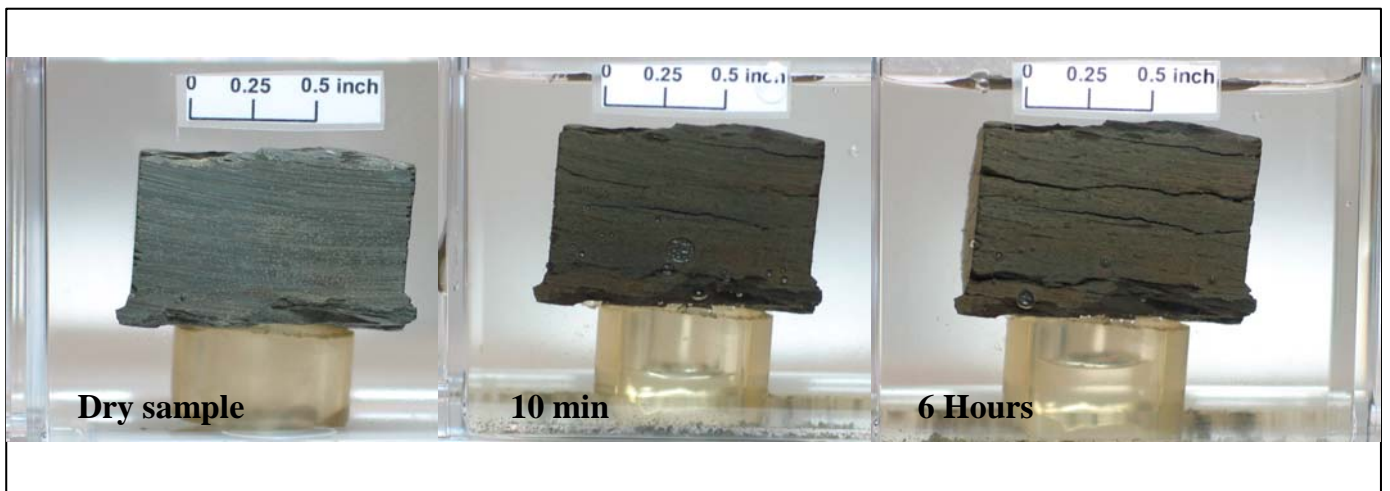


Figure 4. The change of Nahr Umr shale when exposed to seawater with amine inhibitor

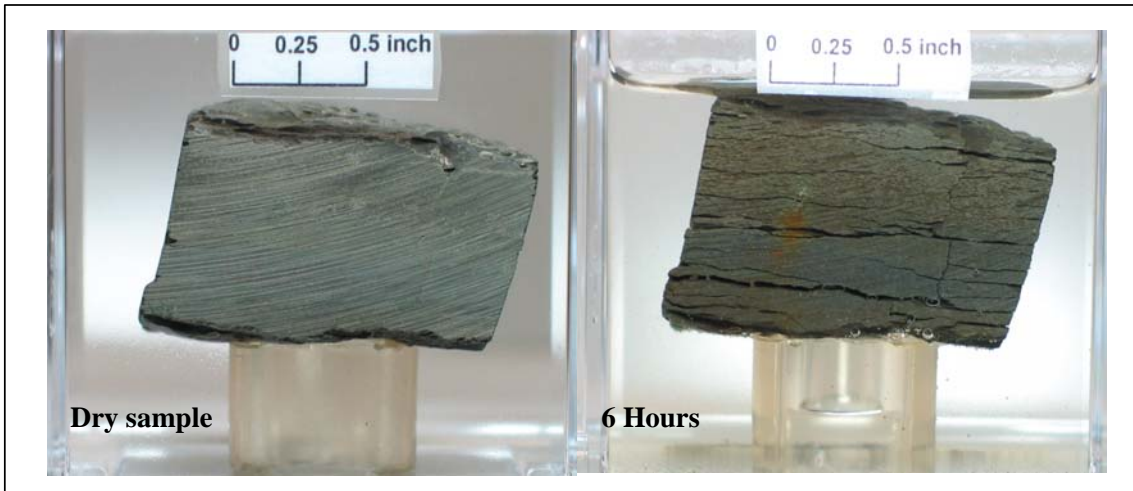


Figure 5. Nahr Umhr shale in KCl brine with a polyglycol additive

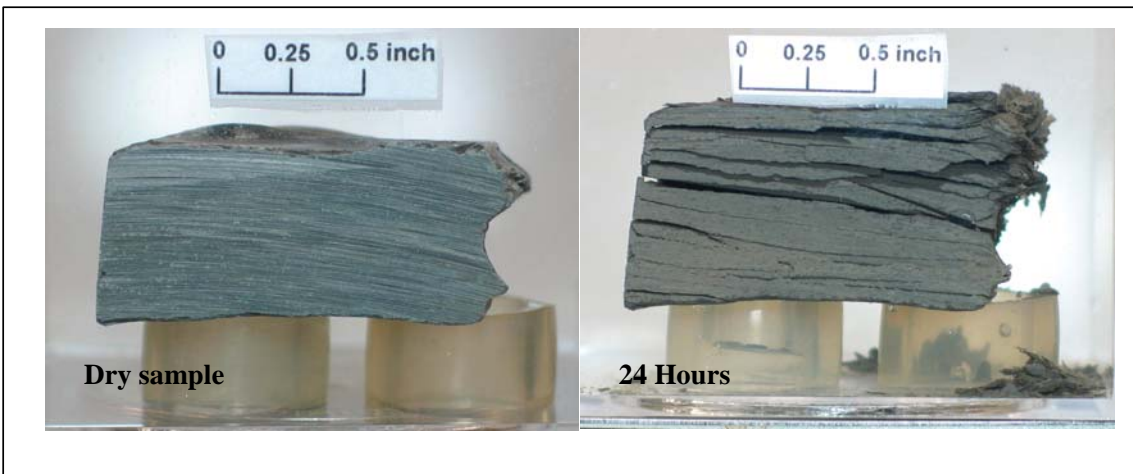


Figure 6. The change of Fortune Bay shale exposed to potassium silicate fluid

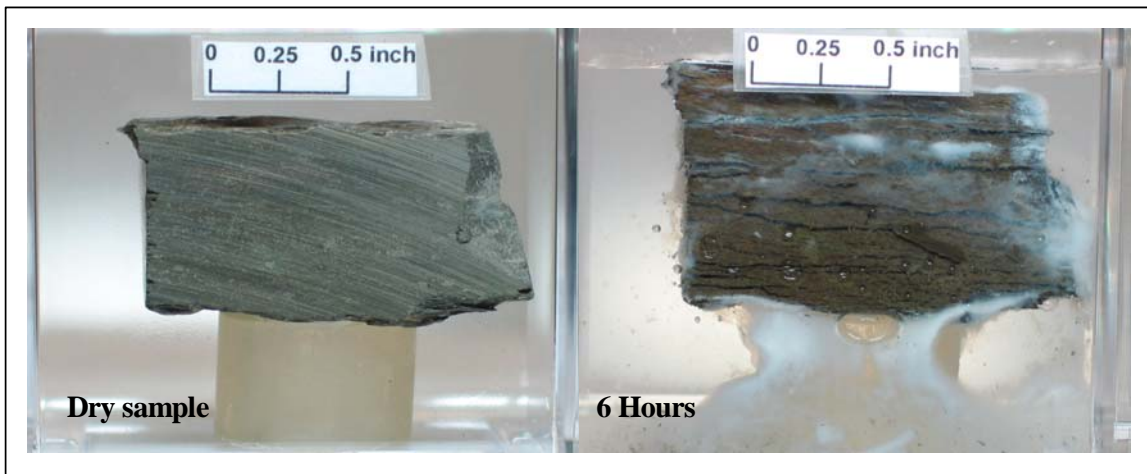


Figure 7. The change of Nahr Umr shale exposed to potassium silicate fluid

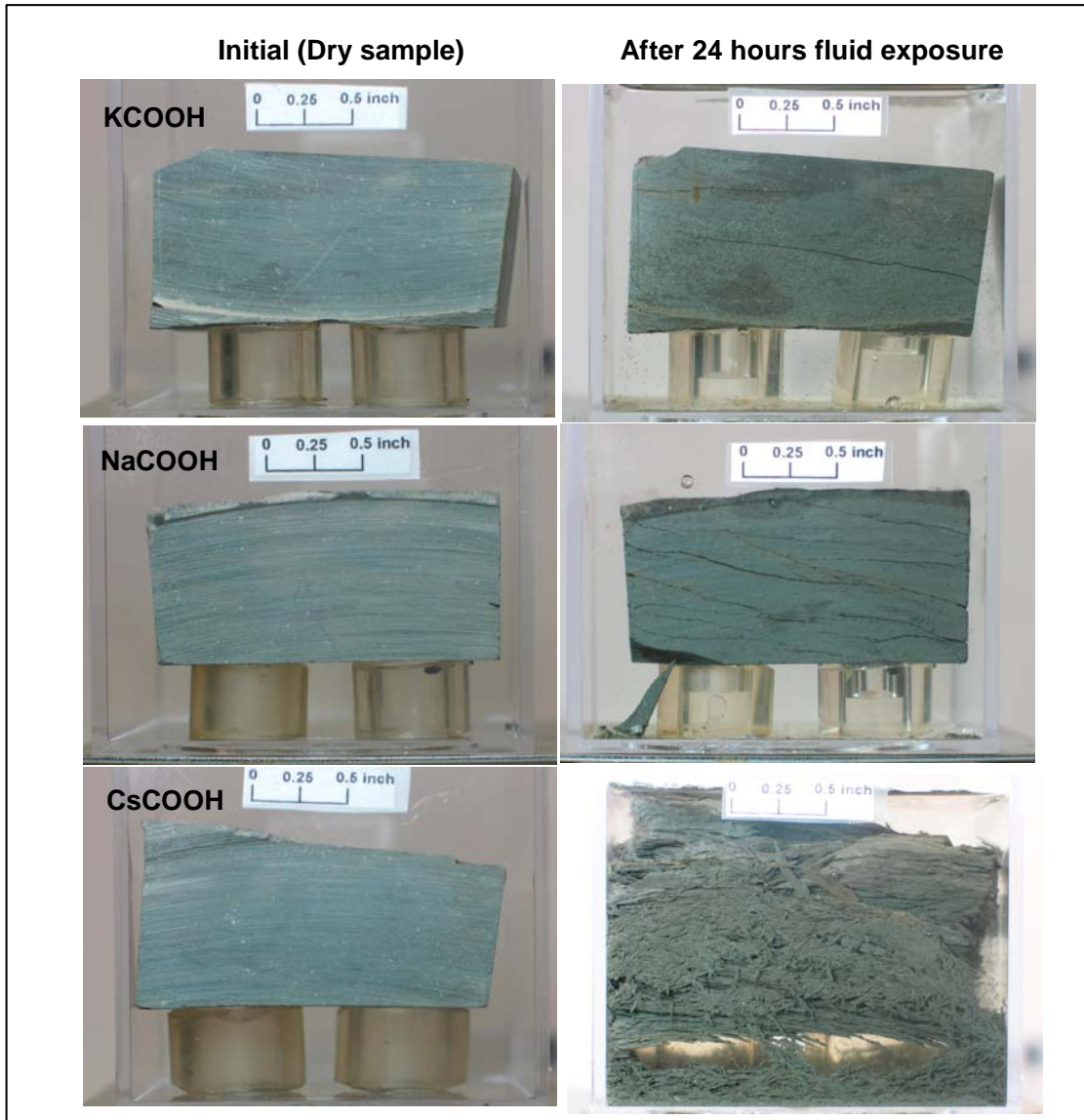


Figure 8. The change of Mexico Iride field shale exposed to three formate brines

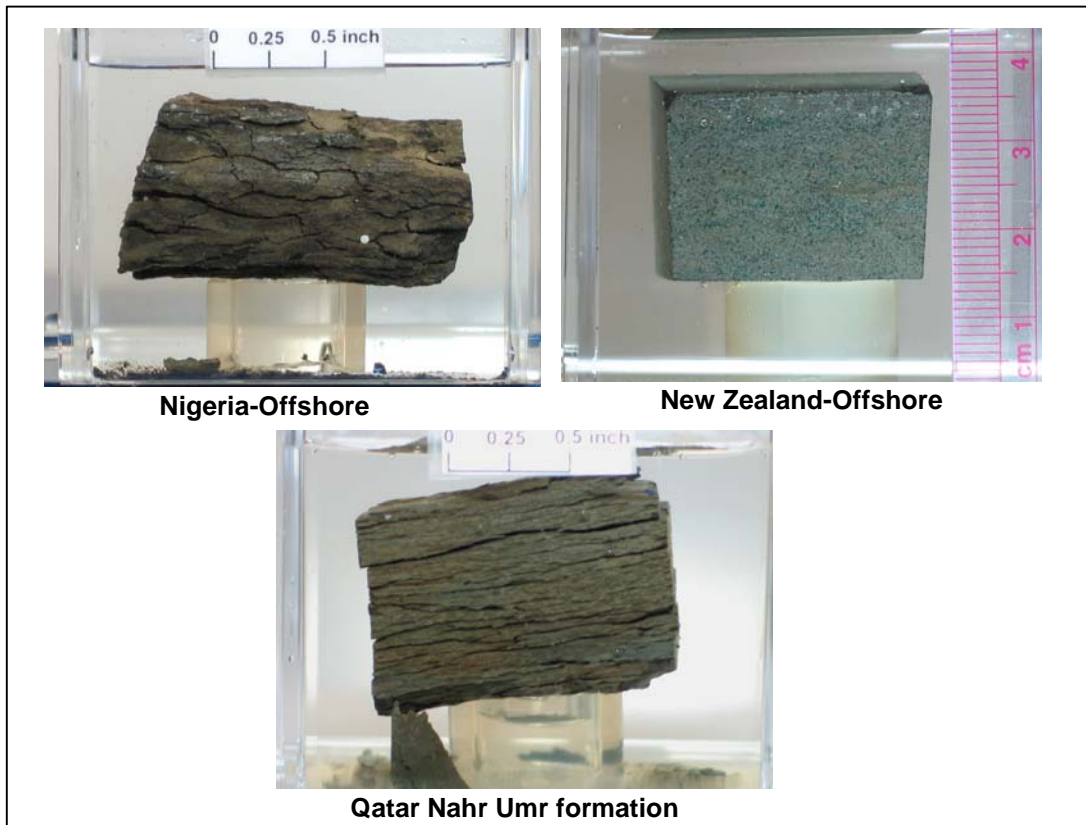
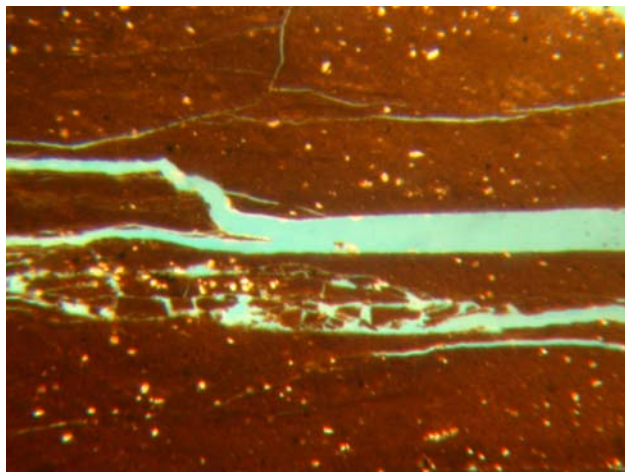
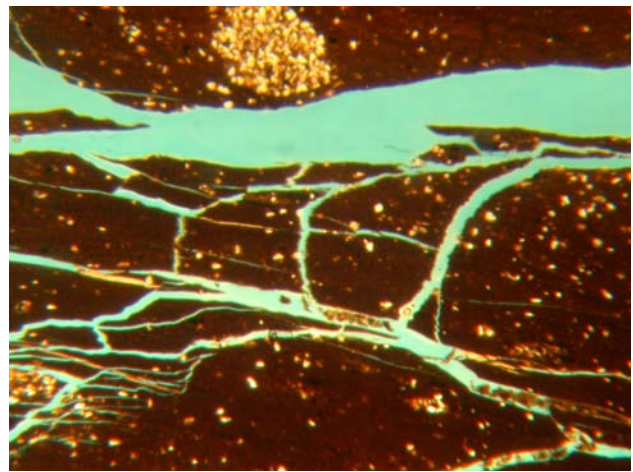


Figure 9. Fracture development in shales when exposed to freshwater after 24 hours



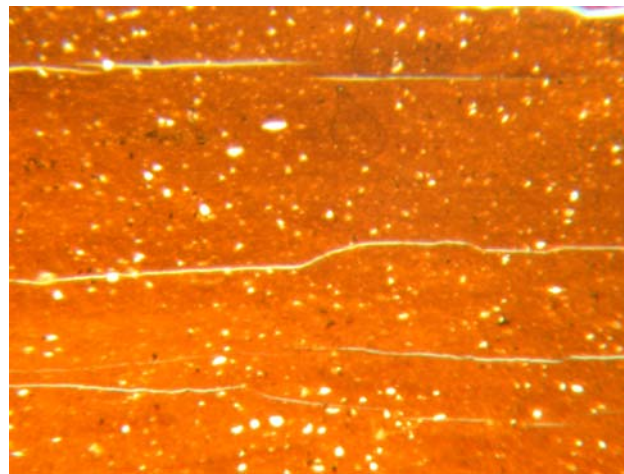
400 microns

Figure 10. Thin section of Nahr Umr shale after exposure to potassium silicate fluid for 6 hours



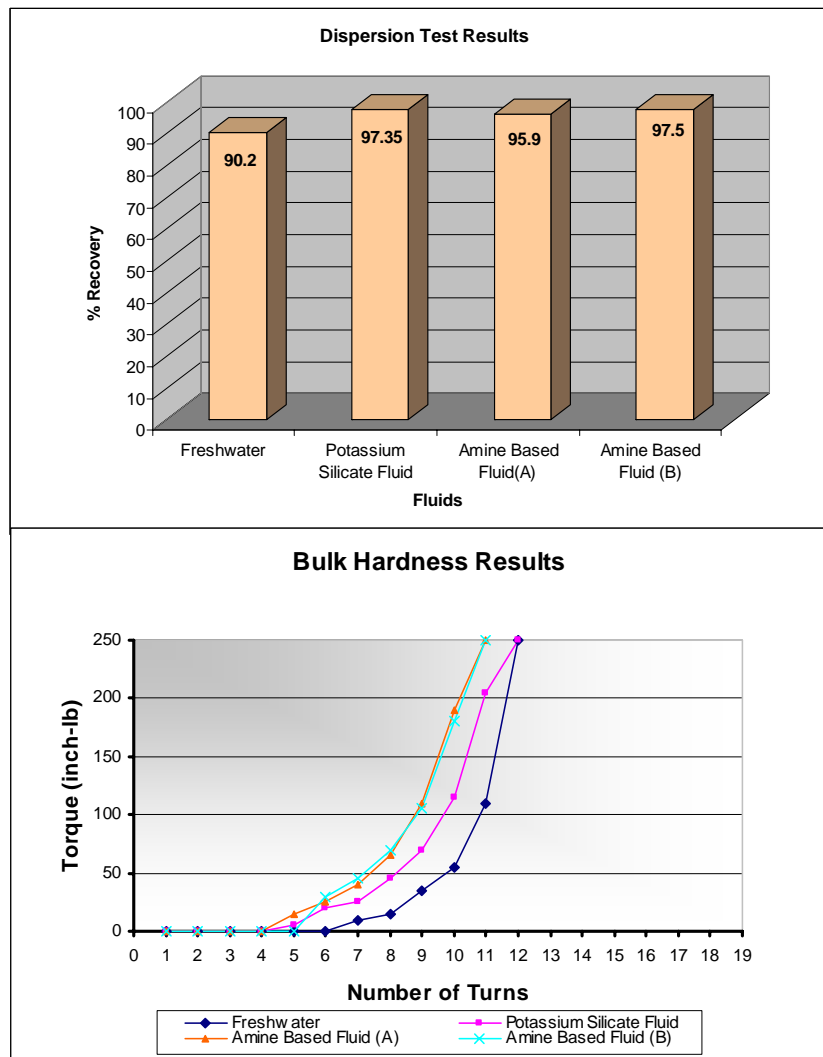
400 microns

Figure 11. Thin section of Nahr Umr shale after exposure to amine-based fluid for 6 hours

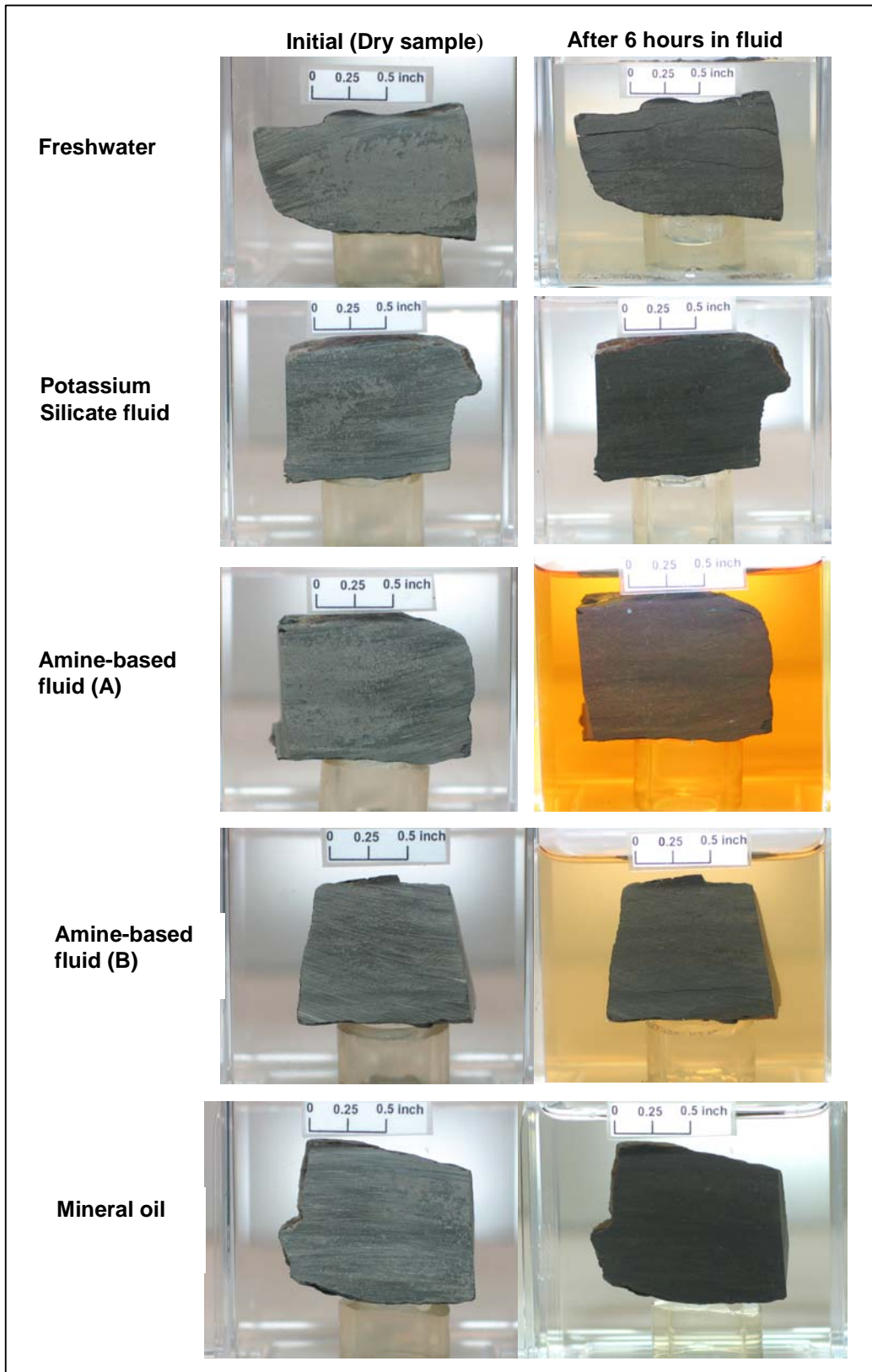


400 microns

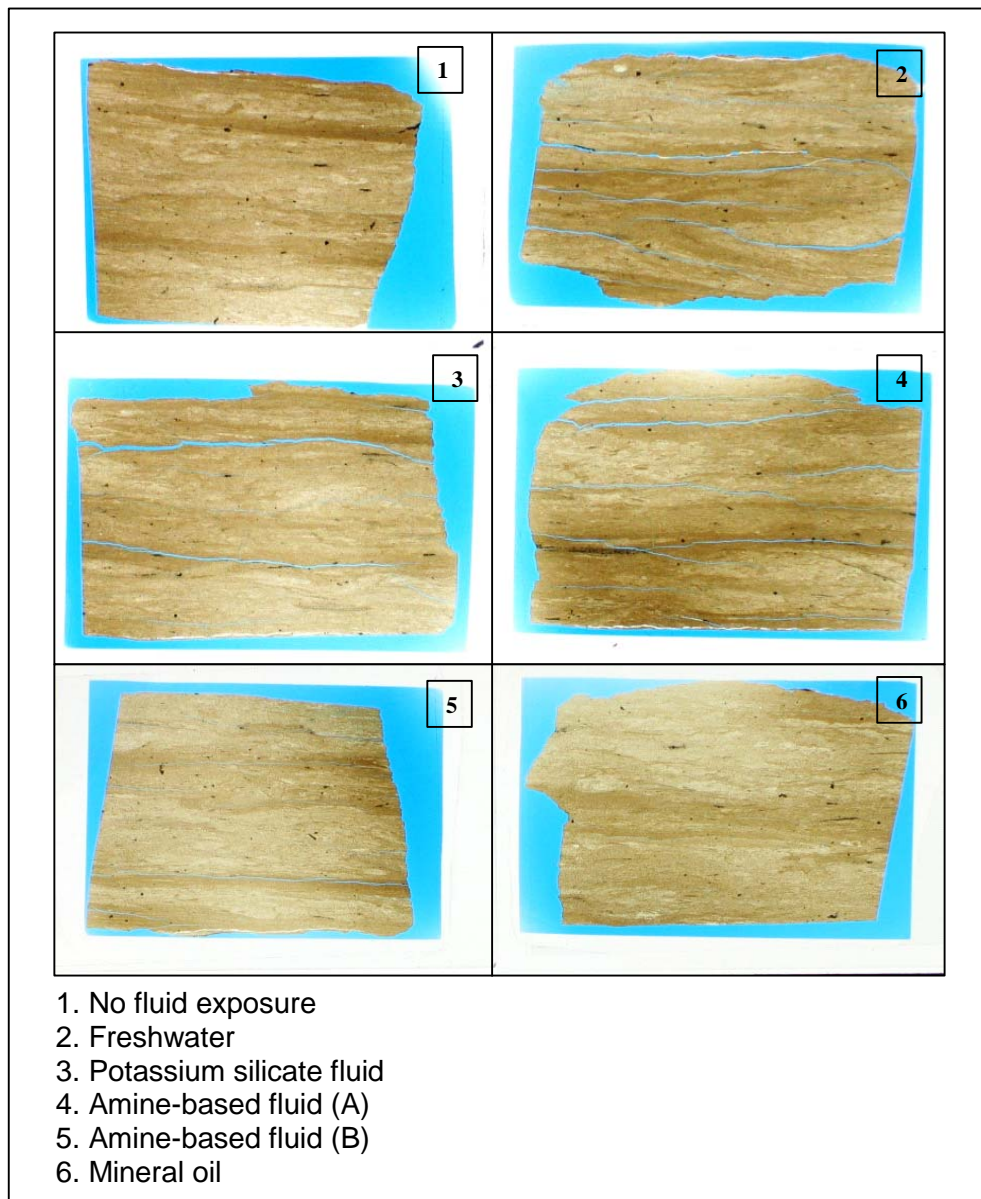
**Figure 12. Thin section of Nahr Umr shale after exposure to mineral oil for 6 hours**



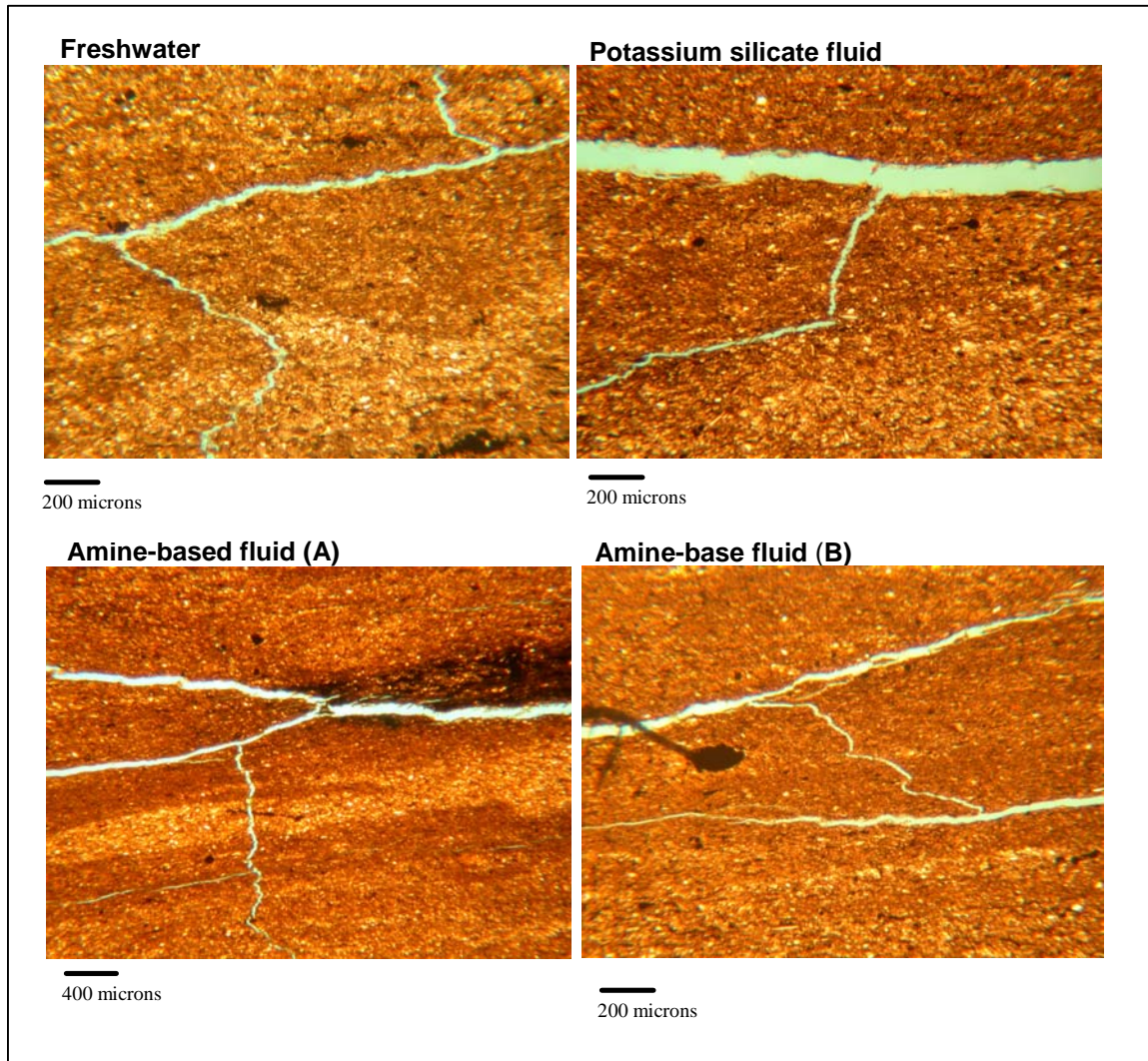
**Figure 13. Shale dispersion and bulk hardness tests for Los Monos shale**



**Figure 14. The change of Los Monos shale after exposure to different fluids for 6 hours**



**Figure 15. Thin Sections of Los Monos shale after exposure to different fluids for 6 hours. Pictures taken with a digital camera.**



**Figure 16. Photomicrographs of Los Monos shale taken with the petrographic microscope after exposure of the samples to four water-based fluids for 6 hours. Notice the intersecting fractures which are not as readily visible to the naked eye (as Fig. 15).**